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The Basics of Testing Plastics

MECHANICAL PROPERTIES, FLAME EXPOSURE, AND GENERAL GUIDELINES

STEPHEN BURKE DRISCOLL, EDITOR

The Basics of Testing Plastics: Mechanical Properties, Flame Exposure, and General Guidelines

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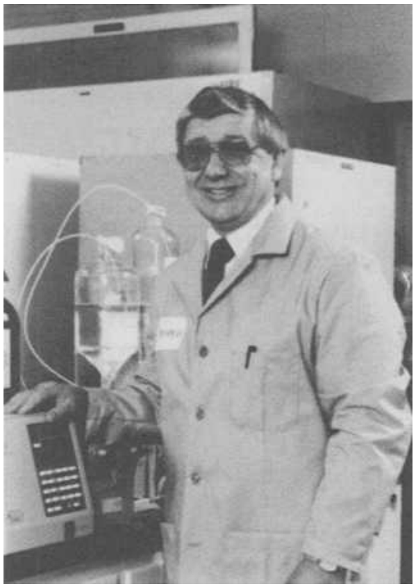
William Grieve

Dedication

This book is dedicated to the memory of William Grieve, a teacher, distinguished colleague and friend, and proactive contributor to the successes of many ASTM standards and projects.

Foreword

THIS PUBLICATION, *The Basics of Testing Plastics: Mechanical Properties, Flame Exposure, and General Guidelines,* was approved by ASTM Committee D-20 on Plastics. This is Manual 35 in ASTM's manual series.

Contents

Introduction

THE GLOBAL PLASTICS INDUSTRY is a dynamic, rapidly growing, and extensively diversified business. The domestic consumption of all plastics in the United States will soon exceed 100 billion pounds. These are found in a myriad of applications including plastics packaging, building and construction products, automotive and other types of transportation components, medical and dental devices, and aerospace composites. Each material and fabricated product demands a series of quality control/quality assurance procedures at every stage of the manufacturing process.

The intent of this manual is to provide basic information for persons who are not well-experienced in the testing of plastics. Such people are most likely to be:

chemists
materials engineers
technicians
salesmen
purchasing personnel
newly-appointed laboratory supervisors
quality control personnel
shop foremen
order correspondents
any others who may work with test data

Accordingly, only a minimum of theory is included. The manual is intended to provide introductions to the various subjects so as to prepare individuals to carry out additional study. It is not a compendium of testing methods. The subjects have been limited in order to best fulfill the above intent.

The material in Chapters 2, 3, and 4 was included, up until 1997, in the short course, "Major Testing Techniques for Plastics," sponsored by ASTM Committee D-20 on Plastics.

Chapter One "Classifications and Definitions," explains briefly some of the more important characteristics of plastics and how these relate to end-use performance. The various classification schemes illustrate the unique versatility of the polymer industry and how simple changes in the composition or structure of the polymer can significantly influence its processability and how it is used. This chapter also includes an abbreviated glossary of terms, definitions, and explanations of those important chemical additives used to modify the performance of many polymers.

Chapter Two, "General Guidelines for Conducting Tests and Evaluating Data," provides basic information and introductions to the various subjects. Typical topics include some basic starting points, the important parts of typical specifications and test methods, as well as how to deal with the variability of test data.

Chapter Three, "Measuring Mechanical Properties of Plastics," addresses the important aspects of many very important properties, including measuring tensile, flexural, creep, impact, fatigue, and weathering behavior.

Chapter Four, "Responses to Flame Exposure," emphasizes the fact that, although there are numerous small-scale test methods, there is no test that will faithfully predict how a specific material or assembly will respond to an actual fire scenario. Case studies will illustrate the problems associated with monitoring the combustibility of plastics.

This manual is the first in a planned series of volumes addressing the challenges and problems associated with testing plastics. Future manuals will focus on electrical characteristics, optical and chemical properties, rheological behavior, as well as global concerns for international testing harmonization, recycling, and other important analytical techniques. Currently, ASTM Committee D-20 on Plastics is responsible for more than 500 standard test methods, recommended practices, and guides. As the plastics industry grows, more sophisticated testing protocols are needed. One of the key components of the mission of Committee D-20 on Plastics is the continuous review and updating of existing documents and the authoring of new protocols that are necessary to ensure product quality.

Many of the Committee D-20 members actively involved in this mission have also contributed generously their time, energy, and expertise in preparing and reviewing this manual. Special appreciation is given to Paul Graboff, Chairman of D-20, for his guidance and encouragement, Alvin J. Flint for authoring these important foundation chapters, and the late William Grieve for his vision and enthusiasm for this project.

Stephen Burke Driscoll
Editor

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Classifications and Definitions

by Stephen Burke Driscoll[1]

BASIC CONCEPTS

AS STATED IN THE INTRODUCTION, the global plastics industry is an aggressively growing, diversified business consisting of a myriad of applied polymer science markets. In fact, the term *polymer* comes from poly-meros: many of the same repeating unit; the *mer* is used to describe many products. Polymeric materials range from structural adhesives and protective coatings to elastomers, man-made fibers, molded and extruded plastics, and reinforced composites.

This chapter will classify these polymeric materials using a series of marketing and chemical categorizing schemes. Many terms and descriptive phrases will be defined, and common examples will be cited to illustrate these distinctions.

It is important to recognize that many terms may be used interchangeably to describe the same characteristics. For example, the term *polymer* is preferred by chemists and physicists to describe their investigation of novel structures. Their discoveries are then synthesized by resin manufacturers, starting with small quantities, 1-L reactor vessels initially, and finally commercial production reactors having capacities of 100,000 L. The resin, usually shipped as small pellets or chips in 50-lb bags, 250-lb drums, 1000-lb gaylord boxes, and 200,000+ lb railcars, is then converted using many processing technologies, such as blow and injection molding or extrusion, into industrial components and consumer products. The companies that manufacture these products refer to themselves as plastics converters. Thus, in the production scheme, from concept to commercialization, different sectors of the industry use preferential terminology. However, the terms *polymers*, *resins*, and *plastics* are synonymous.

Definitions

In fact, according to the eighth edition of the ASTM Compilation of Standard Definitions—1984, these three terms are defined as:

> *Plastics*: a material that contains as an essential ingredient one or more organic polymeric substances of large molecular weight, is solid in its finished state, and at some stage of its manufacture or processing into finished articles can be shaped by flow.
>
> *Polymer*: a substance consisting of molecules characterized by the repetition (neglecting ends, branch junctions and other minor irregularities) of one or more types of monomeric unit. (IUPAC)
> *Resins*: A solid or pseudosolid organic material often of high molecular weight, which exhibits a tendency to flow when subjected to stress, usually has a softening or melting range and usually fractures conchoidally.

Fundamental to a discussion of polymers, resin, or plastics is a basic three-part definition: this unique material must be:

A. *organic*—containing carbon, although some polymers may be partially inorganic in nature
B. *high molecular weight*—25,000+
C. *plastic*—(adjective) able to change shape during the manufacturing process

Polymer Attributes

All three aspects are necessary to define a polymer. For example, wrought iron contains carbon and can be shaped, but it does not have a high molecular weight. Bread dough can also be shaped and contains organic materials, such as starches and carbohydrates, but it also is not high in molecular weight.

The concept of molecular weight and its distribution (MW, MWD, and branching are often referred to as the polymer's architecture) is very important. In fact, the molecular weight of a polymer is considered one of its three fundamental attributes, along with its percent crystallinity and its glass transition temperature, Tg. These attributes, while independent of each other, can be related to how the polymer is manufactured, how it can be processed, and how it can be used in a consumer or industrial product. The attributes will be described later in this chapter.

Ultimate Classification

The most important of the following ten schemes used to classify a resin is whether it is thermosetting (TS) or thermoplastic (TP). This ultimate classification simply indicates if the material can be remelted. Many plastics can be repeatedly remelted, often for several generations. These polymers, the thermoplastics, enjoy the economies of reuse. Their properties are a function of several parameters, including molecular weight, structure, and composition.

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[1] Professor, University of Massachusetts Lowell, Department of Plastics Engineering, One University Avenue, Lowell, MA 01854.

Thermoplastic Resin

A thermoplastic resin will melt when heated and will remain molten until it is cooled. The solidified polymer, which undergoes only a physical change, must be ejected from a cooled mold. If the molded product were not acceptable, it could be ground and recycled. In fact, one of the major advantages of thermoplastics is that these polymers can be recycled many times, often with minimal loss of properties.

An example of a thermoplastic-type process is water freezing into an ice cube, a long, time-consuming process. When properly solidified, again no chemical change, it can be ejected from the cold mold. If the shape of the ice cube were not acceptable, it could be easily ground, remelted, and molded again and again.

Examples of thermoplastics include the polyaddition families (defined later in this chapter): olefins, vinyls, and styrenics, and polycondensation products, including polycarbonate, polyamides, polyesters, and polysulfones. Thermoplastics are commonly processed using injection, blow, and rotational molding, extrusion, thermoforming, and powder-coating technologies.

Thermosetting Resin

A thermosetting resin develops its functional properties as it chemically and physically changes during processing. The polymer "cures" or crosslinks, analogous to the vulcanization of an elastomer. The thermosetting materials solidify through a chemical reaction. Some thermosets are cast as liquids and cured at room temperature, while others require external heating. This class of polymer will flow, often when heated, and will develop a complex three-dimensional structure when exposed to additional heating. It solidifies and can be ejected out of a hot mold. The thermosetting resins are based on the polycondensation reaction mechanism (explained later) and usually are relatively inexpensive. However, since the scrap cannot be reformed again economically, these polymeric materials are considered somewhat expensive.

An example of a thermosetting-type process is a hard-boiled egg. Initially it is liquid, but when exposed to heat, it will undergo a physical and chemical change; it solidifies and therefore can be removed from its hot shell (mold). However, if you do not like the shape of the hard-boiled egg, your only option is to make egg salad since the egg cannot be remelted. Cured concrete is another example of a thermosetting-type material.

Examples of thermosetting resins include phenolics, aminos, unsaturated polyesters, polyimides, silicones (an inorganic polymer), and epoxies. Thermosetting resins are processed using compression and transfer molding, casting, laminating, pultrusion, resin-transfer molding, filament winding or hand layup, and encapsulating and potting of electrical components.

Other Classification Schemes

Other classification schemes used to describe polymers include:

Molecular Composition

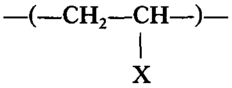
Since plastics are organic, they consist of carbon; however, they might also contain other important elements, including hydrogen, halogens (chlorine, fluorine, and bromine), phosphorous, nitrogen, oxygen, and silicon. The halogen and phosphorous contribute to flame-retarding behavior, and oxygen will often provide flexibility and toughness, but it could also pose a moisture sensitivity problem.

Reading about a fluoropolymer will alert you to the enhanced chemical and electrical properties but will also caution you that this resin must be processed at elevated temperatures using corrosion-resistant metallic alloys.

Chemical Grouping

Along the polymer chain and as pendant side groups: *aliphatic* groups, based on repeating —CH_2—, provide flexibility to the polymer, although they are prone to oxidative attack during processing at elevated temperatures. *Aromatic* groups, based on the large phenyl ring, C_6H_5, contribute stiffness, rigidity, hardness, and creep and heat resistance, but at the expense of impact resistance.

Subtle changes in the polymer structure will dramatically affect the performance (both processability and functional behavior) of the plastics. Listed in Table 1 is a series of examples based on the simplest organic group, vinyl:



$$—(—CH_2—\underset{\overset{|}{X}}{CH}—)—$$

Polymer Architecture or Morphology

Molecular weight (MW), molecular weight distribution (MWD) and branching.

The feedstock for producing a resin is the monomer, which contains the simplest repeat unit, the mer. Polymerization, or chain formation, is based on a three-step process. The first stage in the polymerization scheme is initiation, which involves the monomer reacting with itself. When two mers combine, a dimer is formed. When a third unit attaches to the dimer, it produces a trimer. The fourth link forms a tetramer. As the polymer chain grows, the propagation stage, the number of links in the chain increases. This is called the degree of polymerization, DP. When the DP equals approximately 100, the product is referred to as either an oligomer or a telomer. With the polymerization propagation reaching hundreds or thousands of repeating units, the macromolecule or high polymer has now attained a suitable molecular weight (25,000+) and the polymerization is stopped. The termination stage is the third and final step in the polymerization scheme.

Polyethylene is a good example of a thermoplastic resin that can range from low-molecular-weight products, such as 5000-MW processing aids and lubricants, to high-molecular-weight (75,000+) molding and extrusion compounds, to ultra-high-molecular-weight (3,000,000+) films laminated to ski bottoms.

Unfortunately, plastics are not composed of chains having the same length or degree of polymerization. Rather than having a "monodispersed" molecular weight, most plastics have a polydispersed MW. This mixture of different molecular weights is known as the molecular weight distribution, MWD. The MWD will affect both processability and functional performance. The narrow MWD will not perform similarly to the broad MWD resin grade (Fig. 1-1).

TABLE 1—Properties of Vinyl Polymers.

X is:	Plastics is:	Properties
H	Polyethylene (PE)	flexible, soft, opaque, chemically resistant, burns
	PE is extruded into wire and cable jacketing, blown film, and molded bottles.	
CH_3	Polypropylene (PP)	stiff, rigid, opaque, heat resistant, burns, poor low temperature, flexibility
	PP is molded into automotive components and extruded into packaging films and textile fibers. These two plastics, members of the olefin family (which consist of aliphatic groups) are semi-crystalline since they are based on carefully aligned, closely packed together simple repeating units, *mers*, containing only carbon and hydrogen.	
C_6H_5	Polystyrene (PS)	clear, strong, burns, poor chemical resistance, poor impact resistance
	PS is extruded into film and sheeting and molded into many consumer products, including pens, disposable glasses, and CD and cassette boxes. Although this polymer also contains only carbon and hydrogen, its properties vary dramatically from the olefins. The aromatic phenyl group contributes stiffness, rigidity, flatness, hardness, and good resistance to heat and creep. However, the size, shape, and arrangement of the repeating mer do not allow the polymeric chains to pack closely together. Consequently, crystallinity is not possible, and this material is referred to as *amorphous*, without structure.	
Cl	PVC	clarity, chemical resistance, brittle, slow burning, according to D 635, but marginal heat resistance
	PVC is an extremely versatile material, based on how it is chemically modified. It can be extruded into food-packaging films, calendered into furniture and automotive upholstery, and blow molded into containers. The addition of the third element, chlorine, significantly modifies the processability and performance of the polymer. Chlorine is the most commonly used halogen for contributing flame-retarding properties. Fluorine and bromine are used also, but are progressively more costly. The important combination of molecular weight, crystallinity, and polymer structure—as well as how the single mers combine to form the polymer network (macrostructure)—will influence the processability and will determine the functional characteristics and service performance of the polymer.	

Chain Structure

All plastics can be classified into four broad categories: (Fig. 1-2)

A. *linear*: long chains that can be either aliphatic or aromatic.

B. *branched*: long polymer backbones with pendant side chains.

C. *crosslinked*: linear chains connected to each other by short, low-molecular-weight groups, forming a complex three-dimensional network. The end-use properties are a function of the cross-linking density.

D. *ladder*: this configuration is a highly developed growth of 4-, 5-, and 6-member ring structures, tightly connected at multiple sites.

It is possible for a material to have more than one structure. For example, polyethylene, depending upon how polymerized, can be either linear (HDPE for blow and injection molding) or branched (LDPE for extruded films and electrical jacketing). Further, PE can be cross-linked, using three different chemical and irradiation techniques, for enhanced properties. There are several other plastics that are available in more than one configuration.

Homogeneity

Plastics can be based on one or more starting materials. A homopolymer is based on one starting monomer (ethylene polymerizes to polyethylene). Macromolecules based on two starting materials or co-monomers include impact polystyrene (styrene and butadiene) and styrene acrylonitrile (SAN).

Three starting co-monomers combine to form a terpolymer. Two common terpolymers are acrylonitrile-butadiene-styrene (ABS) and ethylene propylene diene monomer elastomers (EPDM).

The macromolecular arrangements of co- and terpolymers can be seen in Fig. 1-3.

Types of Copolymers

A. *random*: not usually commercial.

B. *block*: containing large segments or blocks of each co-monomer (ethylene/propylene copolymer).

C. *graft*: large molecules of one monomer grafted on to the backbone of the second co-monomer (rigid styrene grafted on to the flexible butadiene backbone).

D. *alternating*: typical of fluorocopolymers and the new generation of aliphatic polyketones.

The copolymer based on styrene and butadiene can be either the graft or block structure. However, the properties and ultimate uses are not similar for these two very different copolymers.

Alloys & Blends

Due to the high cost and prolonged development time for commercializing a new polymer, the trend today is to introduce a polymeric alloy or blend based on existing homo-, co-, and terpolymers that have proven track records of predictable, profitable processability. Each year there are several new alloyed systems introduced, based on either melt or solution blending techniques. Briefly, the good points of Polymer A will offset the limitations of Polymer B, and the selling points of Polymer B will offset the liabilities of Polymer A.

Examples include:

>ABS + PC<
>ABS + PVC<
>ABS + PU<
>PC + PET<
>PVC + PMMA<

Crystallinity

This is considered one of the three important attributes of a polymer. The extent of crystallinity will affect processability and end-use properties as does the effect of molecular weight

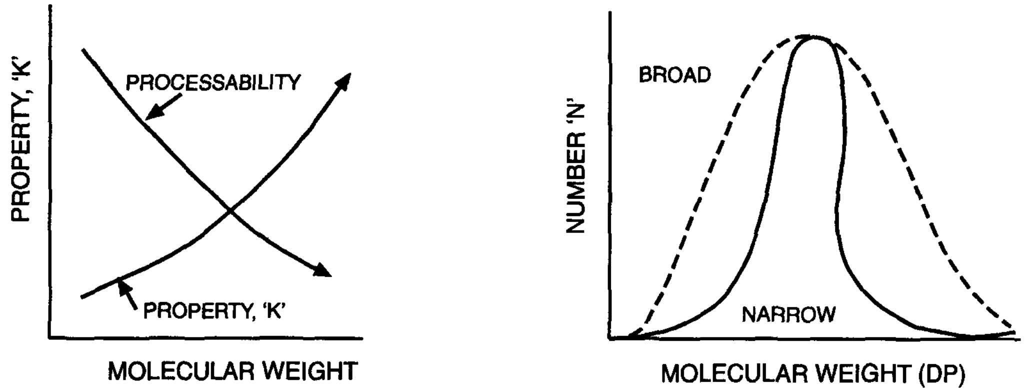


FIG. 1-1.—Effect of molecular weight and distribution of processability and performance.

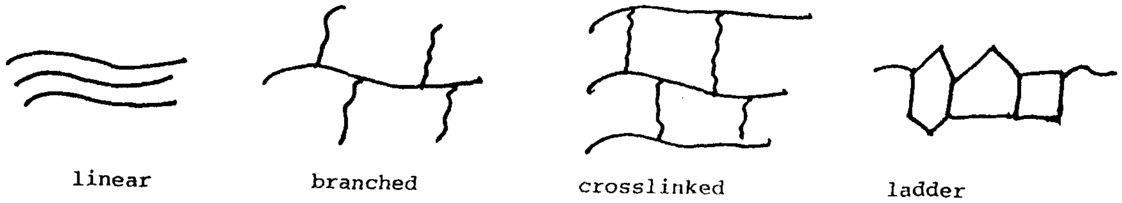


FIG. 1-2.—Basic polymer structures.

RANDOM ABAABABBABAABABBA

BLOCK AAAAAAAAAAAA-BBBBBBBBBBB-AAAAAAAAAA

GRAFT -BBBBBBBBBBBBBBBBBBBBBBBBB-
A A A
A A A
A A A
A A A
A A A

IDEAL (ALTERNATING)

ABABABABABABABABABA

FIG. 1-3.—Structures of co- and terpolymers.

and MWD. Starting with a completely amorphous (no crystallinity) polymer to steadily increasing crystalline content (percent crystallinity), the majority of physical properties will improve, but at the expense of processability. Modulus, strength, heat, and chemical resistance will increase, while impact resistance, clarity, and directional shrinkage will suffer. Additionally, the melting point and heat-sealing temperatures must be increased with increasing percent crystallinity.

Crystallinity can be promoted by selection of catalyst, mechanical design and processing, and thermal treatments (annealing versus quenching). Crystallinity is a function of the summation of bonding forces that combine to align the polymer chains in a very specific pattern or configuration. The macromolecular chains pack in closely and form a regular structure, resulting in a crystalline site. This can be compared to many forks or spoons stacked neatly together. This close packing promotes crystalline growth, although the smaller the size of the crystalline site, the better the clarity. Finally, crystallinity is often referred to as thermally reversible cross-links since when heated they disappear, yet reform when cooled slowly.

Chemical Formulations

Polymers are generally grouped into two major subdivisions based on how the reactants combine and if a by-product were produced:

Polycondensation

A. *(poly)condensation*: A + B═Poly C + by-product.

In the condensation scheme, two or more reactants or feed stocks combine, forming the desired polymer AND a by-product, which is usually water. These polymers can be either thermoplastic or thermosetting.

Examples of condensation polymers include:

1. *thermoplastic polyesters* (PET) and *polyamides* (nylons), which generate water as the by-product.
2. *thermoplastic polycarbonate*, which yields HCl as the by-product.

When preparing to process a condensation polymer, it is very important that it is dried properly. Otherwise, the polymer can depolymerize at the elevated processing temperatures; a small amount of surface or trapped moisture (often less than 0.3%) can lead to a significant loss of molecular weight and, consequently, functional properties, including impact behavior.

The majority of the second generation of engineering or high-performance thermoplastics is based on condensation chemistry.

A third example of a polycondensation resin is an epoxy, which is based on the reaction of bisphenol-A (BPA) with epichlorohydrin, yielding HCl as the by-product. Epoxies are thermosetting resins, and all thermosets are based on the condensation reaction mechanism. Other commonly used thermosets include phenolics, aminos, unsaturated polyesters, and silicones.

Polyaddition

B. *(poly)addition*: monomer A polymerized to Polymer A; there is no by-product

The addition-type polymers are thermoplastic; the most commonly used include members of the olefin, vinyl, and styrenic families.

Polymerization Engineering

Regardless of how many starting monomers, the structure of the polymer, or the molecular weight or its percent crystallinity, there are other classifications schemes, including design of reactor equipment, which can be used. For example, the reaction might be based on acidic or alkaline conditions or use either type of catalyst, the reaction can be anionic or cationic, and the reactor can be either the suspension, emulsion, mass/bulk, or solution type.

In all cases, polymers made using one set of these parameters will yield entirely different products than those based on other combinations. PVC can be produced using these four different polymerization techniques or methods. Although the vinyl chloride monomer (VCM) is common to all four, the final product does reflect major differences in how it can be used. Suspension-grade PVC is a freely flowing white powder having the consistency of granulated sugar. This particle size makes this grade ideal for injection molding, extrusion, and calendering. The emulsion grade PVC, which has a finer particle size similar to baking flour, is more suitable for coating applications, including plastisols and organosols. The mass/bulk polymerized PVC contains no suspending agent or emulsifier, resulting in a cleaner product; however, the absence of a heat-exchanging diluent could pose molecular weight control problems. These three processes yield a resin exhibiting a high molecular weight of approximately 100,000.

To make a low-molecular-weight PVC, it is more desirable to use the solution process. The resin is made in solution, kept in solution, and offers a molecular weight of 25,000, which is suitable for protective coatings, such as vinyl floor "waxes."

The choice of the reactor is a function of capital investment as well as the desired molecular weight, particle size, and output of the product. Generally, the higher the molecular weight of the polymer, the lower the reaction temperature and pressure and the longer the reaction time. Consequently, the choice of the polymerization method, the reactor selected, and the reaction conditions all combine to influence the final product.

For example, low gloss ABS terpolymer, used in automotive applications, is based on the mass/bulk polymerization process, while high gloss products are produced using the emulsion scheme.

Other examples, such as members of the olefinic family of polymers, also vary considerably in both processing and end-use performance based on the polymerization engineering details. Today an important new development is the choice of catalyst used in polymerization. A generic resin family can be expanded by employing different catalysts.

LDPE is based on a free-radical initiator at high reactor temperatures and pressures; HDPE is based on a Ziegler-Natta stereo-regulating or coordination catalyst using conventional low-reactor conditions. LDPE made using a tubular reactor is preferred for enhanced processability due to a broader molecular weight distribution and considerably less branching. However, the film optical quality is not quite as good as the autoclave reactor product, which tends to have a narrower MWD.

Linear low-density polyethylene (LLDPE) is made using a unique gas-phase, fluidized process; the new generations of polypropylenes are now also made using this process. The new generation of metallocene or single-site catalysts are now being used to yield specialty polyethylenes and olefinic elastomers that offer a narrow MWD for enhanced processing ease.

The plastics industry is today experiencing both revolutionary introductions as well as evolutionary developments. It is challenged to identify how to process properly new polymers and to refine compounding and alloying technologies using commercialized members of existing resin families.

The important role of additives will be discussed later in this chapter.

Performance of Polymers

The final classification scheme explained here is based on the performance of the polymer. This is really a marketing approach since the subcategories address key characteristics, including:

A. The number of manufacturers/suppliers.
B. The volume produced.
C. The selling price.
D. The functional properties offered.

There are three major subcategories:

Commodity Plastics

A. Even though many resins have been commercialized for decades, recent innovations in monomer synthesis, new catalysts, polymerization methods, and advances in processing technologies have opened up new market opportunities for these first-generation polymers, such as the olefins, vinyls, and styrenics, which are produced by many suppliers (free enterprise), sell in the billions of pounds annually in the United States, cost under $1.00 per pound, and offer marginal performance with respect to modulus, strength, and heat resistance/continuous use temperature.

Engineering Resins

B. These second-generation polymers, including nylons (polyamides), acetals, polycarbonates, modified-polyphenylene ethers, and polyesters, are supplied by just a few companies (oligopoly), selling in the hundreds of millions of pounds, are usually priced between $1.00 to $5.00 per pound, and exhibit enhanced functional behavior, including impact behavior as well as chemical and creep resistance at elevated temperatures.

Specialty Polymers

C. These are considered the 2.5 generation polymers and are supplied usually by only one company (pure monopoly), selling only several thousands to a few million pounds annually, are priced upwards to $20.00 per pound (and often in the hundreds to thousands of dollars per pound range), but are justified due to their exceptional balance of truly outstanding functional properties. Typical specialty polymers include polyimides, polybenzothiazoles, liquid crystal polymers (LCPs), and aromatic polyketones.

POLYMER MODIFIERS AND ADDITIVES

These first introductory comments have been related to typical molded or extruded polymeric material systems—plastics. However, it is also important to remember that plastics are just one of several members of the extended polymer family. Other members share common terminology, polymerization concerns, and marketing characteristics while also offering significant properties at attractive prices. In fact, very often the sales volume for these materials exceeds that of many commercial plastics. For example, the volume of polyamides used in monofilament applications (sutures, fishing line and marine ropes, filtering screens, and even "weed-wackers" is considerably less than the volume of nylon being used in the conventional textile business: carpeting and fabrics. Similar comments can also be made regarding polypropylene monofilaments (carpeting), polyester staple and fibers (carpeting and textiles), and cellulose (tri)acetate staple fibers (filters).

It is worthwhile, therefore, to mention briefly some of these other important members of the polymer family.

Adhesives

A number of polymers are used to make a variety of adhesives for bonding similar and dissimilar materials together. These include low-viscosity polyvinyl acetate (Borden's Elmer's Glue™); polyvinyl butyral sheeting as the inner tie layer for automotive windshield safety glass; the cyanoacrylate "crazy glue"; resorcinol formaldehyde liquid adhesives for plywood; thermoplastic hot melt adhesives based on ethylene vinyl acetate resins, polyesters, polyamides, and polyurethanes; and specialty epoxy structural adhesives for aircraft components.

Coatings

Decorative and often protective against the elements, such as paint on a metal to prevent rusting from moisture. Coatings can also include specialty products such as conformal electrically insulating silicone and polyurethane coatings on printed circuit boards. Another example is electrostatic or fluidized bed nylon, polyester, vinyl, and cellulosic coatings on hospital furniture and base-board heating panels. A third example is the specialty epoxy coating used for corrosion resistance inside the 900-plus-mile Alaskan pipeline.

Elastomers

These are macromolecules which, when deformed by a weak applied stress, will return rapidly to approximately their initial dimensions and shape after the stress has been removed. Elastomers can be either thermoplastic (styrenics, olefinic, polyesters, polyamides, and polyurethanes) or cross-linked. The term *vulcanization* refers to the Greek mythological god of fire, Vulcan, and is analogous to the concept of permanent chemical cross-links, which provide outstanding functional properties but prevent subsequent remelting and conventional melt processing.

There are a great number of elastomers, and both natural rubber and synthetic polymers are classified together generically. Similar to molded and extruded plastics, these elastomers are often extensively compounded using fillers and reinforcements, plasticizing oils, lubricants and release agents, colorants as well as a variety of cross-linking or vulcanizing agents. The processability and functional properties are related directly to the amount and type of additives used.

Like plastics, the elastomers can be classified as commodity (natural rubber and polybutadiene), engineering (nitriles and ethylene-propylene-diene monomers (EPDM)), polysulfides, and specialty products including silicones, fluoropolymers, and fluorosilicone copolymers.

Similar to molded and extruded plastics, rubber and elastomers are most often used after the base or "neat" polymer has been modified. In only the rarest occasions will a polymer be used unmodified. Examples of unadulterated polymers would be found in the medical and dental sciences.

Polymer Modifiers

The incorporation of a polymer modifier, or additive, is usually a post-reactor process. Only a few of these specialty chemicals can be easily integrated during the polymerization process. The principal advantage of these internal or reactive modifiers is that the added chemical grouping, now chemically linked to the polymer backbone, will be permanent and, therefore, less prone to chemical attack, migration, exudation, or leaching. However, this significant advantage is offset by the potential disruption of the polymerization kinetics.

Consequently, the majority of polymer modifiers are added after polymerization. This external or additive-type modifier does allow versatility in how and when the modifier is compounded into the polymer. Variations in amount and type of modifier, as well as the equipment used, will affect processability (uniformity of dispersion, which influences viscosity) and functional properties.

The following is an abbreviated listing of typical polymer modifiers. Many of these are self-explanatory, although the actual theory and guidelines for their use may be quite complicated.

The trend these days is to develop a single-component, multi-functional additive, one product or "package" that will provide a few or several improvements. A good example is carbon black, which acts as a reinforcing colorant, antistatic agent, and UV absorber (screener). The basic premise of these new products is to enhance several characteristics using only one modifier. Why can't the impact modifier also serve as a processing aid while providing color and flame-retarding properties? This same question can also be asked of so many other types of single-function modifiers and single-component concentrates (fillers, colorants, lubricants).

TABLE 2—Typical polymer modifiers.

Antioxidant	To ensure thermal stability against oxidation during high-temperature processing.
Antiozonant	To ensure protection against ozone attack, especially in rubber-modified polymers.
Antistatic agents	To protect the polymer from excessive static charge accumulation due to processing; often the equipment may be grounded to aid in discharging static electricity buildup.
Blowing Agents	These can be either physical or chemical types. The physical only changes its form; when heated, the liquid will become a gas. Expanded polystyrene (EPS) is a typical product based on this type of blowing agent. The chemical blowing agent (CBA) decomposes when exposed to a critical temperature, and the noncorrosive gaseous product expands the molten polymer. These are commonly used in molding structural foams. Different types and concentration of CBAs will result in very different physical properties, even at the same density reduction.
Colorants	Can be either organic dyes or organic and inorganic pigments. There are very specific rules for colorant selection based on resin(s) used, amounts, and end-use requirements.
Fillers / Reinforcements	A filler can be (1) an *extender type*, which is used primarily to lower overall compound cost; (2) a *functional filler*, which is inexpensive but also contributes one or more desirable performance properties (modulus or impact and heat resistance); or (3) a *reinforcement*, which is usually more expensive than the base resin, but does contribute to a significant enhancement of modulus, tensile and flexural strengths, and upper use temperature. Fillers tend to be fine powders. Reinforcements possess greater physical size (high-aspect-ratio fibers, papers, honeycombs).
Flame Retardant	Often used with smoke suppressants, these additives slow down the burning rate of a polymer, often by generating a heavy smoke or moisture to "cool the fire." However, as with some other additives, many properties suffer.
Impact Modifiers	Significantly enhance impact behavior but at the expense of modulus, strength, and heat resistance.
Lubricants	Also known as release agents, these are added either during compounding or during the actual melt processing. They prevent the hot polymer from "wetting the metal," causing sticking to the metal screw or barrel. Thus, they make it easier to remove formed parts from their molds.
Plasticizer	The primary functions of a plasticizer are: (1) to enhance processability by reducing stock (melt) temperature and head pressure and (2) to improve basic properties, such as impact behavior, low-temperature flexibility, and softness. Unfortunately, modulus, strength, and upper-use temperatures do suffer along with chemical resistance.
Thermal Stabilizer	The primary function of a stabilizer is to ensure short-term stability during processing at elevated temperatures. Thermal stabilizers, a critical component of any vinyl compound, are used to absorb the released HCl generated during processing. If not properly absorbed/controlled, the vinyl polymer will undergo chain scission or *autocatalytic dehydrohalogenation*, most commonly known as degradation.
Ultraviolet absorbers	These modifiers are added in small amounts to absorb the harmful, high-energy UV rays that will attack polymers. Theirs is a long-term function, often over many years, unlike those additives that are used to protect the polymer only for a few minutes during high-temperature processing.

General Guidelines for Conducting Tests and Evaluating Data

2

by Alvin J. Flint, Jr.[1]

INTRODUCTION

THIS CHAPTER COVERS SOME basic starting points and then emphasizes the most important parts of a typical specification and of a typical test method. Special emphasis is placed on the importance of dealing with the variability of test data.

PURPOSES OF TESTING

The test methods that we will cover were generally not written to provide proof testing of a finished product in relation to its intended use. Rather, the methods were written to standardize the measurement of inherent properties of materials, as applied to:

A. *Quality control testing.* Quality control testing is carried out for either of two purposes:
   1. By a producer, in order to determine whether a batch (cook, lot, shipment, etc.) of material meets the requirements of a specification or a contract, or some other agreed-to standard. In this type of testing, people often do not care what the *actual* test data are, as long as it can be certified that the material meets the minimum or maximum requirements of the applicable standard.
   2. By a producer, for in-process control testing. When used this way, it is likely that shortcuts will be taken, such as omitting a 40-h conditioning period, or testing fewer specimens than the number required by the standard.
      a. There is nothing wrong with doing this, as long as the producer also conducts tests that fully conform to the standard on the final product that is to be shipped. If there has been any deviation from the cited test method, or any other requirement, the producer must so notify the customer in writing. Usually, this will appear as a special note on a certification sent to the customer. Failure to do so may very well result in a lawsuit, if the customer has any difficulty processing the material or if failures subsequently occur in the field.
   3. By a customer, to check on the quality of incoming material.

B. *Testing to solve specific problems.* In this type of testing, the specific test data are very important, as decisions concerning future investment in a project often may be based, unfortunately, on relatively small differences in test outcomes. This may include:
   1. Testing in connection with customer service work.
   2. Testing to evaluate the progress of basic research, applied research, product development, etc.

"FALSE FACTS"

> "False facts are highly injurious,
> for they often endure long!" (Darwin)

What Are False Facts, and How Are They Generated?

The author, as well as many other testing professionals, has frequently been involved with scenarios that start out like this:

Phase 1 of a Scenario

"Chester Chemsmith" submits a sample for tensile testing and demands that it be done immediately, without conditioning the material as required by ASTM D 638. "Phil Testgood," the physical testing supervisor, cautions Chester that:

A. The mechanical properties of this material are greatly affected by moisture content.
B. The sample is not worth testing, as it contains numerous voids, surface irregularities, and irregularly dispersed filler.

Chester insists that:

A. He realizes all this, but
B. he needs "a number" immediately, for a prospective new customer, and
C. he will take these factors into account in evaluating the test data.

Taking Responsibility for the Company's Interest

The author has, historically, tried to convince Chester that such testing is not only a waste of time, but dangerous to the reputation of the company. In some cases, this fell on deaf ears, and the author then flatly refused to allow the testing to be carried out unless Chester could get his boss to convince the author's boss to order the author to do so. The author is gratified to report that, in 24 years, he never received such an order—not even once! The reason for the author's stubborn action was to avoid the following possible outcome of such a scenario.

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[1] Formerly, supervisor of the Physical Measurements Laboratory, ICI Polyurethanes, West Deptford, NJ. Now retired. Present address: 109 Hoiland Drive, Shipley Hts., Wilmington, DE 19803-3227.

Phase 2 of the Scenario

Phil finally relents and allows the material to be tested. Chester records, in his notebook, only the average results for tensile strength and elongation. He pays no attention to the wild scatter of the raw data, and he does not bother to record the standard deviation. Also, of course, he makes no notation of the poor quality of the sample or the lack of conditioning even though he had promised to take those factors into account.

The next day, "Mary Goodhand" (secretary to the prospective good customer's purchasing agent) phones in, asking for the results of the above testing. Chester is absent, so "Sue Fastback," Chester's supervisor, obligingly looks in Chester's notebook and phones the averages to Mary. (Of course, the only information Sue is aware of is the averages—not the lack of conditioning, the poor quality of the sample, or the resulting wide scatter of the raw data.) Mary inquires whether the material was tested according to ASTM D 638, and Sue assures her that the company always follows ASTM methods religiously in all of their testing. (Author's note: Ho, ho, ho!)

What Are the Dangers of "False Facts"?

The use of false facts may lead to either of two wrong courses of action:

A. The formulation may really be wrong for the customer's needs. However, not knowing that the data are worthless, the customer may decide that the formulation is uniquely suited to his needs and that further development work should be carried out. This is sure to lead to great disillusionment when it is later found that follow-up testing gives results entirely different from those originally reported.

- NOTE: A good sample, properly tested, might have shown that the formulation was not suited to the customer's needs, thus avoiding a subsequent waste of time and money and allowing time to try other formulations before the customer loses faith in Chester's company.

B. The formulation may really be right for the customer's needs. However, not knowing that the data are worthless, the customer may decide that the formulation is unsuited to his needs and that the project is not worth pursuing.

- NOTE: A good sample, properly tested, might have shown that the project *was* really worth pursuing, with possible opportunity for profit to both Chester's company and the customer.

Phase 3 of the Scenario

Let us say that Case B was the result, so Chester did no further testing of the formulation. Two years later, Chester has left the company. His successor, "Hope Springer," in looking through Chester's notebook, feels that the above formulation relates to a new, completely different project that she wants to work on. Remember, there is nothing in Chester's notebook to indicate that the data are unreliable! Hope then wastes weeks of time and tens of thousands of dollars of the company's money trying to get similar data from good specimens made from the formulation.

The possibilities for unintended mischief are manifold. The worst part of it, these days, is that it is highly likely that the false facts will wind up in a computerized database, thus spreading the misinformation beyond anyone's control.

Other Ways of Generating False Facts

A. *Failure to Follow Instructions Completely.*

1. In using a test method or specification, it is vitally important to read the standard carefully and follow instructions completely if one hopes to avoid false facts. You may think, "Doesn't everyone?" Consider the following true instances:
   a. A few years ago, ASTM Section D-20.10.01 conducted an interlaboratory study to measure the precision of ASTM D 638, Test Methods for Tensile Properties of Plastics. This was, and is, one of the most widely used methods in the field of plastics testing. When the results were tabulated, there were obvious gross differences among the various laboratories' results for tangent modulus. Further checking disclosed that the aberrant laboratories had not used extensometers for the modulus determination in spite of clear instructions in the test method that this was vitally necessary. And these were laboratories whose experienced, supposedly reputable, personnel should have known better, and who were probably certifying that they were testing in full accordance with D 638.
2. There are frequent quirks in physical testing methods where logic would indicate one thing, but experience proves the opposite to be true. An example:
   a. Some test methods require that the specimen have a thickness of at least 3.2 mm (0.125 in.), and the *actual* thickness is one of the factors in calculating the test data. It would seem that one could substitute two 1.6-mm (0.0625-in.) specimens without any problem. The fact is that doing so would be okay in only a few of the methods; in most cases, it would produce a false fact because the composite specimen will behave differently than a single specimen of the same overall thickness.

B. *Basing Conclusions on Insufficient Data*
Is Another Way of Generating False Facts

We shall discuss this more thoroughly later on.

"SAMPLES" VERSUS "SPECIMENS"

These terms can be confusing, as they are defined differently for physical testing than they are for analytical work. For analytical work, a specimen is submitted for analysis and is then divided into samples; the individual determinations are made on two or more samples. For physical testing, a sample may be cut into two or more specimens, and the individual determinations are made on two or more specimens.

NUMBERING SYSTEM FOR ASTM STANDARDS

ASTM standards include specifications, test methods, standard practices, standard guides, classifications, and definitions. Our discussion here will concentrate on specifications and test methods.

ASTM standards are identified by a capital letter, followed by a 1-to-4-digit number, followed by a dash and a two-digit number. For example, in "C 421-88," the "C" indicates that the method was initially written for a ceramic, concrete, or masonry material. The letter codes are:

- A = Ferrous metals.
- B = Nonferrous metals.
- C = Ceramic, concrete, or masonry material.
- D = Miscellaneous materials.
- E = Miscellaneous general subjects.
- F = End-use products.
- G = Corrosion and deterioration standards.

A. The 1-to-4-digit number was assigned chronologically when the standard was first submitted to a Society ballot.
B. The two-digit number indicates the later of two possible events—the year in which the standard was written or the year in which it was most recently revised.

In "D 2124-70 (1994)," the "(1994)" indicates that the standard was most recently reapproved in 1994, but the "70" shows that no revisions have been made since 1970.

ASTM Committee Jurisdictions

There are over 100 different committees that generate standards through ASTM, all composed of volunteers (both as producers and as consumers) from industry, commerce, the academic world, the military, other government branches, etc. Each of the committees has specific jurisdictional boundaries. Some committees have jurisdiction over specific types of materials or products, such as committees:

- D-20 on Plastics.
- A-4 on Iron Castings.
- B-7 on Light Metals and Alloys.
- C-24 on Building Seals and Sealants.
- F-8 on Sports Equipment and Facilities, etc.

Other committees have more general jurisdictions, such as:

- E-5 on Fire.
- E-9 on Fatigue and Fracture.
- E-11 on Quality and Statistics.
- E-28 on Mechanical Testing etc.

Most of the standards that we use in testing plastics come from Committee D-20 on Plastics, but we also use test methods from other committees, such as Committee D-11 on Rubber, D-30 on Composite Materials, E-16 on Thermal Conductivity Measurements, etc.

CHARACTERISTICS AND IMPORTANT PARTS OF A SPECIFICATION

As an example of a specification, we are going to discuss ASTM D 2103-92, Specification for Polyethylene Film and Sheeting.

Section 1, "Scope," defines the limits of applicability of a specification. It is important to read this in order to be sure that you are using the proper specification for the material in question. For instance, in this case, the specification does not apply to material greater than 0.3 mm in thickness.

Section 2, "Referenced Documents," is a convenient listing of the other standards that are referenced anywhere in other parts of the specification.

Section 3, "Terminology" (in older standards, named "Definitions"): The only terms appearing in this section are technical terms whose definitions:

A. are not included in ASTM D 883, Terminology Relating to Plastics or
B. are different from those in D 883.

Section 4 describes a classification system that is applicable to material covered by this particular specification. Not all specifications have this.

Sections 5 and 6, "General Requirements" and "Detail Requirements," and the related Tables 1, 2, and 3, set forth the tolerances and other physical property limits for material covered by this specification. Not all specifications have this.

Section 7, "Sampling," gives instructions as to how samples are to be selected from production quantities of the material for quality control inspection and testing. The procedure described in this specification is somewhat vague. In many other specifications, more stringent sampling plans are defined.

Section 8 states which specific test methods must be used to assure compliance with the general and detail requirements and Tables 1, 2, and 3. In addition, paragraphs 8.7 thru 8.7.4 and 8.9 thru 8.9.1.3 of this particular specification also give detailed instructions for two additional test methods.

Referring to external standard test methods is the most common and most satisfactory way of ensuring that the proper methods will be followed, because the external test methods usually have been subject to more critical review and thus are more complete than methods internal to a specification.

Requirements for packaging and package marking are given in Section 9. Compliance with these requirements is often of vital concern, especially in the case of contracts with the military or other branches of the federal government.

Some specifications also have a section on quality assurance provisions for government/military procurement.

At the end of the standard is an *appendix*. An appendix contains information that is useful, but not mandatory for compliance with the standard. In some cases, it is desirable to include an *annex;* by definition, this contains information that is vital to the understanding and proper use of the standard but is more convenient to include outside the main body of the standard. An annex is a binding part of a standard.

PURPOSE OF ASTM METHODS

Although data from ASTM test methods often are applied to product development or design situations, it is important to recognize that they are primarily written to standardize procedures for quality control and acceptance testing. Therefore, they are written, reviewed, and revised with the primary goal of ensuring consistency of test data, rather than piquing scientific investigation.

CHARACTERISTICS AND IMPORTANT PARTS OF A TEST METHOD

As an example, we are going to discuss ASTM D 882-91, Test Methods for Tensile Properties of Thin Plastic Sheeting.

- *Section 1,* "Scope," and *Section 4,* "Significance and Use," define the limits of applicability of the test method and give other general information about the method. It is important to read these sections to be sure that you are using a method that applies to the material in question and that will give the type of data that is needed. For instance:

A. Properties of flexible materials often may not be measurable by methods that were written for rigid materials.
B. Properties of cellular materials are usually not measurable by methods that were written for solid materials.
C. Mechanical property data obtained at "static" speeds cannot be extrapolated to give data for lower or higher test speeds.

- *Section 2,* "Referenced Documents"—same as for specifications, above.
- *Section 3,* "Terminology"—same as for specifications, above.
- *Section 5,* "Apparatus," describes (or lists) any special equipment that is needed to properly carry out the test. It is often augmented by pictures or diagrams, which are labeled as "figures" at the end of the standard.
- *Section 6,* "Test Specimens" and Section 8, "Number of Test Specimens": It is of vital importance that the method describe, in detail, the geometry and the dimensions of the specimens required for the test, including dimensional tolerances. It is also necessary to state the number of specimens to be tested for the calculation of a test result. Note: (A test result is most often an average (mean) of the determinations from several specimens. However, some methods state that the test result shall be a median or an LD_{50} or some other function of the raw data.)
- IMPORTANT! This information is not always nicely broken out into the separate sections that appear in D 882. Often it is necessary to hunt through the "Procedure" section, and sometimes other sections, in order to find the information.

Many plastic materials or products are anisotropic; that is, the physical properties measured in one direction are significantly different from those measured in another direction. Note that most test methods require testing in both directions if the material is even suspected of being anisotropic (see Section 6.6).

- *Section 7,* "Conditioning": The conditions stated here are the same as for almost all other methods for measuring the mechanical properties of plastics. (See a discussion of this in Chapter 4.)
- *Section 9,* "Speed of Testing": Here, again, the information is seldom nicely broken out into the separate section that appears in this method. It usually appears somewhere in the "Procedures" section, but it may be necessary to hunt through other sections in order to find the information. For mechanical property tests, speed of testing is very important.
- *Section 10,* "Procedure": This section describes all of the action steps that are necessary to follow in making the determinations on the specimens. Obviously, this is the section that all of the preceding ones lead up to. Again, we must stress the importance of following all of the requirements of the method to the letter if you expect your results to be similar to results obtained by someone else on the same formulation!
- *Section 11,* "Calculation," describes how to convert raw data into test results. The information often is given in the form of algebraic equations. Be sure to follow these instructions faithfully, as the calculation of a property in one method may differ from the calculation of the same property in a different test method.

A. However, in the case of equations for calculating standard deviation (which is explained in a later section), note that there are five different equations that have been used, at one time or another, in various test methods. They will all give the same answer. The problem is that the equations sometimes get printed incorrectly in a standard. It is advised that you adopt the equation that is shown further on (in the section on "Standard Deviation") and use it for all of your calculations of standard deviation, regardless of which equation is quoted in the method.

- *Section 12,* "Report": This section lists the information that is necessary to constitute a formal report of the test data and results. If you deviate from the method in any way in carrying out the test (apparatus, conditioning, specimen geometry or preparation, speed of testing, etc.), it is important that you state all such deviations clearly on the test report. Otherwise, you may not legally state that you have performed the test according to the cited method.
- *Section 13*: For the past several years, all ASTM test methods have been required to address the *precision* and *bias* of the method. These two terms, together, constitute the accuracy of the method. We shall discuss this whole problem below, under "Accuracy."
- *Annex*: The annex in this standard is a good example of information that is vital to the understanding and proper use of the standard but is more conveniently included outside the main body of the standard.

EVALUATING TEST DATA

One of the author's respected colleagues was a chemist named Joe Burns. Joe used to say, "Too many people use data and statistics like a drunk uses a lamp post—for support, rather than for illumination."

When examining data of any kind, almost everyone looks at the average (mean) value before they look at the scatter (if, indeed, they pay any attention at all to the scatter). This is a grave mistake, which can promulgate false facts which, in turn, can cause lots of trouble later on. You should always examine the scatter of data before looking at the mean. In many cases, the scatter is far more important than the mean.

A. If the scatter is wide, the mean should be regarded as being much less reliable than if the scatter is tight. In some cases, the scatter of data is so bad that the mean value is of no practical use.

   1. A pesky sea gull was stealing a fisherman's bait, so the fisherman went back to the cottage, got his shotgun, and gave him both barrels! Figure 2-1 shows the pattern of the shots—on average, the sea gull is dead!—but he is still stealing bait!
   2. Also, have you heard about the man who drowned while wading across a river whose average depth was only 1.3 feet?

B. In comparing samples, if data from any sample show much more scatter than data from other samples, then the samples are not equivalent, even though the calculated mean values may be exactly or nearly the same. For example:

   1. Figure 2-2 is graphical representation of two mean values, A-bar and B-bar. With only this information, we would conclude that there is probably no significant difference between the samples.
   2. However, in Fig. 2-3*a*, the values of the five "a" specimens are plotted, and in Fig. 2-3*b*, the values of the five "b" specimens are plotted. With only a glance, we readily see that the scatter of the five specimens "b" is much greater than the scatter of the five specimens "a." Thus, the two samples are really entirely different!

The author's experience has been that most people who use data, of any kind, have an overly optimistic perception of the precision of the data. This makes them think that there are meaningful differences among test results, even though the scatter of the data is so broad that the apparent differences are false. The opposite type of mistake is less likely to occur.

You should develop a "feel" for the usual approximate scatter of data for the materials and tests that are of interest to you.

ACCURACY (PRECISION AND BIAS)

The term "accuracy" has been bandied about for years. It is a term that almost everyone thinks that he or she understands. However, various people have quite different mean-

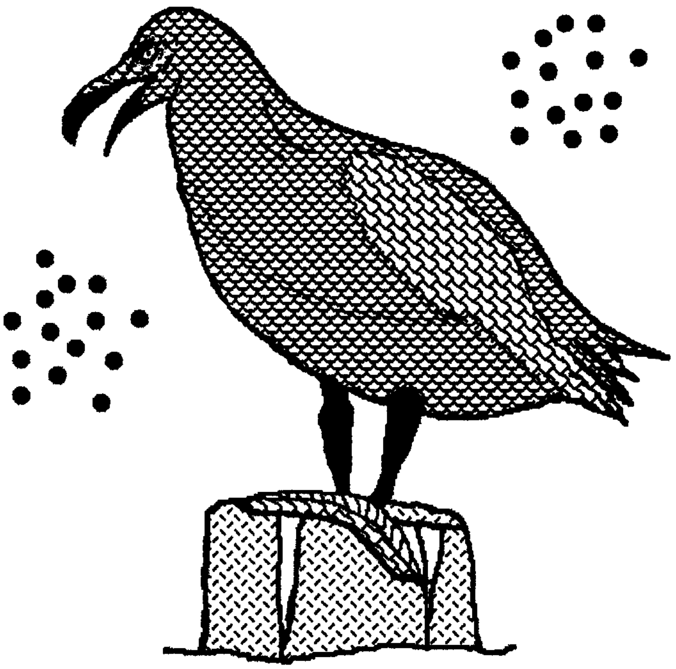
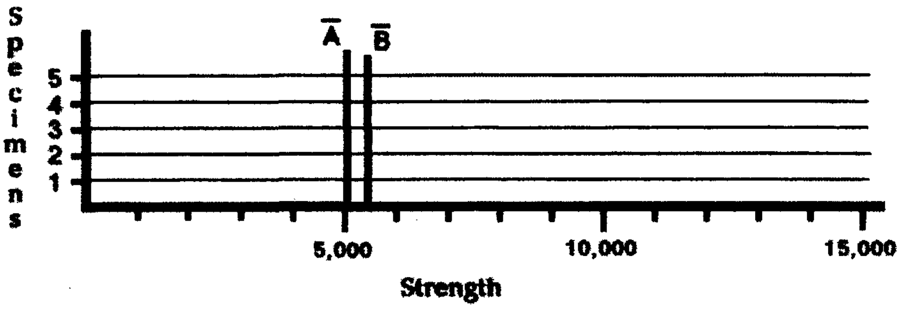


FIG. 2-1—On average, this bird was shot.



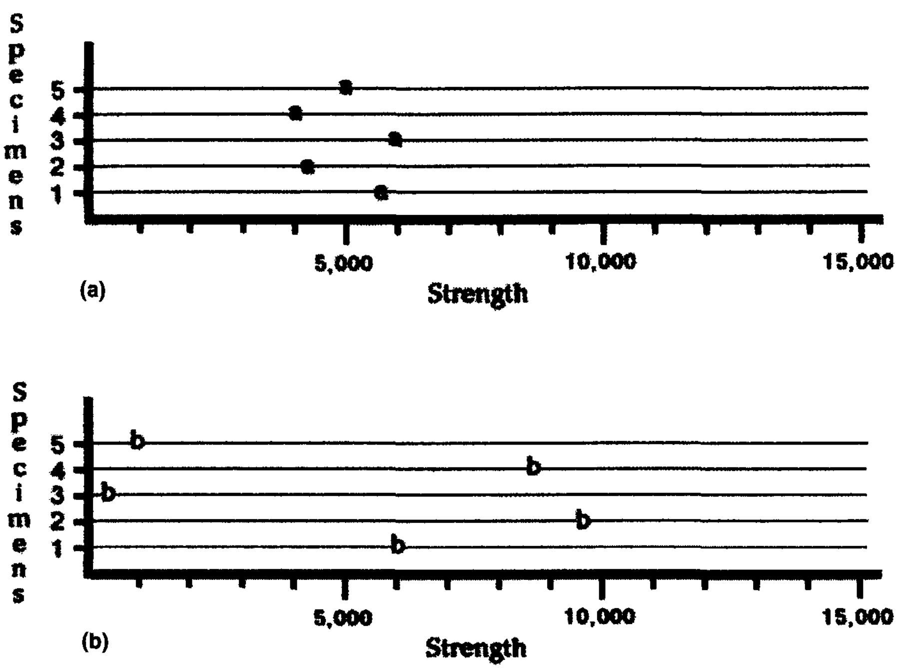


FIG. 2-3*a*—Tight scatter. *b* Wide scatter.

ings in mind when they use it. In ASTM usage, accuracy is defined as consisting of two components—precision and bias.

One way to describe *bias* is to regard it as "by how much you missed the bull's-eye." *Precision* is a measure of the amount of scatter of data. Figures 2-4*a* thru 2-4*d* show the concepts in terms of shooting five single shots at a target:

A. In Fig. 2-4*a*, the spread of the shots is very small, so the precision is good. All of the shots are in the bull's-eye, so the degree of bias is very small. We would like to achieve good precision and low bias in all of our testing.
B. In Fig. 2-4*b*, the spread of the shots is very small, so the precision is good. However, all of the shots are wide of the target, so the degree of bias is large.
C. In Fig. 2-4*c*, the spread of the shots is very wide, so the precision is poor. However, the center of the spread is in the bull's-eye, so the degree of bias is small.
D. In Fig. 2-4*d*, the spread of the shots is very wide, so the precision is poor. The bias is also poor, as all of the shots are wide of the target.

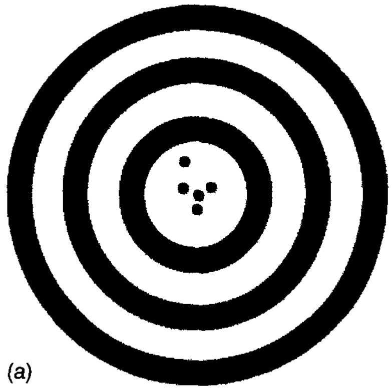


FIG. 2-4*a*—Good precision, little bias.

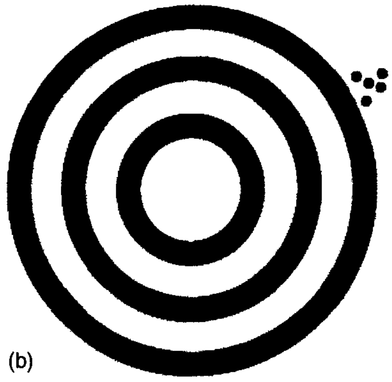


FIG. 2-4*b*—Good precision, high bias.

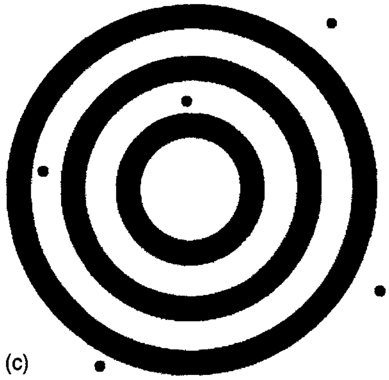


FIG. 2-4*c*—Poor precision, little Bias.

Bias

As applied to test data, bias may be explained as follows. We characterize a sample by calculating a test result from a set of determinations. The determinations are made on speci-

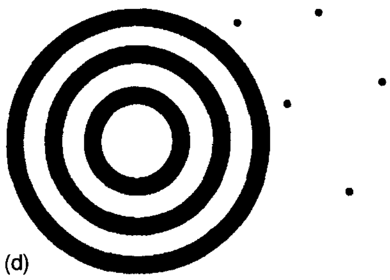


FIG. 2-4d—Poor precision, high bias.

mens that we hope are representative of the sample. In turn, we hope that the sample is representative of the whole batch of material. (In statistical terms, we would refer to the batch as a "population.") In reality, there is almost always some difference between the sample average and the true population average. This difference is bias.

As will be discussed in Chapter 4, there are standards and methods for calibrating the various testing machines. However, there are almost no standard reference materials that can reliably be used to establish the bias of sample test results or of test methods. The few standard reference materials available usually do not exhibit properties in the same magnitude as whatever plastic material is of interest.

For example, the reference standard for measuring thermal conductivity is made of fiberglass; its thermal "R" value is 4.2, while the R value of a rigid polyurethane cellular plastic may range from about 6.7 to 9.1. And, of course, some people use piano wire as a reference material, but its tensile strength is much higher than that of most plastic materials.

A. Because of this, the author strongly encourages participation in interlaboratory testing programs. Doing so is usually the only way of finding out whether your laboratory is generating reliable test data.

Despite the above, it is appropriate to inform the reader, at this point, that the most likely source of reference materials for other materials and uses is the National Institute for Standards Technology (NIST) at Gaithersburg, MD. Until a few years ago, it was named the National Bureau of Standards (NBS).

Precision

The wider the spread among determinations, the poorer is the precision. The narrower the spread among determinations, the better is the precision.

The scatter of test data will be different from one type of test to another. For any given test, the scatter will be different from one material to another. For any given material, the scatter will be different from one formulation to another. For any given formulation, the scatter will be different from one batch to another.

REASONS FOR VARIABILITY OF TEST DATA

What are some of the components that, added together, comprise the variability of test data?

A. In plastics, the largest component of variability is the material itself because even unprocessed plastics are inherently not homogeneous in structure. Processing further reduces homogeneity through molecular orientation, stress patterns, uneven dispersion of fillers or reinforcements, etc., etc.

B. The second largest component of variability is usually specimen preparation.

   1. If specimens are molded, their properties may vary with mold design, mold temperatures and pressures, type and position of gating, etc.
   2. If the specimens are sawed and machined from samples, those operations will produce varying degrees of tiny surface defects that may affect individual test determinations.
   3. In either case, there will be property variations within each specimen, as well as between specimens.

C. In some tests, certain specimen dimensions may not enter into the calculations, and thus deviations from tight dimensional tolerances may produce an error.

D. Other components that may affect variability, to some degree, include:

   - Differences in specimen conditioning or aging times.
   - Differences in speed of testing.
   - Temperature and (or) humidity fluctuation during testing.
   - Fluctuations in electrical power.
   - Variations of technique, skill, and attitude among personnel.
   - Different designs, ages, and maintenance of test equipment.
   - Differences between units of similar test equipment.
   - Phase of the moon (?), etc., etc., etc.

TOOLS FOR EVALUATING SCATTER

Standard Deviation

The amount of scatter (variation) among a set of test determinations is usually reported as standard deviation. Many people who generate or use test data (including professionals) do not ever develop any sense of what standard deviation is or how to use it. The author has seen them incorrectly quote a value of one standard deviation as being the same thing as a 95% confidence limit or a 95% least significant difference between two averages. It is neither of those. It is, of course, merely the basic tool for calculating those (and many other) useful statistical guidelines and evaluations.

Calculating Standard Deviation, "s"

Standard deviation may be calculated as shown in this example. Suppose that five hardness readings have been obtained for a specimen, as follows: 43, 22, 27, 28, and 31. We first add together all of the readings, which total 151 in this case ($\Sigma x = 151$). We have 5 readings ("n" = 5), so we divide the total by 5 to calculate the mean (average), which is 30.2. Then we record the difference between each reading and the mean ($x -$ mean). We then square each difference ($x -$ mean$)^2$ and total the squares [$\Sigma(x -$ mean$)^2$], as follows:

Reading No.	x	Mean	$(x - \text{Mean})$	$(x - \text{Mean})^2$
1	43	30.2	+12.8	163.84
2	22	30.2	−8.2	67.24
3	27	30.2	−3.2	10.24
4	28	30.2	−2.2	4.84
5	31	30.2	+0.8	0.64

$n = 5$ Total, Σx, = 151 Mean = 30.2
Sum of Squares, $\Sigma(x - \text{Mean})^2$, = 246.80

We divide the sum of squares by "$n - 1$": 246.80/4 = 61.7
Taking the square root of 61.7 gives the standard deviation, "s":

$$s = (61.7)^{1/2} = 7.85$$

The above detailed procedure shows the concept of what standard deviation is—a modified average of the individual deviations on both sides of the mean. In actual practice, the above procedure is seldom used, as there are three simpler equations that are algebraically equivalent to the above and will give the same answer with less work. (See page 11, which refers to Section 11 of ASTM D 882).

The equation that the author has found to be the most convenient is:

$$s = [(\Sigma x^2 - (\Sigma x)^2/n)/(n - 1)]^{1/2}$$

where: s = the sample standard deviation,
x = an individual determination, and
n = the number of individual determinations.

Coefficient of Variation (ν or COV)

In many cases (but not always), the larger the mean, the larger will be the standard deviation of a set of data. Therefore, when comparing sets of data having widely differing means, it is often useful to use a tool called *coefficient of variation* (ν, also abbreviated as "COV"), which is the standard deviation (s) divided by the mean and expressed as a per cent:

$$\nu = 100\ (s/\text{mean})$$

As a rule of thumb, applied to most worldly functions, in general:

A. A COV of around 15% is about what one would usually expect for most sets of data from most applications.
B. A COV of 30% or more indicates an unsatisfactory amount of scatter. With high scatter, the calculated mean value is unreliable.
C. A COV of around 5% indicates an unusually tight scatter of data, which is highly desirable. With very tight scatter, the calculated mean value will be quite reliable, as far as precision is concerned.

Range

"Range" is the difference between the highest and the lowest value in any set of two or more determinations. While standard deviation is the most commonly used estimate of scatter, range is considered to be just as good for scanning data. However, standard deviation is more useful for calculating other statistical functions.

Outliers

In the above example, the value "43" is much higher than the other four determinations. Many people would automatically judge this value to be an outlier, discard it, and recalculate a new mean and standard deviation, using only the other four values. Other people would apply a statistical procedure and then automatically discard the value if the statistical procedure indicated that it was an outlier. Both actions are wrong! An item of data should never be discarded unless an identifiable cause is found to explain the deviation, such as:

A. Finding that the specimen was faulty in some way.
B. Discovering that there had been a sudden change in the testing environment during the test, such as an electrical power surge, a change in relative humidity or temperature, an unusual vibration in the room, etc.
C. Discovering that the specimen had slipped in the testing hardware.
D. Discovering that the specimen had been tested at a different crosshead speed from the other specimens.
E. Etc., etc. (Phase of the moon doesn't count here, though!).

PRECISION AND BIAS IN TEST METHODS

For the past several years, it has been a requirement that all ASTM test methods must include a section that addresses the precision and bias of the method. Unfortunately, many of the precision statements found in some methods are essentially useless and often misleading. For instance, Paragraph 8.9.1 of ASTM D 2103 states: "This method is capable of producing measurements with a maximum error of ±0.00254 mm (0.0001 in.)." At first reading, this statement creates a nice, reassuring impression. However, it does not contain anywhere near enough information for it to be statistically valid or to be used in any practical application:

A. What was the mean value to which this error tolerance applied? Without knowing that, we have no idea what the COV was.
B. Such a number must be based on a minimum of 30 individual items of data, and 100 items is a lot better! How many were included in the calculation of this number?
C. Does this refer to data within a single laboratory or to data generated by several laboratories? If several laboratories, how many laboratories participated in the study? The minimum acceptable number is six laboratories for generating data on between-laboratories reproducibility.
D. What was the probability factor used in calculating the cited figures? 75%?, 90%?, 95%, 99%, 99.9%?
E. Was more than one formulation tested? There should have been at least three formulations, showing a typical spread of mean values. The means and standard deviations should be reported for each formulation.

PRECISION AND BIAS SECTION IN ASTM D 882

By comparison, the Precision and Bias section in D 882 complies, in most part, with Appendix X3 of ASTM D 4968, "Guide to Using the Flow Chart for Annual Review of Standards Including the Model Precision and Bias Statement for Plastics," which was written by Committee D-20 and adopted as D-20 policy in 1991.

The precision data in D 882 are listed in Tables 3 thru 8; there is a separate table for each of the mechanical properties that is measured in this test method. The values in the tables were calculated from data obtained in interlaboratory test programs ("round robins") conducted and analyzed according to ASTM Standard Practice E 691, "Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method." In D 882, the data are derived from two separate interlaboratory test programs, as explained in Sections 13.1 thru 13.2.

S_r and S_R

For each material tested in the round robin, the fundamental values that are calculated are the within-laboratory standard deviation (S_r) and the between-laboratories standard deviation (S_R). For each material:

A. The s_r is calculated by pooling the standard deviations of the test results that were obtained within each laboratory:

$$s_r = [[(s_1)^2 + (s_2)^2 + \cdots + (s_n)^2]/n]^{1/2}$$

The value of s_r for each material is listed in the fourth column of Tables 3 thru 8.

B. The S_R is a combination of both the S_r and the variability between the mean values among the laboratories. Expressed another way, it is the between-laboratories reproducibility, expressed as standard deviation:

$$S_R = (S_r^2 + S_L^2)^{1/2}$$

where

S_L is the standard deviation of laboratory means.

- (S_L is calculated during the analysis of the data, as directed in ASTM E 691. However, it is not shown in the tables of precision data, as it is not used directly in making comparisons of test results.)

The value of S_R for each material is listed in the fifth column of Tables 3 thru 8.

From the values of S_r and S_R and the means (which are shown in the third column of Tables 3 thru 8), it is easy to calculate the within-labs coefficients of variation (ν_r) and the between-labs coefficients of variation (ν_R). COV is one good measure of how good the precision of a test method is, using the general guidelines given previously.

Different Materials

As stated, each table lists precision data for each different material that was measured in the interlaboratory test program. Many other styles of precision statement report only the average of the data from all of the materials together. This makes the information largely meaningless for plastics because for any given test method, the scatter of data for one material is very likely to be considerably different from the scatter for another material. For instance (referring to Table 5):

A. For PP, S_R is 0.41, which gives $\nu_R = 12\%$ when divided by the mean value of 3.5.

B. For LDPE, S_R is 3.41, giving $\nu_R = 34\%$ when divided by the mean value of 10.0.

In this example, both the S_R values and the ν_R values differ greatly between the two materials. See, also, the COV values for ASTM D 3894 in Chapter 4.

As another example, note that LDPE shows the smallest variability of all the materials in regard to yield stress (Table 4); but it shows the greatest variability in regard to yield elongation (Table 5).

"r" and "R" Values (formerly "I_r" and "I_R")

The last two columns of Tables 3 thru 8 list an "r" and an "R" value for each material. (In the older precision statements that were written in this format, the symbol "I_r" was used instead of "r," and "I_R" was used instead of "R".) Each "r" and "R" value constitutes a critical interval, [or least significant difference (LSD)] for comparing two test results.

"r" is the within-laboratory critical interval between two test results:

$$r = 2.8\ X\ S_r$$

"R" is the between-laboratories critical interval between two test results:

$$R = 2.8\ X\ S_R$$

Anyone trained in statistical procedures may use S_r and S_R to calculate many other statistical applications of the data.

The basic core of the precision statement, explaining how to apply the "r" and "R" values, is contained in Sections 13.3 thru 13.3.3 of D 882. However, in August of 1996, Subcommittee D-20.13 on Statistical Procedures recommended a new, slightly different model for these sections, to be used in all future revisions of D-20 test methods, as follows:

> ##.2 *Concept of "r" and "R"*—If S_r and S_R have been calculated from a large enough body of data, and for test results that were averages [[or medians or other functions]] from testing __ specimens for each test result, then:
>
> ##.2.1 *Repeatability:* Two test results obtained within one laboratory shall be judged not equivalent if they differ by more than the "r" value for that material. "r" is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the same day in the same laboratory.
>
> ##.2.2 *Reproducibility:* Two test results obtained by different laboratories shall be judged not equivalent if they differ by more than the "R" value for that material. "R" is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories.
>
> ##.2.3 Any judgment in accordance with ##.2.1 or ##.2.2 would have an approximate 95% (0.95) probability of being correct.

Each "r" value constitutes a critical interval for comparing two test results generated by the same testing laboratory. For example, if we had test results of 1.4×10^3 psi and 1.55×10^3 psi from the same laboratory:

$$1.55 \times 10^3 \text{ minus } 1.40 \times 10^3 = 0.15 \times 10^3$$

If the critical interval for "r" has been found to be 0.14×10^3, then we may judge that the second test result is significantly higher than the first, and there is a 95% probably that this judgment is correct.

Similarly, each "R" value constitutes a critical interval for a between-laboratories comparison. For example, if we had a test result of 1.50×10^3 psi from one laboratory and a test result of 1.86×10^3 psi from another laboratory:

$$1.86 \times 10^3 \text{ minus } 1.50 \times 10^3 = 0.36 \times 10^3$$

If the critical interval for "R" has been found to be 0.37×10^3, then we should not judge that there is any significant difference between the two test results.

Most people are greatly surprised when they realize how large are the critical intervals that the "r" and "R" values represent—especially the "R" values. Sometimes, they are so large that they render the test method practically useless for measuring meaningful differences.

The critical interval approach is not the only way to express precision data, but it is one of the easiest ways to provide a precision statement that is understandable to the layman and still satisfies the rigorous requirements of statisticians.

Cautionary Advice

Note 19 contains important cautionary advice to prevent unwarranted application of the precision data. The precision data in the method are valid only for the specific samples used in the interlaboratory study, and they should not be rigorously applied to other materials or to the same generic materials manufactured by other producers.

How the Interlaboratory Study Was Carried Out

The items of information in Sections 13.1 thru 13.2 are of vital importance in establishing the reliability of the whole precision statement because they document how the interlaboratory study was carried out. Without the following information, it is impossible to know how reliable are the results:

A. Number of laboratories which participated.
   1. Ideally, there should be thirty or more laboratories, but it is rare to find that many who are willing to participate.
   2. Twenty laboratories will produce quite reliable data; twelve is still an acceptable number; as the number of laboratories decreases below ten, the reliability of between-laboratories data starts to fall off precipitously.
   3. Six is the absolute minimum number of laboratories—with only six, the reliability of between-laboratories data will be barely acceptable.

B. Number of specimens tested by each laboratory for each material. The total number tested by all laboratories for each material should be at least 30, in order to ensure the reliability of within-laboratories data.

C. Where the specimens were prepared (D 882 is deficient in not reporting this). If the specimens were all prepared at the same laboratory and then distributed to the individual laboratories for testing, the precision data will not include the component of variability that would be due to differences in specimen preparation at the various locations; this is likely to be one of the largest components of the between-laboratories variability.

PROBLEM SOLVING

This last item is for the benefit of people in production, engineering, research, product development, testing, quality control, technical service, etc, etc. The chapter would not be complete without some advice about problem solving.

The author was privileged to take an excellent course in problem solving several years ago under the direction of Harmon Bayer and Gayle McElrath. Two of Mr. Bayer's pearls of wisdom were:

A. "Statistics are a tool for making judgments in the face of uncertainty."
B. "Management is the art of making decisions—some of which are correct."

In analyzing data, one must guard against the following types of error:

A. If one studies data and decides that two or more things are different, when there is really no significant difference between them, one has committed the Type I error.
B. If one studies data and decides that there are no significant differences, when things really are different from each other, one has committed the Type II error.

Richard Freund, former chairman of ASTM Committee E-11 on Statistical Procedures, used to refer to a

- Type III Error: "The mathematically precise solution to the wrong problem."

That is not just a whimsical statement. In far too many cases, people assume that they know what the problem is and spend a lot of time and money in fruitless searches for solutions. Often, they never find the proper solution because they are trying to solve the wrong problem. So, the first step in finding solutions is to find out what the source of the problem really is.

The first piece of advice is to "soak" in the environment of the problem. That means to take time to observe and evaluate all of the circumstances and factors that might be involved. This might include spending several days listening to and watching everything happening near a machine where trouble seems to be occurring. This may often disclose that the real source of the problem is different from what it was thought to be.

The second piece of advice is to enlist the help of a person who is trained in statistical procedures, if one is available to you, provided that he or she:

A. is willing to work on the problem on a "getting the hands dirty" basis—"soaking," along with you, in the environment of the problem, as opposed to trying to solve the problem from the comfort of his or her office, and
B. knows the proper approaches for investigating industrial problems and the statistical techniques for analyzing them.

Too many people are unwilling to accept statistical approaches to solving problems because they think that those approaches and analyses are too cumbersome and too time-consuming. As a result, those people often spend far more time and money in "patchwork" attempts at finding solutions than they would have if they had followed a carefully designed overall plan.

A classic example of such thinking: Years ago, the U.S. automobile companies rejected Dr. Deming's suggestions on statistical quality control procedures. The Japanese then invited Dr. Deming to present his ideas. They adopted his ideas, and they "ate our lunch" with the excellent quality of their products. In the last few years, U.S. companies have started catching up, after they finally adopted Dr. Deming's ideas.

BIBLIOGRAPHY

The Cartoon Guide to Statistics, Larry Gonick and Woolcott Smith, Harper Perennial; 1993.

Fundamental Concepts in the Design of Experiments, Charles R. Hicks, Holt, Rinehart and Winston, 1982.

Engineering Statistics, Albert H. Bowker and Gerald J. Liebermann, Prentice-Hall, 1959.

A Primer of Statistics for Non-Statisticians, by Abraham N. Franzblau, Harcourt, Brace and Company, New York, 1958.

3

Measuring Mechanical Properties of Plastics

by Alvin J. Flint, Jr.[1]

Introduction

MEASURING A MECHANICAL PROPERTY consists of measuring the response of a test specimen to an applied force. The four modes of mechanical strength properties are discussed in this chapter. The most important aspects of measuring tensile, compressive, flexural, shear, impact, surface, creep, fatigue, and weathering properties are reviewed.

In order to understand the mechanical response of plastics to an applied force, it is necessary to understand the concept of viscoelastic behavior. This is explained in its most basic concepts, along with examples of its influence.

This chapter also discusses safety, equipment, stress/strain curves, modulus, and specific cautions concerning the most common tests, as well as computerization and automation of testing equipment.

UNIVERSAL TESTING MACHINE (UTM)

We usually would start a chapter with guidelines concerning safety in order to emphasize its importance to all of us. However, because we will be referring to universal testing machines in the "Safety" section, it is well to start the chapter by describing what a universal testing machine (UTM) is. It is called a universal testing machine because it may be used to perform several different types of mechanical property tests—usually tensile, compressive, flexural, or shear. Although there are horizontal testing machines available, most are vertical machines; our description will apply to the vertical type, but most of the same principles apply to the horizontal type.

A UTM consists of two vertical load-bearing columns, on which are mounted both a fixed horizontal crosshead bar and a movable horizontal crosshead bar. The columns are usually of a worm gear construction to position the movable crosshead. There also must be:

A. Provision for mounting various kinds of test fixtures (generally referred to as "hardware") at the centers of both the fixed crosshead and the movable crosshead. When mounting hardware, it should be affixed to each crosshead through a universal joint or some other device, in order that the applied force will pass through the center of the specimen.
B. A motor for driving the worm gears (and thus the crosshead) at a constant speed. Allied with this, there must be a mechanism for setting the speed of the movable crosshead to any designated constant rate from about 0.025 millimeters (0.001 in.) per minute to 50 mm (20 in.) per minute.
C. A load cell or a weighing system to sequentially read the varying force that is exerted on the specimen as the crosshead moves.
D. For flexural, compressive, and shear tests—a means for reading the linear movement of the crosshead.
E. For tensile tests—a system for reading the electrical signal from an extensometer, which is an instrument in contact with the specimen. It produces an electrical signal in proportion to the stretching of the specimen. (Extensometers are discussed, in detail, later in this chapter under "Tensile Testing.")
F. A chart recorder (as described further on in this chapter) or some other recording device, such as a computer monitor.
G. Appropriate controls for all of the above functions.

UTM Sizes

The size of a UTM is described by the maximum load rating of the columns—there are machines with ratings from a few grams force to 44.5 mega Newtons (ten million pounds force) or possibly more. The larger the size, the greater will be the cost, of course. ASTM mechanical property tests for most plastics may be carried out on 22.25 kN (5000 lbf) machines.

However, the author has known of specimens of glass-reinforced thermosetting polyester, 190 mm (3/4 in.) thick, that had been cut from the wall of a storage tank and that could not be broken, in tension, in a machine rated at 133.5 kN (30,000 lbf). The author has been informed that laminates have been tested to 800 kN (180,000 lbf).

SAFETY

Although physical testing laboratories generally are not particularly dangerous workplaces, several basic safety rules must still be followed. It is not enough for employers to simply *have* written safety rules—they must be enforced. Employers who do not rigorously enforce their own safety rules, as well as safety rules mandated by outside sources, leave themselves open to possible lawsuits. Among the rules that must be enforced are:

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[1] Formerly, supervisor of the Physical Measurements Laboratory, ICI Polyurethanes, West Deptford, NJ. Now retired. Present address: 109 Hoiland Drive, Shipley Hts., Wilmington, DE 19803-3227.

A. All persons entering the laboratory must wear safety glasses. There is always the possibility that tiny pieces from specimens may be propelled about when the specimen breaks; this is especially true of impact tests, but may also be true of tensile and other tests.
B. Safety shoes or foot shields must be worn whenever lifting or carrying any item heavy enough to cause injury to the feet if dropped. Most of the hardware items for UTMs qualify here, along with calibration weights and weights used in creep testing.
C. UTMs have safety stops that will limit the travel of the movable crosshead. Check the position of the safety stops each time the machine setup is changed and at the beginning of each shift.

CAUTION

In compressive testing, if the crosshead is allowed to travel too far, the force generated may be sufficient to wreck the load cell—an expensive mistake!

D. When using powered saws, use all proper guards and guides. Use push rods when it is necessary to work close to the saw blade.
E. Beware of pinch points that may catch a finger in various equipment geometries.
F. Check periodically for exposed electrical wiring or connections. Have them repaired immediately.
G. Have a preventive maintenance program for all equipment, and enforce it.

ACQUIRING TEST EQUIPMENT

When one learns the price that an equipment supplier charges for an item of test equipment, it is often tempting to try to save a few dollars by building the item yourself. In general, the author recommends against it. When buying an established piece of equipment from a reputable supplier, you may expect to get equipment that has been standardized in its construction and has had the "kinks" worked out of it. In the long run, this equipment will be more economical and reliable; also, it will probably provide less scatter of test data, both within-lab and between-labs (see Chapter 2). By comparison, with home-made equipment you will probably waste an enormous amount of time (which is money) before you are able to get it standardized and operating properly.

Equipment for Specimen Conditioning

Nearly all test methods for plastics require conditioning of specimens at 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5% RH before testing. For referee testing, the tolerances are tighter: ±1°C (±1.8°F) and ±2% relative humidity. Highly specialized equipment and installation are necessary in order to hold to these tolerances. If you are going to outfit a laboratory for proper conditioning of specimens, do not simply turn the job over to your engineering department and let them hire the local air conditioning man. If you do, you will probably never get the system to work within the stated tolerances. Instead, track down an experienced supplier of the proper equipment and pay his higher price to have the job done right.

Schedule Calibrations and Maintenance

Remember from Chapter 2—there are almost no material standards for plastics. This makes regular equipment calibration even more important than with some other materials. Therefore, for gages, micrometers, balances, and each of the other items of equipment in the laboratory, it is important to establish, and enforce, schedules for periodic calibrations, as well as for preventive maintenance. If they are not scheduled and enforced, they will not get done.

CLASSIFYING MECHANICAL STRENGTH PROPERTIES

Any specific mechanical strength property may be classified as follows:

A. In terms of the direction in which strain is induced in a specimen when a force is applied to it. The four modes of mechanical strength properties are tensile, compressive, bending, and shear.
B. In terms of the speed at which the force is applied:
    1. *Static tests* involve loading the specimen at speeds from 5 to 51 millimeters (0.2 to 20 inches) per minute.
        a. These tests are typically run on UTMs.
    2. *Impact tests* generally involve loading speeds at, or above, 203 meters (8000 inches) per minute.
    3. Creep and fatigue tests involve reduced loadings over long periods of time.

MAKING MEASUREMENTS WITH UTMS

Generally, measurements of applied force (load) and deformation of the specimen are not made directly on the specimen. Instead, electrical signals are sent to a chart recorder or to a computer.

Today, chart recorders have been largely replaced by computers and their monitors. However, it is necessary to understand how data would be read from a chart recorder in order to understand much of what follows in this chapter.

Load/Deformation Curves

A chart recorder presents, on ruled graph paper, a curved line representing two components—force on the Y-axis versus deformation on the X-axis, as shown in Fig. 3-1.

- Before running a test, the equipment operator must set up the chart recorder so that he/she knows the quantity that each division of the chart paper represents, on both the X-axis and the Y-axis. (As a part of doing so, the operator must be guided by instructions furnished by the manufacturer of the chart recorder.)

A. The signal representing the force comes from the load cell (or other weighing system) in the UTM. It causes the re-

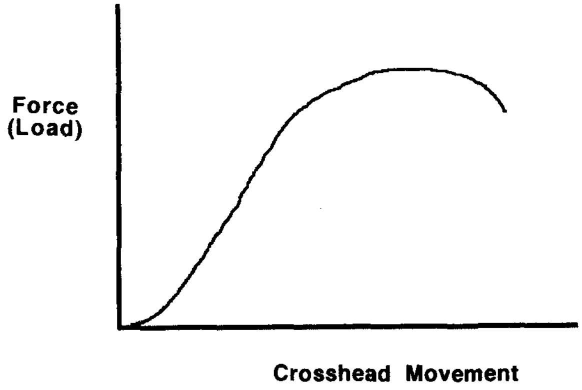


FIG. 3-1—Force versus crosshead movement.

corder pen to move in the direction of the Y-axis, proportionately to the force signal.

B. The component representing deformation may be based either on a direct electrical signal from a monitoring device or on movement of the crosshead of the UTM:
    1. *Direct electrical signal*: for compressive tests, it is necessary to use the electrical signal from a compressometer. For tensile tests, it is usually necessary to use the electrical signal from an extensometer. These are instruments that are mounted in contact with the specimen. When using a direct electrical signal, the chart recorder must be set up in "XY" drive. Again, the operator must be guided by instructions furnished by the manufacturer of the chart recorder.
        - In no case should crosshead movement be used to calculate elongation or modulus for a dog-bone tensile specimen. (These terms will be explained later in this chapter.)
    2. Measuring crosshead movement: This is usually done with the X-axis set up in "time" drive. This simply means that:
        a. For a strip chart, the rate of movement of the chart paper is set by the machine operator.
        b. For a single sheet chart, the rate of movement of the pen along the X-axis is set by the machine operator.

    By knowing the rate of movement of the crosshead, as well as the rate of movement of the paper (or the pen) along the X-axis, one may calculate how much crosshead movement is represented by each division along the X-axis scale of the chart.

TENSILE TESTING

To carry out a tensile test, the specimen is gripped at each end and pulled apart. Because the grips (jaws) hold the specimen by exerting a clamping force, they always inflict some damage to the specimen. In many cases, this damage will cause specimens to break at or near one of the jaws. In order to minimize this, most tensile specimens are designed to have their center sections narrower than the ends, as shown in Fig. 3-2. These are called "dogbone" specimens.

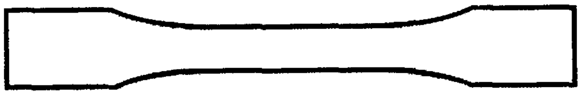


FIG. 3-2—Dogbone tensile specimen.

[In preparing specimens for almost all tests, it is very important that the surfaces of each finished specimen be free from nicks or cracks in order to minimize the chances of premature failure during testing, which will generate false facts (see Chapter 2).]

In preparing tensile specimens, it is also important to shape them carefully in the "shoulder" areas so that the center section blends smoothly into the wider end tabs. The curvatures of the specimens shown in ASTM D 638, Test Method for Tensile Properties of Plastics (described below), are specially designed to accomplish this and thus to reduce the chance that the specimens may break at a shoulder.

The opposite edges in the reduced portion must be as parallel as possible. Also, the side-to-edge corners must be as near to 90° as possible in order to be able to measure the width accurately.

Typically, specimens of thermosetting plastics must be rough-sawed and then shaped on some type of milling machine or grinder that used a template for a guide. Typically, for thermoplastics, most specimens (except those for Izod and Charpy impact, described further on) may be molded.

ASTM D 638 is the test tensile method used most often for plastics. D 638 requires that results be discarded for specimens that break outside the "gage length," which will be explained under "Measuring Elongation and Strain," later in this chapter.

Standard Tensile Specimens

D 638 lists dimensions and tolerances for five different tensile specimens. All have dogbone shapes, but all are of somewhat different size and geometry:

A. Type I is 19 mm (0.75 in.) wide at the ends and 165 mm (6.5 in.) long. The center portion is 13 mm (0.5 in.) wide. It should be used whenever possible for rigid and semirigid plastics up to 7 mm (0.28 in.) thick.

B. Type II is narrower in the center than Type I. It is intended for materials that exhibit a high incidence of breaks in a shoulder or a jaw region when tested with Type I specimens.

C. Type III is wider and longer than Type I. It must be used for all materials with a thickness from 7 to 14 mm (0.28 to 0.55 in.).

D. Type IV has wide end tabs 19 mm (0.75 in.) and is only 6 mm (0.25 in.) wide at its center. It should be used for comparisons between nonrigid and semirigid materials.

E. Type V is only 63.5 mm (2.5 in.) long, and the center portion is only 3.18 mm (0.125 in.) wide. It is intended for use where only limited material is available for evaluation, or where a large number of specimens are to be exposed in a limited space, as in thermal and environmental stability tests, for instance. It is limited to material having a thickness of 4 mm (0.16 in.) or less.

The question obviously arises as to whether these different sizes of specimens will give validly comparable results. The author once designed and statistically analyzed an experiment to investigate this, fully expecting that there would be significant differences in results. Specimens of all of the five different types were cut from a single panel of sheet molding compound. Must to the author's surprise, the results showed no significant differences. This material contained fiberglass reinforcement chopped into strands 13 mm (1/2 in.) long and was a material having very low elongation. As in so many other aspects of plastics, and with the physical testing profession, it is possible that a similar experiment on other materials might give quite different results.

Another tensile method should be mentioned—D 5083, Test Method for Tensile Properties of Reinforced Thermosetting Plastics Using Straight-Sided Specimens. Its title is self-explanatory. While many people claim good success with it, the author found that it gave results that were significantly different from results obtained with D 638 Type I specimens, in one of the initial studies of the method.

Measuring Elongation and Strain

As previously described, a UTM includes a means for recording a curve of force versus crosshead movement or a curve of force versus specimen deformation. In this case of tensile testing, we want to measure the elongation (stretching) of the specimen rather than crosshead movement. Because the specimen is not straight-sided, it will not stretch uniformly throughout its length. It is important that we measure how much it stretches in the straight-sided section. As shown in Fig. 3-3, we do this by establishing, within the center section of the specimen, initial reference points "A" and "B" that establish a known "gage length." The gage length does not need to be any certain value, but it is vitally important that we know very accurately what its actual value is. The longer it is, the more precise will be the calculated result, as we shall see below. Therefore, with Types I, II, or III specimens, we usually use a gage length of two inches, centered in the narrowed section.

Referring to Fig. 3-3, the top of the specimen is held stationary by the jaw attached to the fixed crosshead; the bottom is pulled down by the jaw attached to the moveable crosshead. (In some UTMs, this geometry is reversed.) As this occurs, the initial points "A" and "B" both shift, becoming the continuously changing points "A" and "C." We need to measure the distance from "A" to "C" continuously throughout the test in order to be able to calculate the elongation, and subsequently the strain, at any point on the chart recorder curve:

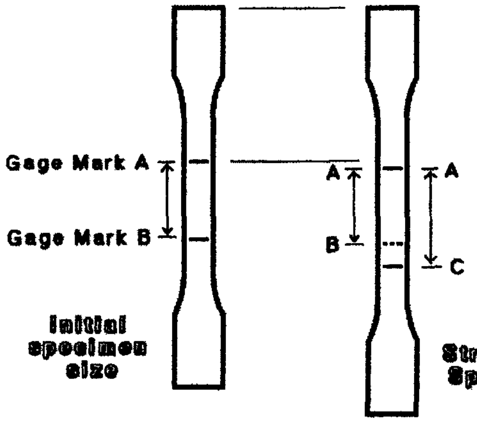


FIG. 3-3—Extension of gage marks on a tensile specimen.

$$\text{Elongation} = \text{stretch (or extension)} = BC = AC - AB$$

$$\text{Tensile strain } (\varepsilon) = \text{elongation/gage length} = BC/AB$$

Because the distance, *BC*, is in millimeters (or inches) and the distance *AB* is also in millimeters (or inches), the units of strain are mm/mm (or in./in.). This, in fact, is how strain is expressed in compressive testing, flexural testing, and shear testing. However, in tensile testing, it is customary to report per cent elongation, rather than strain:

$$\text{Per cent elongation} = 100 \times \text{tensile strain} = 100\ (BC/AB)$$

Extensometers

For measuring elongations up to approximately 50% or for measuring tangent modulus, what is needed is a device called an *extensometer*. (The term "modulus" will be explained later in this chapter under the heading "Properties Measured from Stress/Strain Curves.") The extensometer has line contact surfaces ("knife edges") that clamp across the specimen at Points "A" and "B" to establish the gage length (see Fig. 3-3). As these points shift, the change in the distance between them is translated by the extensometer into an electrical signal. For a tensile test, it is this signal, rather than a signal from crosshead movement, which must be transmitted to the chart recorder. The recorder must be set up in "XY" drive, rather than time drive.

If molded tensile specimens are used, one must be sure always to mount the extensometer on the same portion of the specimen. Otherwise, elongation and modulus results will vary, depending on where the extensometer was mounted, even within the straight-sided part of the specimen.

In no case should crosshead movement be used to calculate elongation or modulus for any type of dog-bone specimen. There are at least two factors that invalidate any such data based on crosshead movement:

A. The specimen does not elongate uniformly; the elongation in the center section is significantly more than the elongation in the wider portions.
B. There is always some movement of the specimen in the jaws while the jaws are seating themselves firmly enough to grip the specimen.

Many people violate the above rule in order to avoid the cost of an extensometer. All they are doing is amassing false facts, as there is no dependable correlation between crosshead movement and elongation when using a dog-bone specimen! The error is especially magnified when trying to measure tangent modulus by crosshead movement.

- The author has personally conducted experiments on reinforced thermosetting polyester specimens to try to establish some such correlation, for obvious reasons. He had no success. In addition, over a 24-year period he has talked with many other testing professionals who have tried similar experiments, all without success, with one lone exception. There is one laboratory who claimed success by calculating what they called a "theoretical gage length" when testing a specific polymer. They offered their technique to ASTM Committee D-20 on Plastics for consideration. At least three members of D-20 tried the technique on other polymers, and none were successful.

Types of Extensometers

There are four general types of extensometers. In the type most often used in the plastics industry, an internal strain gage regulates its outgoing electronic signal. This type is light enough so that most rigid specimens will support its weight without serious distortion. However, for thin or inherently flexible materials, the extensometer may need to be suspended independently, in such a way that its knife edges contact the specimen but the specimen does not support any of the weight of the extensometer.

The second type of extensometer uses an LVDT (linear variable differential transducer). This is a reverse solenoid wherein the movement of the core varies its electronic signal, instead of vice versa. It is much heavier than a strain gage extensometer, and thus it is much more likely to distort a thin or flexible specimen.

The third type, used for highly extensible material, does not generate a continuous signal to represent the extension of the material as the other three types do. (Therefore, the chart recorder is set on time drive when using this type.) It pulls a plastic tape through a signal generator, which initiates small "blips" at predetermined intervals of extension along the load/time curve.

The fourth type is an optical extensometer, wherein there is no contact with any part of the specimen. Precise gage marks must be ink stamped on the specimen. The extensometer has two optical heads, each of which focuses on a gage mark. As the gage marks shift, the optical heads follow the marks and generate an electronic signal that represents the distance between them. Somewhat offsetting the obvious advantages of this system are three factors:

A. First, the system is five to ten times more expensive than either the strain gage or the LVDT extensometers.
B. Second, with highly extensible materials, the stamped gage marks widen as the material stretches, and the optical heads have difficulty in tracking them.
C. Third, for materials having very low elongation, the system may not have sufficient sensitivity.

Capacities of Extensometers

Extensometers are available with various gage lengths, and with various ranges, going as high as 1000%. The range should be chosen according to the material being tested. For instance, for materials having very low elongations, such as sheet molding compound (SMC) and some other reinforced thermosets, use an extensometer with a range of 0 to 5% or 0 to 10%, rather than 0 to 50%.

For High Elongations

We have emphasized the importance of using an extensometer to measure elongations less than 50%. However, for materials that give elongations higher than 50%, one may use the following procedure for measuring elongations (but for tangent modulus, an extensometer is still necessary):

A. Apply two gage marks on the specimen. It is imperative that the distance between the gage marks be known precisely.
B. As the specimen elongates during the test, hold alongside the specimen a ruler that is clearly marked in millimeters (or in tenths of an inch).
C. Keep the index of the ruler aligned with one of the gage marks. At the other gage mark, read the ruler value continuously, estimating to 0.25 mm (0.01 in.). Be prepared to record the position of this gage mark at the instant the specimen breaks.
    1. An alternate procedure is to attach a thin strip of graph paper to the upper gage mark with tape or a paper clip and use it as the measuring reference.

REPEAT WARNING!—DO NOT use the above procedure for measuring tangent modulus!

Slippage of Knife Edges

There is a problem that sometimes occurs with materials having a slippery surface—the knife edges may slip and not track the original gage mark reference points properly. This is usually evident by strange shifts in the load/extension curve. The most satisfactory solution, if the knife edge clamping mechanism has enough space to allow it, is to mount the extensometer so that the knife edges bear on an edge of the specimen, instead of on a side. Another treatment is to attach small patches of cloth adhesive tape to the specimen at the points where the knife edges make contact.

Testing Film

When testing film, it is not practical to prepare dogbone specimens. ASTM D 882, Test Method for Tensile Properties of Thin Plastic Sheeting, allows crosshead movement to be used to measure elongation of straight-sided strips of polymeric film. The method should be read very carefully, however, as grip slippage can be a major problem.

MORE ON LOAD/DEFORMATION CURVES

The above discussion has been specific to tensile testing. The following will apply to all four modes of mechanical property testing.

Part of the chart recorder system is a multiposition switch for selecting the "full-scale load" to be used for recording the force (load) during the test. The full-scale load is selected by the machine operator. Selecting the full-scale load simply sets the load value for the top of the force scale on the recorder chart. The reason for this is:

A. To be sure that the force/deformation curve does not run off the top of the force scale.

B. To be sure that the force/deformation curve reaches into the upper part of the chart, in order to enable one to read force values from the lower parts of the curve more precisely.

Figure 3-4*a* shows a curve that has been recorded with one inch of chart equal to 50 Newtons (11.25 pounds-force) on the force (load) scale and 1 in. of chart equal to 0.2 mm (0.008 in.) on the deformation scale. Figure 3-4*b* shows the same force/deformation spectrum plotted with 1 in. of chart equal to 100 Newtons (22.5 lb-f) on the force scale and 1 in. of chart equal to 0.1 mm (0.004 in.) of the deformation scale. Note that neither the force scale nor the deformation scale take into account the size or geometry of the specimen. This illustrates that it is usually impractical to compare data directly in terms of load and deformation. The problem is readily solved by converting the values of load and deformation to values of "stress" and "strain," respectively, as will be explained in the next section.

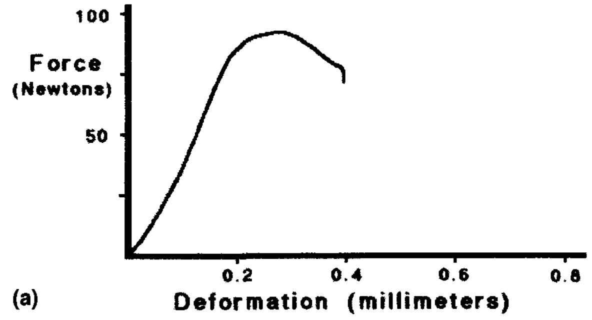


FIG. 3-4*a*—Force versus deformation, 100 N full scale.

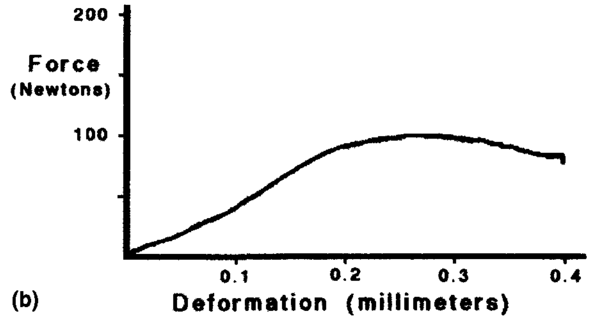


FIG. 3-4*b*—Force versus deformation, 200 N full scale.

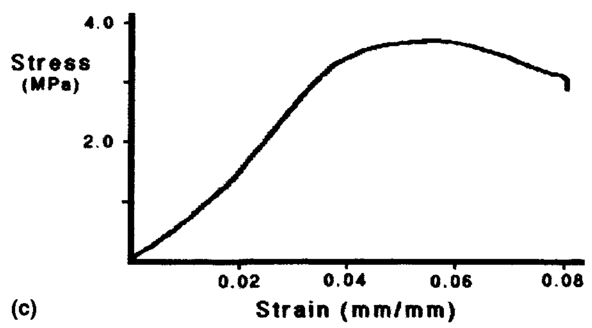


FIG. 3-4*c*—Stress versus strain.

STRESS/STRAIN CURVES

Before conducting tensile, compressive, or flexural tests, one must carefully measure and record the width and the thickness of each specimen, using either a micrometer, vernier calipers, or a dial gage. For a tensile test or a compressive test, calculate the cross-sectional area of each specimen:

$$\text{Cross-sectional area} = \text{width} \times \text{thickness}$$

Any chosen point on the load/deformation curve may be converted to a stress/strain value as follows:

A. stress is calculated by dividing the load (W) by the cross-sectional area (A):

$$\text{Stress } (\sigma) = W/A$$

1. *English units*: In the English system of measurements, if the load is in pounds and the specimen dimensions are in inches, the unit of stress is then pounds per square inch, abbreviated "psi."
2. *SI units*: The SI system is not the same as the metric system. The SI system is derived from the metric system. In general, their units are the same, but there are some important differences. For instance, silly as it seems, the SI system does not allow the use of the centimeter as a unit.
   a. The unit of force (load) is Newtons.
   b. Specimen dimensions are in millimeters (mm) or meters (m).
   c. The unit of stress is the Pascal (Pa), which is one Newton per square meter. Because it takes approximately 6895 Pa to equal 1 psi, prefixes must nearly always be used when citing stress or modulus values in the SI system:

1 psi = 6895 Pascals
= 6.895 kPa (kiloPascals);
1000 psi (1 Kpsi) = 6,895,000 Pascals
= 6.895 mPa (megaPascals);
(1 Kpsi is also known as a "Kip.")
1,000,000 psi (1 Mpsi) = 6,895,000,000 Pascals
= 6.895 gPa (gigaPascals).

You will see these units throughout this chapter because ASTM requires their use in all of their publications.

B. Strain (ε) = elongation/gage length

$$\text{Strain } (\varepsilon) = BC/AB$$

The units for strain are millimeters per millimeter (inches per inch).

Comparing Data

In Fig. 3-4*c*, the data from Fig. 3-4*a* have been used to replot the force/deformation spectrum in terms of stress and strain. As can be seen, values from this curve could easily be compared to other data expressed in terms of stress and strain. We will discuss the specific data points that are generally of most interest in a later section.

Toe Compensation

In tensile, compressive, and flexural testing, it is common for a non-linear region, called a "toe" (or "foot"), to appear at the start of the load-deformation curve, as shown in Fig. 3-5. One of the possible causes may be slack in various parts of the "loading train."

- The loading train consists of all of the hardware through which the force is transmitted through the specimen from one crosshead to the other. It includes the pull rods, to which are attached the jaws or other fixtures that hold the specimen. In most cases, universal joints should also be installed in the loading train in order to align the specimen automatically, so that the force will be applied through the centerline of the specimen, as nearly as possible.

In some modes of testing, the toe may be partly due to the sides of the specimen being slightly non-parallel, causing uneven seating on the test fixture. In many cases, there may not be any apparent reason for the toe. For example, a toe may occur in tensile testing even when using an extensometer.

Before calculating modulus or elongation values from any load/deformation curve, it is necessary to make a correction to the curve to eliminate the effect of this toe, as follows [see, also, the appendices (which should be changed to Annexes) to D 638, D 695, or D 790):

A. If there is a linear portion of the curve, draw a straight line through the linear portion and extend the line down to the baseline of the chart, as shown in Fig. 3-5.
B. If there is no linear portion in the curve, there will be an inflection point. The inflection point is the point at which the curve of the toe stops bending upward and starts bending laterally. Draw a straight line through the inflection point and extend the line down to the baseline of the chart as shown in Fig. 3-18.
C. The intersection of the straight line and the baseline is the "zero deformation point." All measurements along the "X" axis must be made from this point—not from the start of the toe.

COMPRESSIVE TESTING

Compressive testing denotes a squeezing force distributed through the specimen. For materials 3.2 mm (0.125 in.) and thicker, the standard specimen is a right prism whose length is twice its principal width, as shown in Fig. 3-6*a*.

For materials less than 3.2 mm (0.125 in.), the specimen is shaped as shown in Fig. 3-6*b*. See ASTM D 695, Test Method for Compressive Properties of Rigid Plastics, for specific details. When using the thin specimen, it must be clamped in a metal jig (see Fig. 3-6*c*) to prevent sideways buckling during the test. The thin specimen may conveniently be cut from the center of an ASTM D 638 Type I tensile specimen.

For optimum results, it is best to have the force distributed as uniformly as possible against the ends of the specimen. Three things are necessary to accomplish this:

A. Care should be used during specimen preparation to be sure that the opposite ends are as nearly parallel as possible, and the opposite sides, as well. (It is almost impossible to achieve absolute parallelism.) In the case of the right-prism specimens, all the corners should be as close to 90° as possible.
B. The force must be transferred through the specimen by two rigid, flat metal members bearing on the ends of the specimen. The flat members must be mounted in such a way that at least one of them will be self-aligning in the load train. This is to ensure that both ends of the specimen will automatically make maximum contact with the flat members.

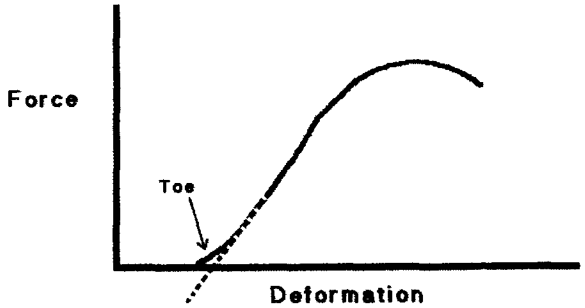


FIG. 3-5—Toe compensation.

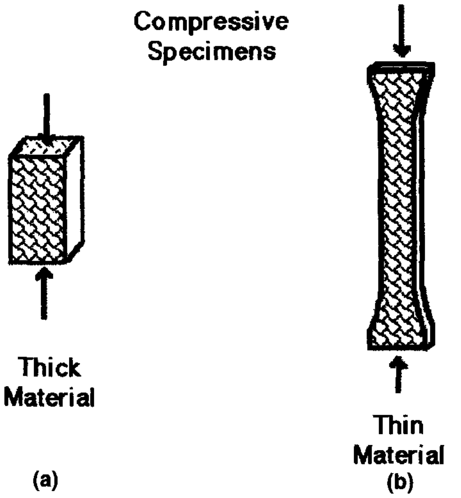


FIG. 3-6*a*,*b*—Standard compressive specimen. Thin compressive specimen.

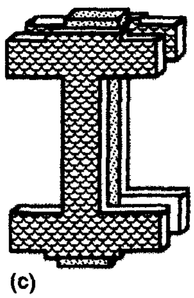


FIG. 3-6*c*—Anti-buckling jig for thin compressive specimens.

C. The specimen must be placed precisely in the center of the plates to achieve axial loading through the specimen

   1. One type of compressive hardware looks somewhat like a cage, with the plates forming the top and bottom. The specimen must be centered in the plates, which are pulled toward each other to exert compressive force. This hardware must be mounted with universal joints above and below it in the loading train (which is set up as for a tensile test) in order to assure maximum contact of the specimen against the plates.
   2. Another type of compressive hardware has one of the plates mounted on a ball-and-socket device in order to achieve automatic adjustment of the bearing surfaces. The author regards this type as being less satisfactory because some degree of friction along the contact surfaces of the ball and socket is unavoidable. Also, no universal joints may be used to ensure axial loading when using this hardware.

It is no longer permissible to measure the deformation of solid compressive specimens by monitoring crosshead movement. Although it was formerly allowed, the 1991 revision of D 695 removed this option—D 695-91 requires the use of a compressometer for measuring deformation. A compressometer is the converse of an extensometer. Like an extensometer, it must be attached to the specimen, and its electrical signal, rather than crosshead movement, must be transmitted to the chart recorder to provide the curve component in the direction of the "X" axis on the chart. The chart recorder must be set up in "XY" drive, rather than time drive.

Compressive Strength

Reporting compressive strength can be a little tricky:

A. If a sudden failure of the specimen occurs, as is likely with rigid materials, the specimen will usually exhibit a rupture that is diagonal to the axis of the specimen, and the force-deformation curve will drop off sharply. In such a case, the maximum stress is reported as "compressive strength at failure."

B. If a sudden failure of the specimen does not occur, as is likely with ductile materials, the force-deformation curve will gradually reach a maximum force value; then, the force will decrease. The test should be stopped when that occurs, and the maximum stress value should be reported as "compressive strength."

   1. If the crosshead movement is allowed to continue, the force-deformation curve will reach a minimum force value; then, the force will increase. The specimen may be deformed until it becomes a flat disk, but no rupture will occur.

CAUTION

If the crosshead is allowed to continue its travel, the resulting force generated may be sufficient to wreck the load cell—an expensive mistake!

SHEAR TESTING

Wringing a dishrag or applying torque to a metal bolt are good illustrations of shearing action. One may get another concept of shear by considering the action of punching small round holes in paper with a simple hand tool. The apparatus for ASTM D 732, Test Method for Shear Strength of Plastics by Punch Tool (see Fig. 3-7), also provides a similar concept. (D 732 is little used, however.)

Figure 3-8 shows how in-plane shear is induced from the base of one square notch to the base of the other square notch in ASTM D 3846, Standard Test Method for In-Plane Shear Strength of Reinforced Plastics. The notches are, simply, saw cuts. The specimen is compressed longitudinally. The same antibuckling jig is used as is shown in Fig. 3-6c for ASTM D 695, Compressive Properties.

Figure 3-9 shows the geometry for short-beam shear per ASTM D 2344, Standard Test Method for Apparent Interlaminar Shear Strength of Parallel Fiber Composites by Short Beam Method, where the specimen is supported at two points very close to the loading nose at the center. (Refer to "Span-to-Depth Ratio" in the discussion of Bending Tests, which follows this discussion of Shear Testing.)

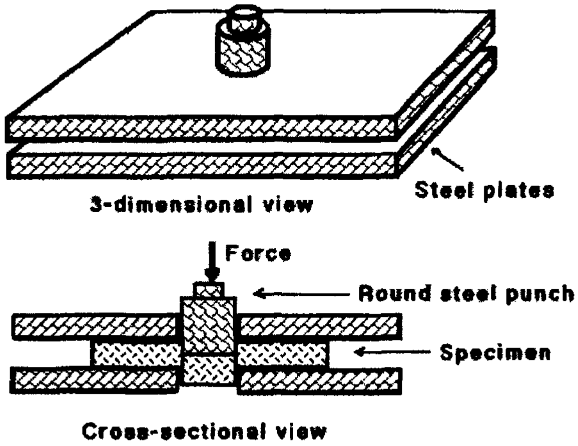
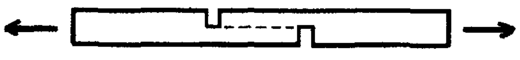


FIG. 3-7—Punch shear, ASTM D 732.



Shear will occur along dotted line

FIG. 3-8—In-plane shear, ASTM D 3846.

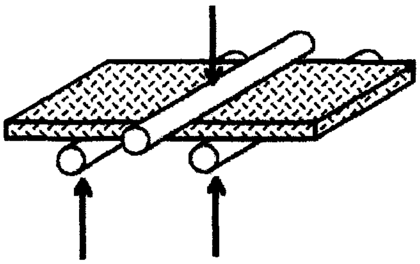


FIG. 3-9—Short-beam shear, ASTM D 2344.

Tear Testing

Other commonly used shear tests are tear tests. Figure 3-10 shows a Graves Die C specimen for ASTM D 1004, Standard Test Method for Initial Tear Resistance of Plastic Film and Sheeting. Pulling the two ends of the specimen in opposite directions causes a shearing action at the 90° corner in the center of the specimen. The force to initiate tearing is reported. For this test, the specimens must be cut with a die of the proper shape and tolerances. Maintaining very keen sharpness of the die, especially at the 90° corner, is of critical importance to avoid erroneous data as the die becomes worn, through use, or becomes nicked. Remember, down with false facts!

Figure 3-11 illustrates another tear test, ASTM D 1938, Standard Test Method for Tear Propagation Resistance of Plastic Film and Thin Sheeting by a Single-Tear Method, which measures the force necessary to propagate tearing; a thin specimen is slit with a razor blade, and the two resulting "trousers" (or "pants" or "legs") are pulled in opposite directions.

Reporting Results of Tear Testing

All of the above tear tests have somewhat different ways of reporting the results. In D 732, the thickness of the specimen enters into the calculation; in the others, it does not. Section 3.3 of D 1004 warns:

> "The resistance to tear of plastic film and sheeting, while partly dependent upon thickness, has no simple correlation with specimen thickness. Hence, tearing forces measured in pounds-force (Newtons) cannot be normalized over a wide range of specimen thickness without producing misleading data ... "

Apparent Shear

Apparent shear is a term associated with dynamic mechanical analysis (DMA) and should not be associated with the above shear test methods. (Dynamic mechanical analysis is mentioned further on, following the discussion of viscoelastic behavior.)

FIG. 3-10—"Die C" tear, ASTM D 1004.

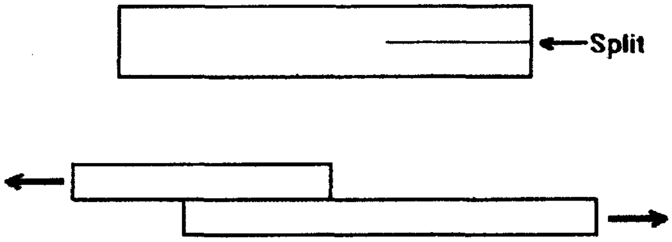


FIG. 3-11—Split tear, ASTM D 1938.

BENDING

There are two types of bending—cantilever beam and simple beam. ASTM D 747, Standard Test Method for Apparent Bending Modulus of Plastics by Means of a Cantilever Beam, is the common cantilever beam test. In it, the specimen is held in a clamp that rotates the specimen against a stop (Fig. 3-12). The method is little used and seldom discussed.

However, the simple beam (flexural) method, ASTM D 790, Standard Test Method for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials, is one of the most widely used of the mechanical property tests, as the specimen geometry is very simple and the hardware for supporting the specimen is quite simple (Fig. 3-13). However, the method is rather complicated to read, and there are a number of restrictions that must be observed in performing the test.

Measuring Deflection (Deformation)

In flexural testing, the deformation of the specimen is measured by monitoring the deflection at the center of the specimen. Most people do this by correlating the rate of crosshead movement with the time drive rate of the chart recorder.

However, an instrument called a deflectometer may be used. Its projecting arm contacts the movable crosshead, and an electrical signal is sent to the chart recorder proportionate to the amount of the crosshead movement. The chart recorder must be set up in "XY" drive, rather than time drive.

Scope of D 790-92

In the past, the complications have stemmed mainly from the fact that D 790-92 was divided into two submethods,

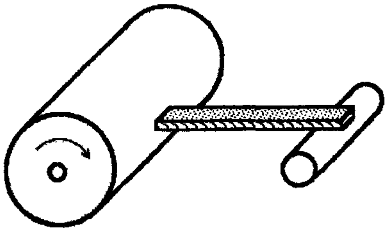


FIG. 3-12—Cantilever beam bending.

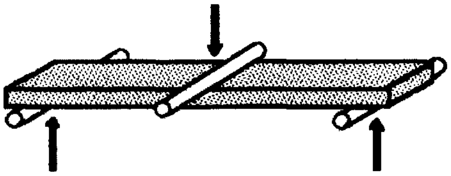


FIG. 3-13—3-Point bending, ASTM D 790.

Methods I and II, and that each submethod has a Procedure A and a Procedure B. As shown in Fig. 3-13, Method I consists of supporting the specimen at two points and loading it at the center with a single loading nose. In Method II, there are two loading noses. To further complicate matters, there are two different configurations for Method II.

- NOTICE. As this is being written, ASTM Committee D-20 is in the process of trying to simplify D 790. It is likely that Method II will be broken out into a separate method, as it is used mainly for high-modulus composites.

We will limit our discussion here to Method I. (Except for the loading geometry, the following discussion applies to Method II, as well as to Method I.)

Test Geometry and Limitations

Section 1.2.1 of D 790-92 explains that Procedure A was written "principally for materials that break at comparatively small deflections." Section 1.2.2 explains that Procedure B was written "particularly for those materials that undergo large deflections during testing."

At this point, we must pause to explain the term *outer fibers,* which is sued in D 790. Note that, as the specimen is bent (Fig. 3-13), the side of the specimen in contact with the loading nose is put into a compressive strain mode, while the opposite side is put into a tensile strain mode. There is a gradient of strains across the specimen, ranging from maximum compressive strain at the surface next to the loading nose, gradually changing to a balanced strain somewhere inside, and then gradually changing to a maximum tensile strain at the opposite surface. The term *outer fibers* refers to the material near the specimen surfaces, where the maximum strains occur, even though there may be no actual fibers in the material.

Note that, due to the fact that the greatest strains occur in the outer fibers, flexural test data are more dependent on material characteristics near the surfaces of the specimen than at the center. (That is not the case for tensile, compressive, or shear testing.) Accordingly, note that nonhomogeneous materials, such as laminated products, will give different test data, depending on which side is placed against the loading nose.

Procedure A Versus Procedure B

The only difference between Procedure A and Procedure B is in the crosshead speed. Rates of crosshead motion for Procedure A are listed in Table 1 of D 790-92. For Procedure B, use a crosshead speed ten times as fast as the speed for the applicable thickness, as shown in Table 1.

A. Alternatively, there are equations for calculating crosshead speed for Methods I and II. The variable "Z" in each of those equations is the rate of straining of the outer fibers:
   1. For Procedure A, "Z" shall be 0.01 (Paragraph 10.1.3).
   2. For Procedure B, "Z" shall be 0.10 (Paragraph 10.3.2).

Terms: Depth and Width, Instead of Width and Thickness

Note that the equations for calculating the various test data from a flexural test are entirely different from the equations involved in tensile or compressive testing. It is important to notice that the equations for strength and modulus are functions of the square and the cube of the depth, respectively. At this point, it is well to explain the terms "depth" and "width" of a specimen, as opposed to the more common terms, "width" and "thickness."

Let us suppose that we are testing specimens that are 9 mm (3/8 in.) thick by 13 mm (1/2 in.) wide. The specimen could be placed on the loading fixture with either dimension in the vertical plane. In order to prevent confusion as to which-dimension-to-use-where in the calculations:

A. We define the dimension in line with the direction of the force application as the depth of the specimen.
B. We define the shorter dimension perpendicular to the direction of the force application as the width of the specimen. (The longer dimension perpendicular to the direction of the force application is, of course, the length of the specimen.)

Restrictions on the Test Procedure

The rigorous engineering equations for calculating forces in simple beams are very long and complex. When D 790 was originated, before the days of computers, solving those equations for each set of specimens would have consumed an unacceptable amount of calculating time. By placing certain restrictions on the test geometry and procedure, it was valid to greatly simplify the equations into the formats as they now appear in the method. The following is a discussion of some of the restrictions to which one must adhere when running the test.

Span-to-Depth Ratio

Comparing Fig. 3-13 with Fig. 3-9, note that testing for flexural properties is very similar to testing for short-beam shear. The mean difference is in the span-to-depth ratio. D 790 sets this ratio nominally at 16:1 for most plastics. (But read carefully to determine the proper specimen size and span-to-depth ratio for your particular material!) Decreasing the ratio much below 16:1 would move the test from a flexural mode into the realm of a shear mode (see "Short-Beam Shear," earlier in this chapter). Therefore, one of the restrictions of the flexural test geometry is a tolerance (Section 6.2.1) that translates into a minimum span-to-depth ratio of 14:1 (and a maximum ratio of 20:1).

Strain Limit of 5%

Section 9.1.6 states, "Terminate the test if the maximum strain in the outer fibers has reached 0.05 mm/mm (in./in.)." An equation is provided for calculating the deflection at which 5% strain will occur for any given specimen depth and support span. If the material does not rupture within this limit, prepare an additional set of specimens and repeat the test, using Procedure B—this simply means increasing the crosshead speed to give a rate of straining of the outer fibers of 0.10 mm/mm (in./in.), which is ten times the straining rate of Procedure A. This will often produce rupture within the 5% limit. Note that the 5% limit also applies to Procedure B.

If rupture does not occur within 5% strain, it should be so noted on the test report, and the stress at 5% strain should be reported. In the author's experience, people outside the testing laboratory do not like to be bound by this 5% limit and will often insist that the specimens be tested to rupture and that the stress at rupture be reported. If this is done, the test report should clearly state that the test results do not conform to the restrictions of ASTM D 790. The thoughtful person will realize that, even when this statement appears, we are flirting with false facts in this situation.

Toe Compensation

As in tensile and compressive testing, a "toe" often appears at the start of the load/deformation curve. It is probably due to a combination of the hardware seating itself and the specimens seating themselves on the support noses as load is applied. The latter may be because the bearing surfaces of the specimen are not exactly parallel. Again, it is necessary to correct the effect of this toe before calculating modulus or elongation values. For instructions, refer to the "Stress/Strain Curves" Section earlier in this chapter.

PROPERTIES MEASURED FROM STRESS/ STRAIN CURVES

From here on, in our discussions of strength testing, although we may refer specifically to tensile testing, the principles we set forth will apply equally to compressive, flexural, and shear testing.

Tensile Strength and Elongation

Figure 3-14 illustrates a curve for an essentially brittle material, where there appears to be an initial straight portion, then a curved portion that ends when the specimen ruptures. Ultimate tensile strength (or compressive or flexural strength at break) and per cent elongation (or strain) at break are measured by calculating the stress and the elongation, respectively, at the end of the curve.

Modulus: A Term Often Misunderstood

Modulus is a measure of a material's resistance to deformation. In the bending mode, it is easy to visualize modulus as a measurement of stiffness.

Most people, if they have any knowledge of modulus from a high school or college physics course, think in terms of Young's modulus ("E"). In plastics testing, elastic modulus and tangent modulus are two other names for Young's modulus. Each is defined as the ratio of stress (σ) to strain (ε) at any point along the initial straight portion of a stress/strain (load/deformation) curve (line OP in Fig. 3-14):

$$E = \sigma / \varepsilon$$

However, there are also other kinds of modulus. At least two will be explained in later sections of this chapter.

- A general definition of modulus is that it is the ratio of stress to strain at any point on a stress/strain curve.

The unit for modulus in the SI system is the Pascal. For example, a semi-rigid polymer might have a tangent modulus of 2,413,000,000 Pa, which is 2.413 gigaPascals (gPa). In the English system, this would be 350,000 psi.

When listed in data tables, modulus values in the English system are stated in engineering notation. For example, 350,000 psi would be written as 3.5×10^5 psi. When using such data tables, be sure to notice, at the top of the column, what factor is being used for that column, as the factor may range anywhere from 10^3 to 10^{12}.

Yield Point

Figure 3-15 illustrates a curve for a material that is somewhat less brittle than the material in Fig. 3-14. As with the more brittle material, there is an initial straight portion, then a curved portion. In this case, the curve continues until it reaches a maximum; then it drops downward until the specimen breaks. In plastics, a point at which a curve first shows a continued increase in deformation without an increase in load is defined as the yield point. Yield point is also defined as the first point at which a curve shows zero slope. When

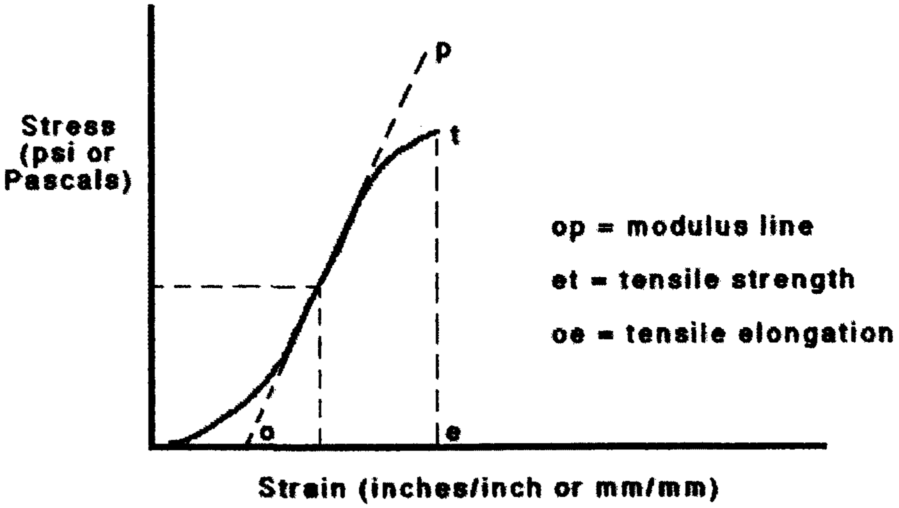


FIG. 3-14—Tensile testing terms, ASTM D 638.

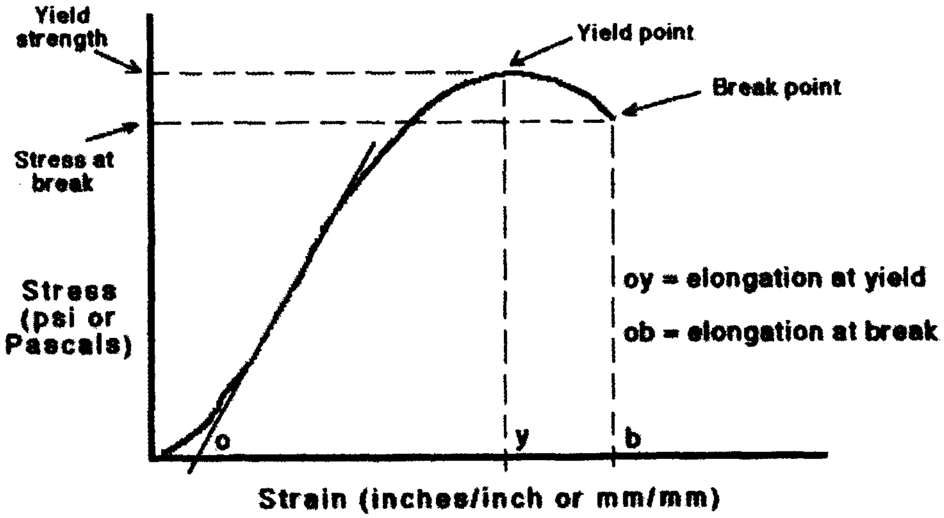


FIG. 3-15—Yield point.

this occurs, yield strength (stress at yield) and strain at yield should be measured and reported, as they are more important than the stress and strain at break.

If talking with someone in the metals industry, be careful about the term "yield strength," as it probably has a different meaning to that person. For instance, what the aluminum industry defines as a yield point is determined by drawing a straight line parallel to the initial straight portion of the curve and offset from it by 0.2%; the *point where this line intersects the curve* is the yield point. ASTM D 638 defines such a point as an "offset yield" point (Fig. 3-16), but the author has never encountered offset yield *data* in the plastics industry.

There are a few materials that may produce a curve as shown in Fig. 3-16, where the curved portion reaches a maximum, drops downward to a minimum, and then climbs back upward until the specimen breaks.

There is a huge variety of polymeric materials, with widely varying properties. Accordingly, their stress-strain curves may show all sorts of variations from the model curves shown in Figs. 3-14 thru 3-18.

Proportional Limit

For any material showing a straight-line portion in its stress/strain curve, as illustrated in Fig. 3-17, one may measure a property known as the proportional limit. Discounting the toe, it is the point where the curve first starts to deviate from a straight line. It is very close to the elastic limit, but not exactly the same thing. It appears that few people pay any attention to it. This is unfortunate because it can be very useful in characterizing the mechanical behavior of a material. It may be reported either as "stress at proportional limit" or as "strain at proportional limit."

Secant Modulus

Flexible materials usually have no straight portion in their load/deformation curves. In such cases, no proportional limit or tangent modulus may be identified. For flexible materials, one may evaluate stiffness by measuring a secant modulus.

A. To do so, the first step is to establish the "zero elongation" point, As explained in the section earlier in this chapter, this is done by drawing a straight line through the inflection point and extending it down to the baseline of the load/elongation chart. That is Point "O" in Fig. 3-18. All measurements along the "X" axis must be made from this point—not from the point where the toe starts.
B. The next step is to choose a specific value of elongation at which we wish to calculate the secant modulus. (Often this choice will be made by the customer, or by a specification.) This may be any point on the load/deformation curve. For an example, in Fig. 3-18, suppose that we choose to measure "secant modulus at 300% elongation":
   1. We need to know (from the setup of the chart versus the testing machine) what distance along the "X" axis of the chart represents 100% elongation. In this example, 25 mm along the chart represents 100% elongation.
   2. For an elongation of 300%, we must measure along the baseline from the "zero elongation point," three times that distance (in this example, 75 mm). At that point (Point "E" in Fig. 3-18), we draw a perpendicular line upward to intersect the curve (at Point "I" in Fig. 3-18).
   3. From the intersection, we draw a line parallel to the base line (Line "IL" in Fig. 3-18) back to the Y axis and read the load value. In Fig. 3-18, the load at 300% elongation is 180 Newtons (40.5 pounds-force).
C. We must convert the load value to a stress value:
   1. Let us assume that the width at the center of the specimen was 6 mm:

      6 mm = 0.006 meter (0.006 m) = 0.25 in.

   2. Let us assume that the thickness was 3 mm.

      3 mm = 0.003 m = 0.125 in.

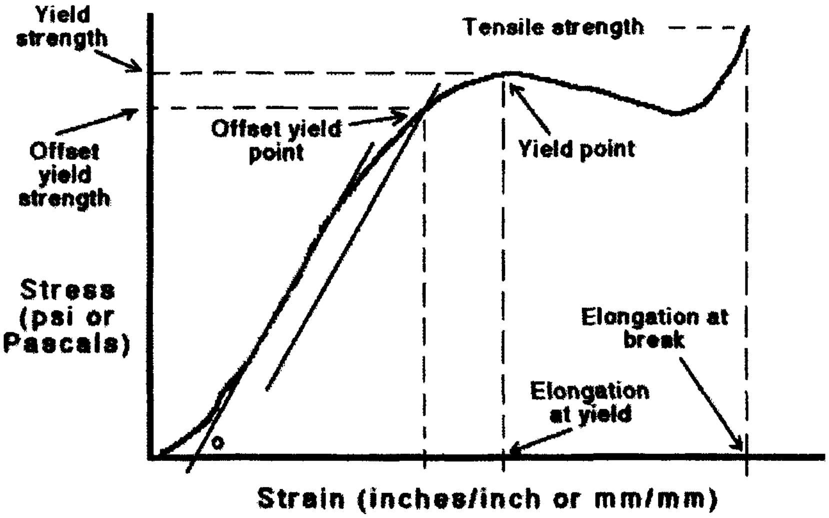


FIG. 3-16—Tensile testing terms, ASTM D 638

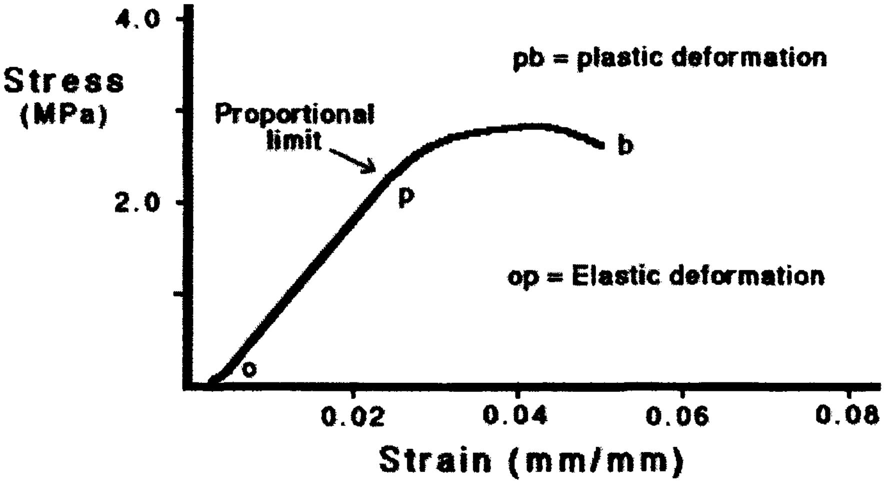


FIG. 3-17—Proportional limit.

3. Then cross-sectional area = width X thickness
   = 0.006 m × 0.003 m
   = 0.000018 m^2
   (= 0.25 in × 0.125 in.
   = 0.03125 in.^{2})
4. Stress (σ) = load/cross-sectional area
   = 180 Newtons/0.000018 m^2
   = 10,000,000 Pascals = 10 mPa
   (= 40.5 lbf/0.03125 in.^{2} = 1296 psi)

D. We chose 300% elongation as the point at which we would measure the secant modulus. This is a strain (ε) of 3 mm/mm. Recall that modulus is stress (σ) divided by strain (ε). Therefore:

1. Secant modulus = σ/ε
   = 11,666,667 Pascals/3 mm/mm
   = 3.89 mPa
   (= 1510 psi/3 in./in. = 503 psi)

For elastomeric materials, it is common for people to request that secant modulus be reported at 100, 200, and 300% elongation.

Characterizing Materials by Stress/Strain Curves

As shown in Fig. 3-19, plots of stress versus strain may be used to characterize various materials.

Note that the areas under the curves are quite different from each other. The area under a stress/strain curve is a measure of the energy necessary to break the specimen. This area is also defined as *toughness*. If we have a computer connected directly to our testing machine, it is a simple matter to measure this energy by integrating the area under the curve. However, before computers were made so obliging, such an integration was very time-consuming and usually could not be done accurately; thus, it was very seldom carried out.

A. In the past, many people tried to do it by cutting out (using scissors) the stress/strain areas from the paper on which they were drawn and then weighing the cutouts. This proved to be unsatisfactory because the thickness and the density of the paper were usually not uniform enough to give good repeatability.

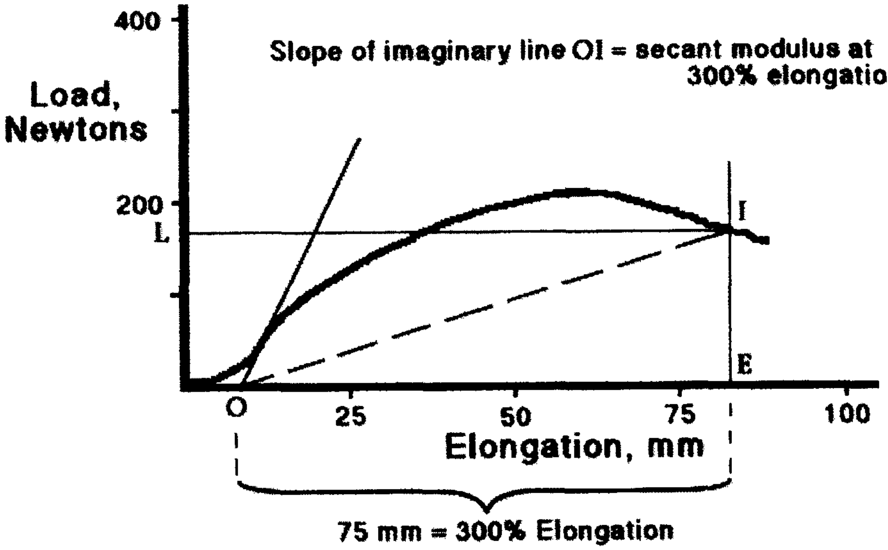


FIG. 3-18—Secant modulus.

In a later section of this chapter, we shall discuss impact testing, which is how toughness has been measured for many years.

THE PRINCIPLE OF VISCOELASTIC BEHAVIOR IN PLASTICS

The author considers this section to be the most important in this chapter. It is impossible to understand the mechanical behavior of plastics unless one understands the basic idea of viscoelasticity.

The term *elastic* is used to describe a material that will completely return to its original size and shape after being deformed to a small degree. *Webster's New World Dictionary of the American Language* defines the term *viscosity* as "the property of a solid of yielding steadily before a constant stress." The term *viscoelastic*, then, would seem to be self-contradictory—how can a material be both viscous and elastic? Yet, that is exactly how all plastics behave! That characteristic sets plastics apart from most other materials.

Three things occur whenever a force is applied to any plastic object:

A. There is immediate elastic deformation that is proportional only to the amount of the force. The amount of this purely elastic deformation will remain the same no matter how long the force is applied.
B. Concurrently, along with the immediate elastic deformation, there also is slow elastic deformation. This deformation is dependent on the time over which the force is applied, in addition to being proportional to the amount of force. The longer the force is applied, the greater the slow elastic deformation.
C. Concurrently, there also is plastic (viscous) deformation. This deformation is also proportional both to the amount of force and to the time over which the force is applied.

However, only two of the above deformations are reversed when the force is removed:

A. The object will immediately completely recover the immediate elastic portion of its total deformation.
B. The object will also completely recover the slow elastic portion of its total deformation. However, the recovery will take place gradually, over some period of time. The longer the force had been applied, the longer will be the recovery time.

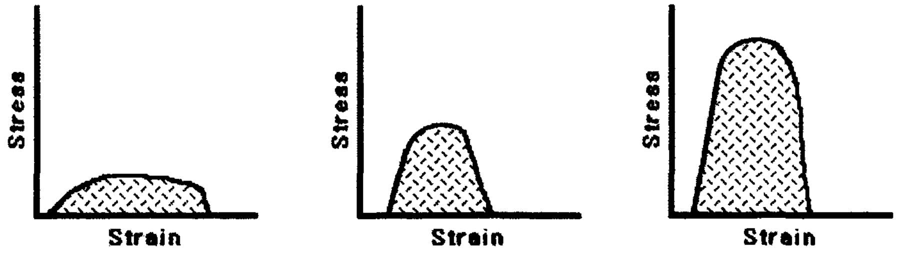
The object will not recover any of the plastic (viscous) deformation. Thus:

A. There is some degree of permanent deformation induced in every plastic whenever it is subjected to an outside force. *In many cases, it is hardly detectable, but it is there.*
B. With a moment's reflection, one will realize that this permanent deformation is cumulative. If a plastic object is subjected to more than one cycle of loading and unloading, each additional loading will add some more permanent deformation.

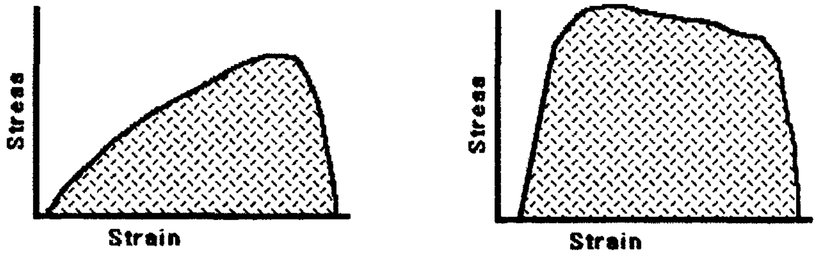
Example

Visoelastic behavior may be illustrated by an example which will be familiar to most readers:

A. Whenever we get into our automobile, we immediately sink down into the polyurethane seat by an amount directly proportional to our weight. (This is mostly immediate elastic deformation.)
B. At the start of a long trip, we noticed that the rear-view mirror was not adjusted properly. We very carefully adjusted it so that the image of the rear window was perfectly framed in the mirror as we sat in our normal driving position. A couple of hours later, we realized that the image of the rear window was no longer centered in the mirror. What happened? Was the mirror loose? Probably not! During that hour, we had gradually sunk down farther into our seat, to the extent that our eyes were now at a slightly lower level in relation to the mirror. We then readjusted the mirror. If we drove for another couple of



Flexible & Weak **Stiff & Brittle** **Stiff & Strong**



Flexible & Tough **Stiff & Tough**

FIG. 3-19—Types of materials as characterized by stress-strain curves.

hours, we may have again needed to adjust it. (This is mostly slow elastic deformation.)

C. When we got out of the car for a short stop, the seat seemed to return to its original shape (immediate elastic recovery), and when we got right back into the car, we sank down again, but the mirror was still adjusted properly. (The time span was too short for slow elastic recovery to occur.

D. Eventually, we stopped and were out of the car for several hours. This time, when we got back into the car, the mirror was again not adjusted properly. During this longer period of time, the polyurethane seat had exhibited slow elastic recovery, and our eyes were at a higher level in relation to the mirror than they were when we stopped. In fact, our eyes were at almost the same level as when we started the trip; there was not enough difference to be discernible.

E. If we drove the same car for several years, we eventually may have noticed that we were no longer positioned as high in relation to the windshield as when the car had been new; the seat had gradually taken on a permanent amount of deformation. This was viscous deformation due to the combined phenomena of creep and fatigue, which are discussed near the end of this chapter.

The magnitudes of the three separate (but concurrent) deformations will be different from each other, and each magnitude will vary from one polymer to another. Each polymer has its own combination of these three responses. These variations result in enormous ranges of various mechanical attributes among different plastics, and even from one formulation to another for a given plastic.

In a short-duration test (such as a standard tensile test) on a brittle or semi-rigid material, consider what is really occurring when the load-deformation curve shows what appears to be a straight-line segment. It means that, at any point along the straight segment, the immediate elastic portion of the total deformation is very large compared with the two time-dependent portions that are also present. The line appears to be straight because our instruments are not sensitive enough to measure the time-dependent deformations within the short duration of the test. As stated earlier, we calculate a tangent modulus and a proportional limit as though there was, indeed, a true straight-line relationship between the load and the deformation.

A Four-Way Mechanical Model to Explain Viscoelasticity

Figure 3-20 shows what is known as a four-way mechanical model, which is useful to illustrate the above three types of deformations. It includes the following components:

A. A *spring*: this represents immediate elastic behavior, which is completely recoverable.

B. A *dashpot*: this is a cylinder containing oil. There is a piston inside the cylinder immersed in the oil. The piston fits loosely enough for oil to flow slowly past it when a force is applied. This represents purely viscous behavior, which is not recoverable.

The four-way model consists of two combinations:

A. A combination of a spring and a dashpot, working in parallel with each other at the ends of two parallel bars. This is known as a Voigt-Kelvin body.

B. A combination of a spring and a dashpot working in series. This is known as a Maxwell body. Its components are independent of each other. In our version of the four-way model, the spring is attached to the "top" side of the Voigt-

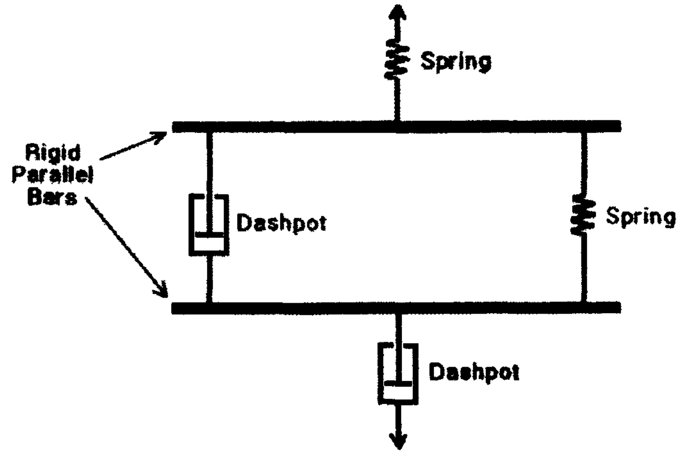


FIG. 3-20—4-Way mechanical model to explain viscoelastic behavior.

Kelvin body and the dashpot is attached to the "bottom" side.

Two restrictions (assumptions) must be invoked:

A. All parts of the assembly are completely weightless.
B. The two bars will remain parallel to each other and completely rigid, regardless of what forces act upon them.

The four-way model works like this:

A. A force is applied to pull on the spring at the "top" and on the dashpot at the "bottom." How do the components of the system react?
  1. In the Maxwell body:
    a. The spring (at the "top") will immediately stretch in direct proportion to the amount of the force. This is elastic behavior.
    b. The dashpot (at the "bottom") will react by allowing slow, steady movement of the piston away from the lower bar. This is viscous behavior.
    c. The separate movements allowed by the spring and the dashpot are additive.
  2. In the Voigt-Kelvin body:
    a. The connecting spring at the one end of the bars will try to react immediately, like the spring in the Maxwell body. However, because the bars must remain parallel, the movement of the lower bar will be slowed by the viscous response of the connecting dashpot at the other end of the bars.
  3. The restricted movement within the Voigt-Kelvin body is additive to the total movement allowed by the Maxwell body.
B. When the force is removed, how do the system's components react?
  1. In the Maxwell body:
    a. The spring will immediately recover to its original length (elastic behavior).
    b. There will be no movement of the piston in the dashpot (viscous behavior).
  2. In the Voigt-Kelvin body:
    a. The connecting spring at the one end will try to return to its original length immediately, like the spring in the Maxwell body. However, because the bars must remain parallel, the return movement will be slowed by the viscous behavior of the connecting dashpot at the other end of the bars. But eventually this spring, too, will resume its original length. Therefore, the behavior of the Voigt-Kelvin body represents slower elastic behavior.
  3. The eventual recovery of the assembly-as-a-whole is equal to the total of the recoveries of the two springs.
  4. A permanent deformation, or set, of the assembly-as-a-whole has been created equal to the amount of movement in the dashpot portion of the Maxwell model.

Temperature and Rate of Straining

All mechanical properties of plastics are dependent, due to viscoelastic behavior, on temperature and on the rate at which load is applied.

(Dynamic mechanical analysis is a technique that may be used to measure viscoelastic properties of very small specimens, over an extremely wide range of temperatures, in a single experiment. The equipment, however, is very expensive, and the resulting data require interpretation by someone well versed in the subject.)

Varying the Rate of Straining

Figure 3-21 illustrates a hypothetical family of stress/strain curves that might be obtained by testing the same homogeneous material at a given temperature but at various crosshead speeds. Depending on the ductility of the material, the inflections shown in this figure may, or may not, occur. From a practical standpoint, crosshead speeds within one decade of each other will not usually produce a significant change in response.

An interesting simple experiment will illustrate dependence on rate of loading. We are all familiar with "silly putty" (which is a silicone):

A. Try molding a piece of it into a small dumbbell shape having about 9 mm (3/8 in.) diameter in the center. Now, pull it apart very slowly with your fingers. It will not require much force. It will stretch several inches, with the center necking down to a thread (similar to taffy, when it is pulled). This is primarily viscous behavior. (Another example of viscous behavior is when the material slowly as-

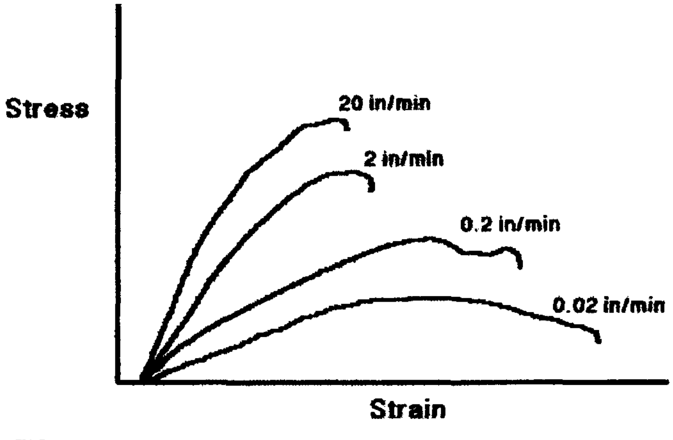


FIG. 3-21—Effect of testing speed at constant temperature.

sumes the shape of its container when it is stored for a few days.)

B. Reshape it into another similar dumbbell. This time, pull it apart as quickly as you can. It will require significantly more force than before. It will stretch only slightly, and it will break with an audible snap. There will be very little neck-down. Examine the two broken halves at the break point; they will show brittle fracture. This is primarily elastic behavior. (Another example of elastic behavior is that, as we all know, a ball of the material will bounce readily when dropped on a hard surface.)

Varying the Temperature

Figure 3-22 illustrates a hypothetical family of stress/strain curves that might be obtained by testing the same homogeneous material at a given crosshead speed but at various temperatures.

Characterizing a Polymer

When testing to characterize a material, a variety of mechanical property tests should be carried out at a minimum of one temperature below the standard test temperature of 23°C (73°F) and one temperature above, in addition to the standard test temperature.

Similarly, a variety of mechanical property tests should be carried out at several different rates of loading. It is risky to rely on data from only one temperature or one rate of loading.

IMPACT TESTING

Because mechanical response depends on the rate of loading, we often need to be able to measure this response at very high test speeds, much higher than the maximum speed of 51 mm (20 in.) per minute obtainable with a UTM. Therefore, several different impact test methods have been developed. They all measure the energy needed to break the specimen. This, in turn, is a measure of toughness, which (as we stated in a previous section) is the area under the stress/strain curve.

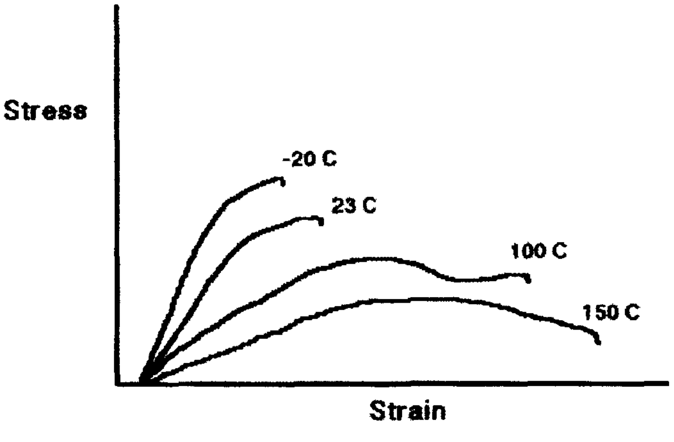


FIG. 3-22—Effect of temperature at a given speed of testing.

Izod and Charpy Testing

In the past, ASTM D 256, Test Method for Determining the Pendulum Impact Resistance of Notched Specimens of Plastics, has consisted of five methods. Method A is the Izod test method. The Charpy test method was Method B.[2] These methods were the first impact tests for plastics. (They were adapted from similar tests of metals.) Consequently, nearly every producer has published data from one, or both, of these two test methods for years. Because of this, there is much insistence on having Izod or Charpy data on each new product so that customers may make comparisons with the old data, even though some other impact test might be more useful for a specific application.

Those who have taken the ASTM course, "Major Testing Techniques for Plastics," have been invited to phone questions to the instructors after they return to their jobs. The only questions that have been phoned to the author were ones concerning Izod. That is why so much space in this chapter will deal with Izod testing.

Mechanism of Fracture

In general, the process of breaking anything involves two steps:

A. Initiating a crack in the object.

B. Propagating the crack until a complete break is achieved. Thus, in measuring impact properties, we are dealing with both:
   1. Measuring the energy to initiate a crack in the specimen.
   2. Measuring the energy to propagate the crack to the failure point.

Many people criticize the basic premise of both the Izod and Charpy test methods because notches are machined into the specimens before testing in order to provide crack initiation at a specified point in the specimen. Thus, the Izod and Charpy methods measure mostly the propagation energy. There is almost no initiation energy involved.

To satisfy the need for a cantilever beam impact test that measures both initiation and propagation energy, Committee D-20 developed an unnotched test, ASTM D 4812, Test Method for Unnotched Cantilever Beam Impact Strength of Plastics. It calls for a specimen having a depth of 12.70 mm (0.500 in.). For both the Izod and the Charpy methods, the depth below the notch is 10.16 mm (0.400 in.). Thus, it is not valid to compare data from D 4012 with either Izod or Charpy data.

Test Machines and Test Geometries

All of the methods in D 256-93a use a rectilinear specimen, 12.7 mm (0.5 in.) in depth, which is notched at the center of one side.

A. The terms *width* and *depth* are applied in the same manner as described in the section on flexural testing. Thus,

---

[2] As this is being written, ASTM Committee D-20 is in the process of splitting the Charpy method out of D 256 and writing it as a separate method. This is being done to make it easier to harmonize the ASTM methods with the corresponding ISO methods.

the width is usually the sample thickness (but "it ainnn't ne-ce-ssarr-ily sooo!").

These tests are conducted using pendulum machines especially designed for the purpose. DO NOT ATTEMPT TO BUILD ONE OF THESE MACHINES YOURSELF! You will spend far more than the cost of a commercial machine, and YOU WILL NEVER GET IT RIGHT!

Both the Izod and the Charpy test may be carried out on the same machine by attaching different striking noses to the end of the pendulum, which also often is called a "hammer" (see Fig. 3-23*c* of this chapter and Figs. 1*a* thru 6 of ASTM D 256-93A). The only other differences between Izod and Charpy testing are:

A. 1. For Charpy, the specimen length is 126 mm (5 in.); see Fig. 7 of D 256-93A.
   2. For Izod, the specimen length is 63 mm (2.5 in.); see Fig. 4 of D 256-93A.

B. 1. For the Charpy test, the ends of the specimen rest on two steel supports built into the base of the machine. The ends of the notched side are placed against massive rigid stops so as to induce three-point bending when the specimen is struck across the center of the opposite side (Fig. 3-23*a*).
   2. For the Izod test, the specimen is clamped vertically in a vise that is build into the base of the machine. The root of the notch must align precisely with the top of the vise (Fig. 3-23*b*). The specimen is struck across a point precisely 22.0 mm (0.866 in.) above the top of the vise so as to induce cantilever bending along a precisely controlled moment arm.

Operation of Pendulum Impact Machines

Figure 3-23*c* depicts the general idea of a pendulum impact test. The striking nose is mounted at the end of a rigid steel pendulum (see also Fig. 2 or Fig. 5 of D 256-93a). The free end of the pendulum is raised until it becomes automatically held at a preset starting height. This height was calculated to ensure that, when the pendulum is released from its starting position, the striking nose will reach a velocity of 203 meters (8000 inches) per minute at the bottom of its swing, just at the instant before it strikes the specimen.

At its starting position, the pendulum possesses an amount of potential energy that is stated for each individual pendulum. When the pendulum is released, its potential energy is gradually transformed into kinetic energy as it swings downward; at the bottom of the swing, all of the potential energy has become kinetic energy. As the pendulum continues to swing (upward now), the kinetic energy is transformed back into potential energy until the transformation of energy is complete and the pendulum stops swinging upward. The machine automatically measures the distance of this upward swing and converts it to a scale value of energy lost during the swing.

Pendulum Ranges

It is not possible for any one pendulum to be able to break the toughest plastics and still provide precise results when breaking plastics that are not tough. The problem is solved in two different ways, depending on the machine design:

A. With some machines, the mass of a given pendulum is designed to give precise results over a stated range of impact energies. To test material that is outside that range, one replaces the entire pendulum with one designed for the range of interest.

B. With some other machines, the pendulum is permanently mounted in the machine. The various ranges are achieved by mounting designated weights on the sides of the pendulum.

Corrections

When a machine is first set up, the following corrections must be established. They should be repeated, periodically.

A. With no specimen in place, the difference between the stated pendulum energy and the scale value represents energy lost due to air resistance and to friction in the pendulum's bearings. (See Sections 10.3 thru 10.3.3 and Note 21 of D 256-93a.)

B. With a specimen in place, the difference between the stated pendulum energy and the scale value represents energy absorbed in breaking the specimen, in addition to the energy losses due to air resistance and friction.

   1. When testing specimens, it is necessary to correct each scale value by subtracting an appropriate value for the air resistance and friction losses. But the corrections are not constant—the greater the energy absorbed in breaking the specimen, the smaller will be the correction. Therefore, it is necessary to construct a correction chart as described in Appendices X2 and X3 of D 256-93a.

Method C of D 256-93A is for very small Izod impact energies; it is the same as Method A, except that it requires an additional correction for tossing the broken half of the specimen. These corrections, too, must be subtracted from each scale value (or from each average of scale values).

Calculations

It is important to record whether the readout scale of your particular machine shows foot-pounds-force or inch-pounds-force (or joules, if you have a metric machine). After making the above corrections, the only calculations necessary are:

A. (If the machine readout is in inch-pounds-force, divide by 12 to convert to foot-pounds-force.)

B. Divide each energy value of foot-pounds-force (or joules) by the measured width of the specimen in order to be able to report results as "foot-pounds-force per inch" (or "joules per meter").

   1. Some old data, generated under older versions of D 256, may be labelled "foot-pounds per inch of notch." The data were calculated in the same manner as now and may be legitimately compared to newer data—it's just that this was an older nomenclature; many years ago, the author helped instigate a revision to D 256 that replaced this confusing expression.

In the Izod method, the type of failure must be reported for each specimen, i.e., complete break, hinge bread, partial break, or non-break.

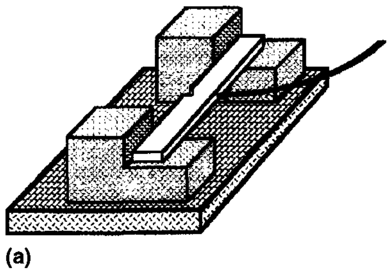


FIG. 3-23*a*—Charpy impact, ASTM D 256.

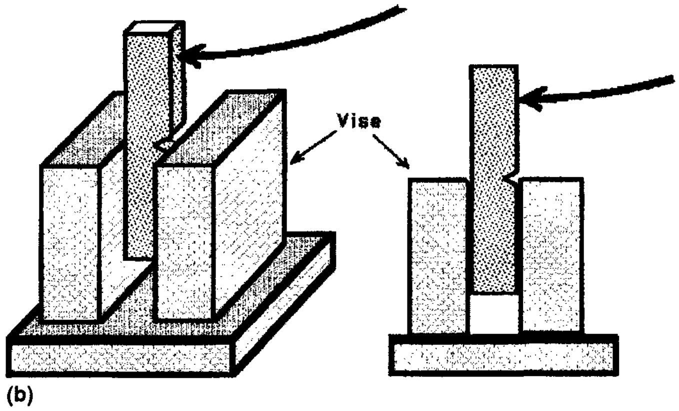


FIG. 3-23*b*—Izod impact, ASTM D 256.

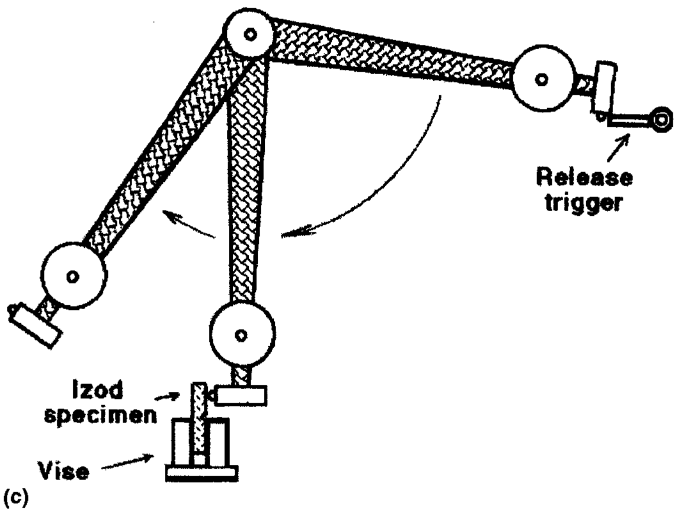


FIG. 3-23*c*—Pendulum impact.

A. Section 5.7 of D 256-93A gives definitions and instructions in regard to this. Be sure to comply carefully with all of that section.

B. When there is a hinge break or a partial break, be sure to "catch" the pendulum, by hand, as it swings back downward, so that it will not hit the specimen a second time.

Logical Minds: Beware!

Despite the fact that the impact energy is normalized for the width of the specimen, comparisons are valid only between specimens of approximately the same widths. Do not try to compare results from a 3.2-mm (0.125-in.) specimen with a 6.4-mm (0.250-in.) specimen. The reason is that the narrower the specimen, the more it tends to buckle when the striker hits it, thus absorbing more energy than is necessary to break it.

For the same reason, do not try to adhere two thinner specimens together unless that is one of the factors being measured.

Specimen Preparation

Despite all of the above explanations, the actual Izod or Charpy testing is very easy to perform and may be carried out very rapidly. The number 1 problem with these tests is that machining of the notches is very exacting and thus may be extremely time-consuming. As a result, people often do not bother to ensure that specimens are correctly prepared, thus generating false facts.

The blanks for the specimens may be molded, but do not try to mold the notch into the specimen! The extremely tight tolerance on the notch radius (see below) cannot be achieved through molding. Some laboratory equipment suppliers offer a special tool for notching the specimens. Many people use a milling machine. If using a milling machine, better results are usually obtained with a single-tooth cutter than with a multi-tooth cutter.

A. It is difficult to obtain a single-tooth cutter. One solution is to buy a three-tooth cutter and grind back two of the teeth slightly so that those two teeth will not contact the specimen.

Notch Radius

The chief problem in preparing specimens is that there must be a very small radius at the root of the notch [0.25 mm (0.010 in.], and the tolerance on this radius is extremely tight [±0.05 mm (0.002 in.)]. Several years ago, as part of a round robin testing program in ASTM Committee D-20, specimens notched by fourteen different laboratories were carefully measured on an optical comparator, which magnifies images 1000-fold. Specimens from twelve of the laboratories failed to meet the above notch radius tolerance. What was particularly discouraging was the fact that those were all experiences laboratories who thought that they were in compliance with D 256!

Having a cutting tool that meets the above tolerance does not ensure that the notch produced from it will also meet the limits. Also, a given tool may produce notches of different sizes in different plastics. Getting the notch correct for your particular material is likely to be a matter of trial and, hopefully not too much, error. Therefore, it is necessary to measure notches in actual specimens until one feels confident that the process is under control.

The most precise way to measure is with an optical comparator (mentioned above); however, they are rather expensive. Another way is to photograph the specimen through a microscope; if doing so, be sure also to photograph something of known dimension with the same setup, or substitute some other way of calibrating the distances in the photograph, because the total magnification is dependent on both the microscope and the camera, and any stated nominal magnifications may be approximate only.

The simplest technique that the author has found is to use a small six-power magnifier with calibrated reference circles, which is available from Edmund Scientific Co.; 101 E. Gloucester Pike; Barrington, NJ 08007-1380 (phone 609-573-6250). While this will not give actual measurements of the notch radius, the author has found it to be satisfactory for determining whether or not the notch radius is within the allowed tolerance. It was described in Edmund's 1992 catalog as "6X Pocket Comparator with Contact Reticle, Catalog No. A41,055, Price: $79.00."

Depth Below the Notch

The second-most-exacting (although not particularly difficult) aspect of machining the notch is to ensure that the distance from the bottom of the notch to the other side is 10.16 ± 0.05 mm (0.400 ± 0.002 in.) as specified in Figs. 4 and 7 of D 256-93A. In order to measure this distance, it is necessary to mount a knife-edge accessory on the measuring device. The radius of the knife-edge must be sharp enough to rest in the very bottom of the notch in order to get a valid measurement (Fig. 3-24).

The reason for the tight tolerance on the 10.16-mm (0.400-in.) dimension is that this depth below the notch is not included in the calculation when testing according to D 256. Do not try to compare D 256 results directly with results from the comparable ISO test methods or British Standards because those methods include in their calculations the actual cross-sectional area below the notch, which gives results of an entirely different magnitude.

Cutting Proper Notches

Regardless of what equipment is used, cutting notches within the allowed tolerance will probably be a matter of trial and error. Some things to try are:

A. Vary the rotational speed of the cutter.
B. Vary the rate of feeding the stock into the cutter.
C. Try machining with, and without, lubrication of the cutting tool by water.

Also, do not try to notch individual specimens—the specimen blanks must be ganged together, with at least two extra specimen blanks at each end of the "gang"; these extra specimens will not have the proper notch radius, and they must be discarded.

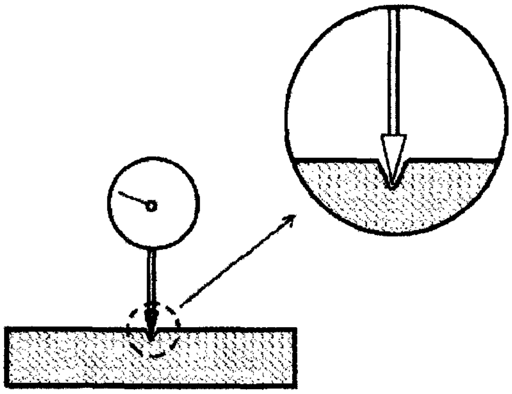


FIG. 3-24—Izod specimen—measurement of depth below notch.

Notch Sensitivity

Some materials are very *notch sensitive*, that is, tiny cracks in the surface of the material will make it much more susceptible to rupture than with material having a smoother surface. Method D of D 256-93A is a procedure for measuring notch sensitivity of a polymer by measuring its response to various notch radii in an Izod-type test. Polycarbonate, for example, is a polymer that is notably notch sensitive.

Reversed Notch Versus Unnotched

Method E of D 256-93A is a reversed notch cantilever beam test. It was added to D 256 many years ago in an attempt to satisfy people who wanted a test that measures propagation energy plus initiation energy. The method is superior to unnotched methods because:

A. It is difficult to machine a specimen with precisely parallel sides, which is what is necessary for an unnotched test—very uniform depth is important.
B. In Method E, the 10.16-mm (0.400-in.) depth of the bar is easily controlled at the root of the notch.
C. Thus, there is no confusion as to the depth of the specimen, and it is valid to compare results between Method E and Method A (Izod) in order to estimate initiation energy.

However, Method E gives data that are slightly different from unnotched test data. Therefore, it never came into popular use, and people insisted on being provided with data from unnotched tests even though there is no evidence that unnotched data are more valid.

A. The greatest problem with this was in deciding whether to use specimens 10.16 mm (0.400 in.) in depth, so the results could be compared with D 256 results, or whether to make the depth 12.70 mm (0.5 in.) so that the loading nose of the pendulum wold strike the specimen precisely at the bottom of its arc of swing.
   1. In the author's experience, no one (and I mean *no one*) who requested an unnotched test knew which of these alternatives he or she wanted.
   2. Furthermore, they would not make a decision in regard to it! They depended on testing laboratory personnel to choose the thickness.
      a. That means, of course, that they didn't understand what they were requesting and probably didn't pay any attention to which thickness was actually used, even though it made serious differences in the test data:
   3. There has been at least one instance where a company published unnotched data without stating what the specimen thickness was. It was presented in such a way as to imply that comparisons with notched data were valid. This, in turn would imply that 10.16-mm (0.4-in.) specimens had been used.
      a. By testing some comparable material in the author's laboratory, it became evident that the data must have been obtained with approximately 12.7-mm (0.5-in.) specimens, so comparisons with notched data were really not valid.

Finally, A Standard Test Method for Unnotched Impact

In view of the above, it eventually became evident that a standard method was needed for an unnotched impact test in order to decrease the confusion. Thus, as stated earlier, D 4812, Test Method for Unnotched Cantilever Beam Impact Strength of Plastics, was adopted in 1988. It used a specimen that is 12.70 ± 0.15 mm (0.500 ± 0.006 in.) in depth [instead of 10.2 mm (0.400 in.) as in the Izod test]. Therefore, data from this test are not comparable with data from the Izod test, even though the impact energies are expressed in "joules per meter" ("foot-pounds-force per inch")—the same units that are used in the Izod test.

Although it would seem that any unnotched test method would be extremely simple and easy to write, such was not the case. Anyone doing unnotched impact testing should follow this method exactly. Section 6 on Test Specimens is especially important. There is a special caution in Section 6.1.1 of D 4812-93:

> "6.1.1 The tolerance for test specimen depth was chosen on the basis of molding cosiderations (Caution—Interlaboratory testing has indicated that this tolerance range appears to be suitable for most, but not all, materials. For a few materials, the impact strength may be found to vary with the specimen depth at the extremes of the above tolerance range. Users of the test method should check each material type; if such a dependency is found, a tolerance of ±0.05 mm (±0.002 in. is required.)."

Tensile Impact

ASTM D 1822, Test Method for Tensile-Impact Energy to Break Plastics and Electrical Insulating Materials, is a very good impact test from the standpoint that it is very straightforward—the specimen breaks in pure tensile mode at 203 m (8000 in.) per minute. Most of the newer Izod/Charpy pendulum machines are designed so that the tensile impact test may be carried out on the same equipment, with only some simple changes in the specimen-holding and striking hardware. The idea of the test is illustrated in Fig. 3-25.

Specimens

There are two specimen geometries. The Type L specimen is the same as the Type V specimen of D 638 except that a small bolthole (see below) is drilled through each end. The Type S specimen is shorter, also having a small bolthole drilled through each end. With the Type L specimen, the extension is comparatively high. With the Type S specimen, the extension is comparatively low. In general, the Type S specimen gives greater reproducibility, but less differentiation among materials.

Test Geometry

One end of the specimen is both bolted and clamped into a narrow set of jaws attached to the end of the pendulum.[3] The other end is both bolted and clamped into a light-weight (but rigid) crosspiece that rides in its own holder on the end of the pendulum. When the pendulum reaches the bottom of its swing, the leading (narrow) jaws pass between two rigid metal stops that are firmly attached to the base of the machine. The crosspiece attached to the other end of the specimen is too wide to pass between the two rigid stops; when it hits the stops, the specimen must break if a massive enough pendulum (hammer) has been chosen. The crosspiece, still gripping one half of the broken specimen, slides out of its holder, allowing the pendulum and the other half of the broken specimen to continue its swing.

Limitations

Data obtained with hammers of different capacity may not be comparable. Read Section 5 of D 1822-93 very carefully for other limitations of the method. Also, read Appendix X1 for determination of a correction for specimen bounce and Appendix X2 for setup and calibration procedures.[4]

Calculations

After applying corrections for specimen bounce and for air resistance and friction, divide each energy value by the minimum cross-sectional area of the specimen to calculate kiloJoules per square meter (or foot-lbs-force per square inch).

Advantages and Disadvantages of the Method

Advantages:

A. The geometry is simple—the specimen breaks in pure tensile mode.
B. Specimens may either be molded, machined, or die cut.
C. Specimens that are too flexible or too thin to be tested by D 256 may be tested by this method.

Disadvantage: About the only disadvantage of the tensile impact method is that it is very time consuming. This is due to the necessity of bolting each specimen in place and then unbolting the broken specimen.

Falling-Weight Tests

There are several falling-weight test methods for plastics (see Fig. 3-26). A free-falling weight (tup) having a rounded nose is dropped directly on the center of the specimen. Typically, the specimen rests on a circular support ring. Specimens may be round or square, but they must be large enough to overlap the entire circumference of the support ring. A typical specimen size might range from 60 mm (2.4 in.) square to 100 mm (4 in.) square. The specimen may, or may not, be clamped to the support ring; data from clamped specimens are likely to be different from data from unclamped specimens. Clamping is recommended, as it improves the precision of the data.

Failure Criteria

Unlike all other impact methods, falling weight methods measure energy at the onset of damage.

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[3] With some machines, one end of the specimen is attached to the base, and the two stops are attached to the pendulum. The net effect is the same. With either setup, possible grip slippage is a problem; that is why both bolting and jaw gripping are used.

[4] These appendices should really be designated as annexes, because they contain information that is vital to obtaining correct data.

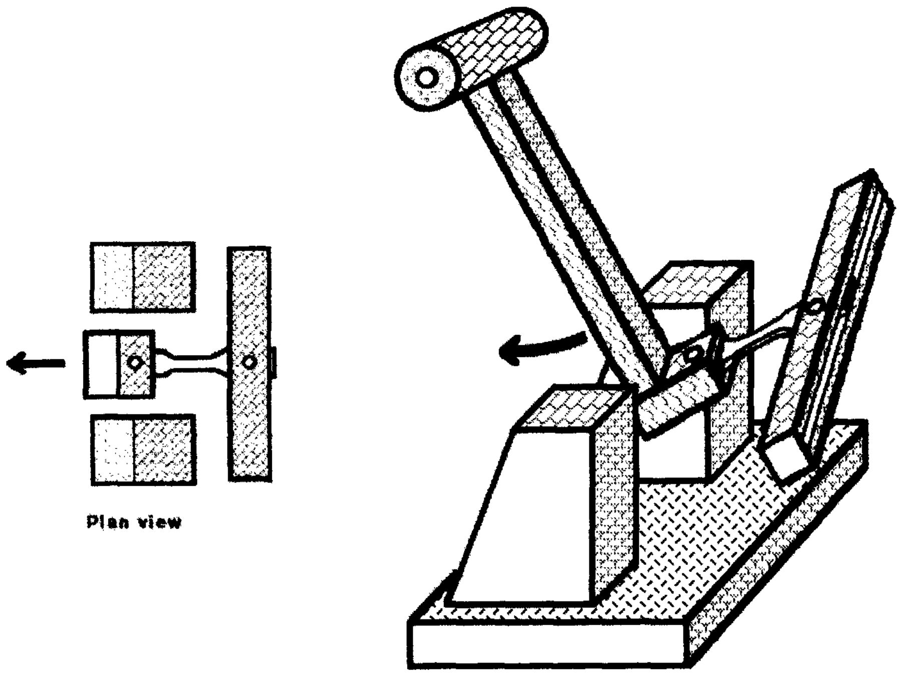

FIG. 3-25—Tensile impact hardware.

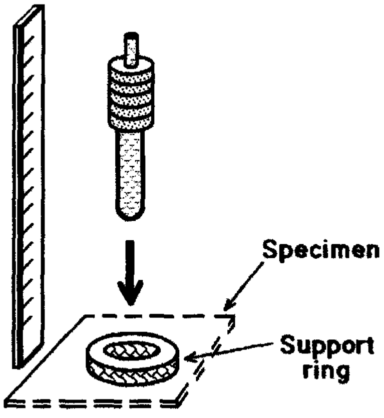

FIG. 3-26—Falling weight impact.

Before undertaking any falling weight test, the first problem that must be addressed is establishing agreement as to what constitutes the onset of damage (which is "failure") for the particular plastic that is to be tested.

A. With ductile materials, failure might be defined as the presence of an indentation, or perhaps an indentation exceeding a stated diameter, in the top surface of the specimen.
B. With some brittle plastics, the center of the specimen may shatter, leaving a hole. With some other brittle plastics, the failure is likely to be a star crack in the reverse side of the specimen. It is often difficult to determine the presence of such a crack. In testing various formulations of a reinforced thermosetting polyester, the author found that some cracks could only be detected by smearing ink on the reverse surface of the specimen after impact and then wiping it off; subsequently, it was found that different inks gave different degrees of detection. We eventually standardized on "black stamp pad ink #10," for our particular plastic.

Test Methods

The most common falling-weight test methods for plastics are:

- D 1709, Test Methods for Impact Resistance of Plastic Film by the Free-Falling Dart Method.
- D 5628, Test Method for Impact Resistance of Flat, Rigid Plastic Specimens by Means of a Falling Dart (Tup or Falling Mass).
- D 5420, Test Method for Impact Resistance of Flat, Rigid Plastic Specimens by Means of a Striker Impacted by a Falling Weight (Gardner Impact).
  - NOTICE—It was decided to split Gardner Impact out of D 3029, Test Methods for Impact Resistance of Flat Rigid Plastic Specimens by Means of a Tup (Falling Weight). It now appears as D 5420, as shown above. D 5628 consists of the balance of the D 3029 subject matter, with several modifications. Subsequently, D 3029 was withdrawn in 1996.

In the Gardner impact test, the indentor rests on the surface of the specimen at its center. The weight falls thru a guide tube, striking the indentor, which transfers the energy to the specimen.

Procedure

This discussion will concern chiefly D 5628, which is the most general method. Figure 1 of D 5628-94 shows five different geometries for tups, labelled "FA" thru "FE." D 5628 covers two submethods.

- *Constant weight, variable height:* The same tup is dropped from various heights.

- *Constant height, variable weight:* The tup is dropped from the same height each time, but small increments of weight are added to the tup or removed from the tup before each drop.

The following is the procedure for the constant weight, variable height submethod:

A. *Set-up procedure:* One should assume that up to five specimens may be necessary to complete this phase:
  1. An arbitrary height is chosen, and the tup is dropped on a specimen.
  2. The appropriate specimen surface is then carefully examined to determine whether the blow caused a failure (see "Failure Criteria," above).
    a. If failure did not occur, the tup is dropped on another specimen from a higher height. If failure occurred, the tup is dropped on another specimen from a lower height.
    b. This is repeated about two to four times, until a height is found that may be expected, just barely, to cause failure. This will be the starting height for the actual test.

B. *Test procedure:* At least twenty specimens are required:
  1. An increment of height must be chosen. This is the amount by which the height must be increased or decreased after each specimen is hit. The increment is arbitrary (usually, 10 mm or 1 in.), but the same increment must be used throughout the test.
  2. The tup is raised to the starting height, which was determined in the setup procedure. The tup is dropped on a specimen.
  3. The appropriate specimen surface is then carefully examined to determine whether the blow caused a failure.
  4. The tup is then dropped on another specimen—one which has not previously been hit:
    a. If failure did not occur in the previous specimen, the tup is dropped from a height that is higher by one increment.
    b. If failure occurred, the tup is dropped from a height that is lower by one increment.
  5. Steps B.3 and 4 are repeated until 20 specimens have been tested.

The procedure for the constant weight, variable height submethod parallels the above steps, except that:

A. A starting weight must be determined in the setup procedure.

B. The weight of the tup is varied in chosen increments, from one specimen to the next, during the test procedure.

With either submethod, specimens must not be hit more than once, even if no failure is detected, because some fatigue failure may have been initiated without detection. This could lead to a false failure if the specimen were struck again.

Calculation

After 20 or more specimens have been tested, use the Bruceton Staircase Method (the Up-and-Down Method) to calculate the mean failure energy, which is the energy that will cause 50% of the specimens to fail. This is a rather obscure procedure to learn, but it is easy to calculate once it has been learned. Follow the example contained in the appendices of D 5628.

Advantages of Falling Weight Methods

A. The equipment is relatively simple and is not expensive.

B. Specimen preparation is very simple, no machining is required.

C. Exacting calibration procedures are not required.

D. It takes only a short time to actually test the 20 specimens.

Disadvantages

A. Traditional falling weight tests consume large quantities of material—up to 5 setup specimens plus 20 test specimens, in order to obtain a single test result.

B. Data are not comparable between:
  1. Different nose or support ring sizes.
  2. Clamped and unclamped specimens.
  3. Specimens of greatly different thicknesses.
  4. Materials that require different criteria for determining specimen failure, such as materials of widely differing modulus.

C. Results should be used only to obtain relative rankings of materials. The impact values cannot be considered absolute unless the geometry of the test equipment and specimen conform to the end-use requirement.

D. Even relative rankings will not be valid for end-use situations where the velocity of impact differs greatly from the velocity during the tests.

Instrumented Tests

One drawback of all of the above impact tests is that they measure only the energy to break the specimen—there is no measurement of force during the event.

Instrumented Tensile Impact

A very useful modification of the tensile impact test was accomplished in the author's laboratory, as follows:

A. A strain gage was installed in the end portion of the pendulum that held the leading jaw.

B. During the extremely brief time that force was being transmitted thru the specimen, the strain gage generated an electronic signal in direct proportion to this varying force.

C. A data logger recorded this signal in extremely small increments of time until the specimen broke.

D. Then, the data logger fed the entire series of signals, at a slower rate, into a storage oscilloscope.

E. The signals appeared on the oscilloscope as a curve of force versus time and could be regarded somewhat like a force/deformation curve.

The advantage of this technique is illustrated in Fig. 3-27, where we have drawn Curves A and B to represent two tests that show the same amount of area under each curve.

A. If these two specimens had been tested with a non-instrumented test, their performances would seem to be identical because non-instrumented tests measure only

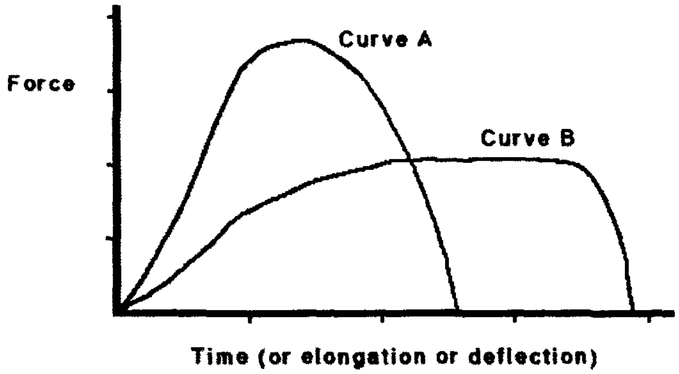

FIG. 3-27—Instrumented impact force curves.

the total energy absorbed, which was the same for both specimens. However, it takes only a glance at two curves like those in Fig. 3-27 to see that the two specimens actually responded quite differently to the impact event.

As yet, the author is not aware of any standard test method for tensile impact. However, a proposed method for an instrumented version of the Charpy impact test is being balloted in Committee D-20 now (1996) as Project X-10-127.

Instrumented Punch Impact

The more widely used type of instrumented impact test is the punch impact test, D 3763, Test Method for High-Speed Puncture Properties of Plastics Using Load and Displacement Sensors. The instrumentation works essentially the same as explained in the previous section. The specimen and test geometry is somewhat analogous to the (non-instrumented) falling weight tests, with the following exceptions:

A. The punch must be driven entirely through the specimen with no more than a 10% loss of velocity. Therefore, there is no correlation with results from any of the non-instrumented falling weight tests.
B. Only five specimens are needed to calculate a test result. The specimen size is about the same.

There are three general types of machines built for instrumented punch impact—hydraulically driven, pneumatically driven, and falling weight. The hydraulically driven machines can provide higher initial punch velocities than the other machines, but they are far more complex and a lot more expensive.

Curves

These machines provide, for each specimen, two curves (see Fig. 3-28). One is a curve of force versus time (force versus deflection of the center of the specimen is also possible, in some machines). The second curve is an integration of the first one, thus becoming a curve of energy absorbed up to any chosen point on the curve. Thus, one may analyze the rate of force buildup and the rate of energy absorption. One may also measure the force needed to initiate yielding (at the highest point on the force/time curve), as well as the energy needed to initiate yielding (at that same time point on the energy curve).

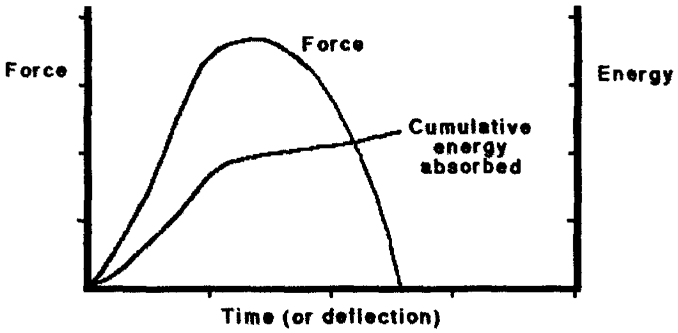

FIG. 3-28—Instrumented impact force and energy curves.

Thus, instrumented impact tests can be especially useful in research, development, and trouble shooting. Some companies even use them for quality control testing in spite of the high cost of the equipment. Most of the automotive companies use them extensively.

There is no standard instruction for identifying the point of total failure along either the force curve or the energy curve; this is left to the discretion of each laboratory, depending on the mode of failure. The reason for this is a sound one: If the mode of total failure is a shattering of the specimen, the curve will drop precipitously, and the failure point will be rather obvious. However, if the punch forces a hole through the specimen without shattering the specimen, there will be considerable friction between the sides of the punch and the hole as the punch continues to move through the specimen after the failure point. This maintains a false force (and thus also a false energy) signal over an indefinite portion along the tail of the curve. The false portion is different for each material and thus defies general standardization.

SURFACE PROPERTIES

Hardness

Hardness is the most commonly measured of the surface properties. ASTM D 2583, Test Method for Indentation Hardness of Rigid Plastics by Means of a Barcol Impressor, is used for relatively hard plastics. As implied in the title, the method is based on a specific commercial instrument. In use, a spring-loaded steel point is forced, by hand, against the surface of the material. The degree of penetration is read as a Barcol Hardness value from a dial gage built into the impressor.

Even with very hard, stiff material, it is important to place the material on a very firm, hard surface while using the impressor. Differences in readings may be obtained with a specimen placed on a stone surface as compared with the same specimen placed on a wood surface.

"Shore A" and "Shore D" Durometer

ASTM D 2240, Test Method for Rubber Property—Durometer Hardness, is used for plastics that are too soft to be measured with the Barcol impressor. The method includes two instruments—the Type A durometer for plastics in the softer part of the range and the Type D durometer for plastics in the harder part of the range. Both instruments work somewhat like the Barcol impressor. Each has its own independent scale of hardness values, referred to respectively as "Shore A" and "Shore D." In reporting data, one must designate which scale applies.

Rockwell Hardness

Although it is not as widely used in the plastics industry as the preceding hardness tests, we should mention Rockwell hardness. This is another case where a test that is widely used in the metals industries has been adopted by the plastics industry.

It is done with a rather massive specialized instrument, which uses one of several possible steel ball indentors. There is a different Rockwell hardness scale for each indentor. The indentor first is forced against the specimen for a short period under a "minor" load and then under a "major" load. A hardness reading is then taken from the appropriate scale. In view of the partially viscous behavior of all polymers, it is evident that more care is required in conducting Rockwell hardness on plastics than on metals. If you conduct Rockwell hardness on plastics, make absolutely certain that the procedure is carried out to the letter according to ASTM D 785, Test Method for Rockwell Hardness of Plastics and Electrical Insulating Materials. Otherwise, the data will be worthless.

Do Not Compare Hardness Scales

Values read from any hardness scale—Barcol, Shore A, Shore D, any given Rockwell scale, or any other scale—are not comparable to values that have been read from another hardness scale—there is no correlation between the various scales.

Abrasion

Taber Abraser

The most commonly used (and misused) abrasion test method for plastics is ASTM D 1044, Test Method for Resistance of Transparent Plastics to Surface Abrasion. It is based on a specific apparatus, the Taber abraser. A circular specimen is prepared and subjected to a light source. The amount of transmitted light is measured using an integrating sphere photometer.

The circular specimen is then mounted on a driven turntable (see Fig. 3-29). A pair of small abrasive wheels are mounted on the ends of two pivoting arms, which allow the wheels to rest on the surface of the specimen. The abrasive wheels are mounted on bearings so that they may be rotated freely by the motion of the turntable, thus causing an abraded path near the circumference of the specimen.

After a stated number of revolutions, the specimen is removed and a portion of the abraded path is subjected to the same light source. Again, the amount of transmitted light is measured. From this, the percentage of transmitted light that is diffused by the abraded track is calculated.

- CAUTION. There are a great many people who misuse this method by using it for opaque or translucent materials and calculating abrasion by weight loss. Task groups of ASTM Committee D-20 have twice conducted round robin studies with the intent of incorporating weight loss as an approved procedure. In both studies, the between-laboratories reproducibility was so bad that it was decided to specifically recommend against any procedure based on weight loss.

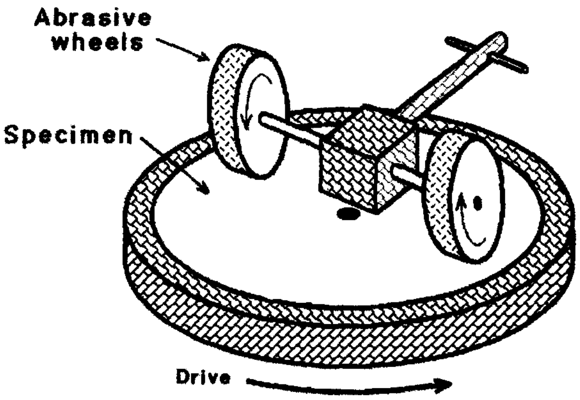

FIG. 3-29—Taber abraser.

NBS Abrader

There are a few other abrasion methods, but most of them are not very satisfactory. The chief problem is that the abrasive material becomes, itself, abraded during the test, causing poor repeatability.

An exception is ASTM D 1630, Test Method for Rubber Property—Abrasion Resistance (NBS Abrader). It is one of the few methods for which a reference material is available. The problem of the abrasive is handled by conditioning each new strip of abrasive paper with a specified break-in compound. This is followed by a second break-in run, this time using the specified reference material. Section 8.3 of D 1630 warns, "No more than 18 runs of three specimens each (excluding standard reference compounds run before each six tests) shall be made on one abrasive paper after the break-in runs."

Friction

ASTM D 1894, Test Method for Static and Kinetic Coefficients of Friction of Plastic Film and Sheeting, is the most widely used method for measuring friction. As illustrated in Fig. 3-30, a bottom specimen approximately 250 mm (10 in.) long by 127 mm (5 in.) wide is attached to an aluminum

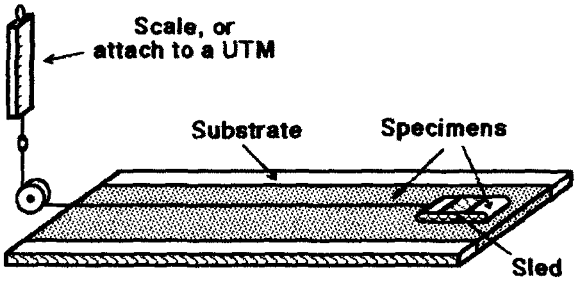

FIG. 3-30—Coefficient of friction, ASTM D 1894.

support plate. A top specimen is attached to a "sled," which is a metal block 63.5 mm (2.5 in.) square by approximately 6 mm (0.25 in.) thick. If the top specimen is a flexible film, it shall be approximately 120 mm (4.5 in.) square. It is wrapped onto the sled after first wrapping the sled with a piece of sponge rubber[5] 3.2 mm (0.125 in.) thick. If the top specimen is any other material, it shall be 63.5 mm (2.5 in.) square. The bottom and top specimens need not be of the same material.

Using a nylon filament,[6] the sled is attached, via one or more pulleys, to the crosshead of a UTM, which will record a curve of force versus time as the sled is pulled across the bottom specimen. (A spring gage may be used instead of a UTM.) Typically, the curve will have a maximum point (just as the sled starts to move), followed by a ragged horizontal track along some average lower value of force.

A. We draw a straight line through the center of this horizontal track in order to estimate this average force. We then divide the average force by the weight of the sled to calculate kinetic coefficient of friction.
B. We read the initial maximum force and divide it by the weight of the sled to calculate static coefficient of friction.

CREEP TESTING

The tests to be discussed in this section and the next two sections (on fatigue and weathering) are long-term tests. Unlike the tests we have discussed so far, these long-term tests are more likely to be used for design purposes rather than for quality control.

What is Creep?

Creep is permanent change of shape, induced over a long period of time, due to viscous deformation. In our discussion of viscoelastic behavior, we offered an example involving automobile seats, which are made of semi-rigid polyurethane cellular plastic. We noted that, over a long period of use, a seat will assume a permanent set.

Test Methods

The test method for creep is ASTM D 2990, Test Methods for Tensile, Compressive, and Flexural Creep and Creep Rupture of Plastics. Note that, in a creep test, we do not want the specimen to break. Instead, we want to measure its strain response, over a long period of time, under a constant load.

Flexural Creep

A flexural creep test is illustrated in Fig. 3-31. A constant weight is applied to the center of the specimen, and deflection measurements are than made and recorded according to the following time schedule: 1, 6, 12, and 30 minutes; then 1, 2, 5, 20, 50, 100, 200, 500, 700, and 1000 hours.[7] For creep tests extending beyond 1000 hours, the additional deformation is measured at least monthly. For screening purposes, tests are often terminated at 1000 hours, and the apparent modulus at 1000 hours is reported. However, for design purposes, the tests are carried out much longer—sometimes for years!

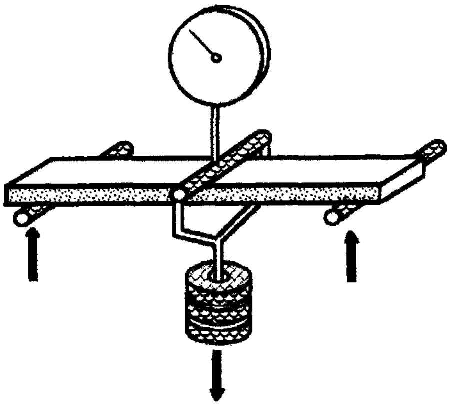

FIG. 3-31—Flexural creep setup.

Critical Requirements

A. The test site must be as free from vibrations as possible.
B. The temperature of the test space must be controlled within ±2°C (3.6°F) throughout the duration of the test. The relative humidity must be controlled within ±5% throughout the duration of the test unless it is known that the material is not sensitive to changes in humidity.
C. At the start of the test, the weight must be applied rapidly, but it must also be applied smoothly and gently so as not to shock the specimen and induce failure.
D. The deflection measuring device must be sensitive enough to measure the very small increments of deflection that occur in the latter stages of the test.

Methods of Plotting

A strain value is calculated from each measurement of deflection. (For the flexural mode, use the appropriate strain equation from ASTM D 790). Each strain value may be plotted against the elapsed time from the start of the test. This will produce a curve somewhat like Fig. 3-32*a*. However, there are more useful ways to plot the data.

Because the applied force is constant throughout the test, the stress is also constant throughout the test. In previous sections, we explained how to calculate stress. (For the flexural mode, use the appropriate stress equation from ASTM D 790.) For each measurement, we have already calculated strain for the above plot.

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[5] Caution 1—In the past, there have been problems in obtaining the sponge rubber backing material whose requirements are stated in Section 5.1 of D 1894. In a round robin study several years ago, it was found that the properties of the sponge rubber may seriously affect the test data.

[6] Caution 2—The method formerly allowed the use of a bead chain, as well as nylon cord, to pull the sled. In the above round robin, it was found that the bead chain gave results different from those obtained with the nylon filament.

[7] The reason for this seemingly weird schedule for reading data is that the creep of the specimen is quite rapid immediately after application of the load, but the rate of creeping diminishes markedly as the test proceeds.

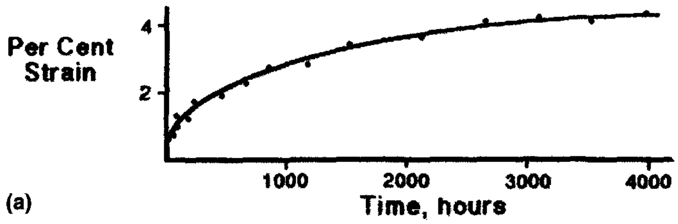

FIG. 3-32*a*—Plotting creep data as strain versus time on linear coordinates.

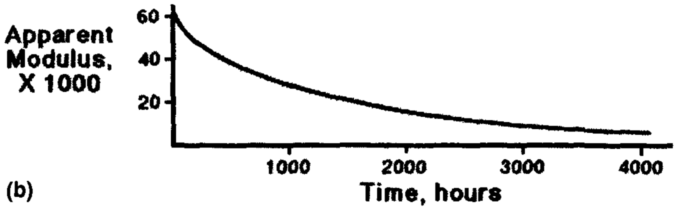

FIG. 3-32*b*—Plotting creep data as apparent modulus versus time on linear coordinates.

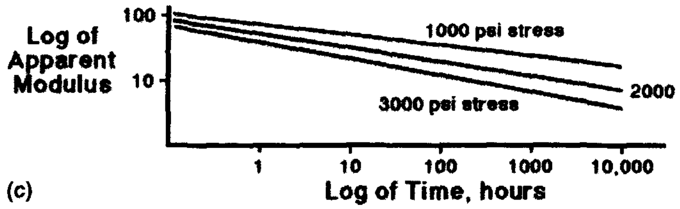

FIG. 3-32*c*—Plotting creep data as apparent modulus versus time on log/log coordinates.

In a previous section, we gave a general definition of modulus as "stress divided by strain." Therefore, for each measurement of deflection, it is easy to calculate an apparent modulus (creep modulus) value by dividing the constant stress value by the strain value. Plotting these apparent modulus points versus elapsed time from the start of the test will produce a curve somewhat like Fig. 3-32*b*.

A. Some people prefer to use "creep compliance," which is simply the arithmetic reciprocal of the creep modulus.

If, instead of plotting on linear coordinates, we plot the logs of the apparent modulus values versus the logs of the elapsed time values, a very interesting and useful thing usually results, as shown in Fig. 3-32*c*—the curves usually become straight lines. The utility of this is that, according to accepted practice, it is safe to extrapolate such a line for one decade. That is, if the test was conducted for 1000 hours, one may extrapolate the data to 10,000 hours; if the test was conducted for 10,000 hours, one may extrapolate the data to 100,000 hours!

Three lines are shown in the bottom plot of Fig. 3-32*c* in order to illustrate that, as one would expect, different dead weight loadings will produce different curves.

Compressive Creep

Creep tests may also be carried out in compressive and tensile modes, following the same precautions as listed previously in the section on "Critical Requirements." Compressive creep testing is about as simple as flexural; however, the same cautions as for static compressive testing must be observed also.

Tensile Creep

Although tests of flexural and compressive creep are easier to carry out than tests of tensile creep, design engineers for automotive companies and other companies often prefer to have data from tensile creep tests because the "finite element analysis" programs in their computers were written to accommodate tensile creep data only.

The equipment for tensile testing is more exacting because we need to measure the extension in the specimen's gage area only, as explained in the earlier section on static tensile testing. This may be done with extensometers, but a more precise technique is to mount a strain gage on each specimen.

Strain Gages

Strain gages are very small, thin, electrical resistors that may be adhered to the surface of the specimen. A low-power electrical current is then passed through the strain gage, and the amperage is measured by a very sensitive ammeter. When the specimen becomes distorted, a properly adhered strain gage experiences the same distortion as the specimen, and the resistance of the strain gage changes proportionately. This, of course, also changes the current slightly, as measured by the ammeter. This change of current is translated into a strain value.

The most critical parts of strain gage performance are:

A. Selection of the proper strain gage for the specific testing situation. There are many different size, shapes, etc. Rosettes are often (but not always) the best shape.
B. Adherence of the strain gage to the specimen. The technique for mounting the gage onto the specimen is very exacting! Before attempting it, one should attend a strain-gage mounting clinic sponsored by one of the strain gage suppliers.

Dummy Gage

When using a strain gage on a tensile creep specimen, the precision may be further improved by the following technique:

A. Mount a second strain gage on a second ("dummy") specimen made from the same piece of material.
B. Connect the electrical leads of the dummy gage to the ammeter, electrically in parallel with the first ("active") gage.
C. During the test, keep the dummy specimen as near as possible to the active specimen, but do not subject it to any loading or other distortion.

With this technique, the dummy gage will be distorted only by localized temperature fluctuations to exactly the same extent as the active gage. With the parallel electrical hookup, the effect of temperature fluctuations of the active gage will be compensated automatically by the signal from the dummy gage.

Creep Rupture Curves: A Pre-Requisite to Creep Testing

It is important to plan creep tests carefully to ensure that none of the applied loads will cause a specimen to break within the intended time span of the test. An inadvertent break after several weeks or months of testing can be an expensive loss! Therefore, we need to avoid excessively high specimen loadings. The following procedure, called creep rupture testing, will determine the limit of loading for a creep test:

A. Carry out a static test on several specimens in the same mechanical mode that will be used for the creep test. (Use test method D 695 for compressive, D 638 for tensile, or D 790 for flexural.) Calculate the stress at break for each specimen.
B. Next, insert a specimen in the creep apparatus.
C. Apply a deadweight loading at a stress level slightly less than the minimum breaking stress that was measured in Step 1. Proceed to record measurements of deformation and time, as with a creep test, until the specimen breaks. *Record the deformation and time just before the onset of fracture.*
D. Convert each deformation value to a strain value. Plot the values of strain versus time.
E. Repeat Steps A thru D several times, with slightly different deadweight loadings each time.
F. After a few specimens, the series of plots should start to look somewhat like those of Fig. 3-33*a* if the specimens are from a rigid material. Start drawing another curve, this one connecting the break points[8] (like the one proceeding downward from left to right in Fig. 3-33*a*).
G. Continue the above procedure until the curve through the break points seems to define an asymptotic limit of strain. Calculate and record the stress value which produced the strain at this asymptotic limit. Any stress below this level should result in a complete long-term creep test without breakage.

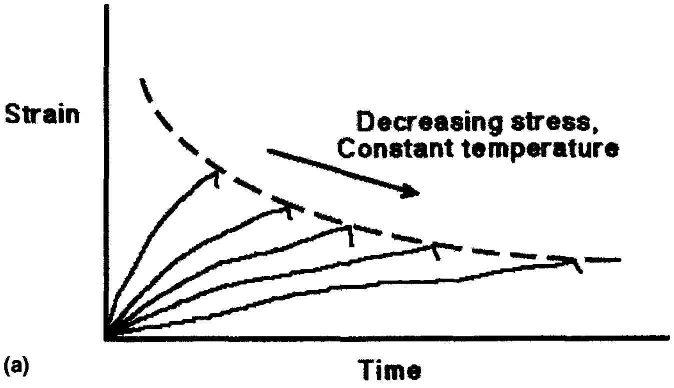

FIG. 3-33*a*—Creep rupture curves for rigid plastics.

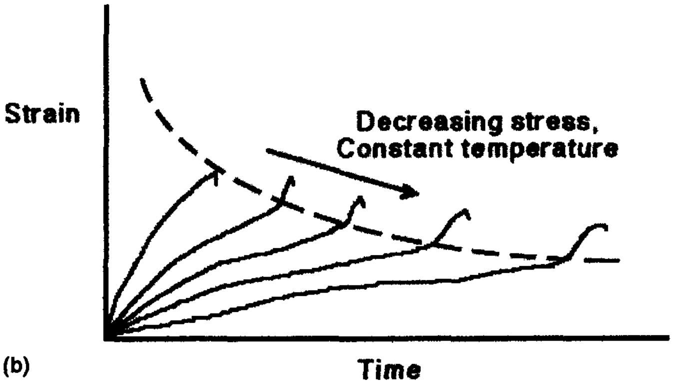

FIG. 3-33*b*—Creep rupture curves for non-rigid plastics.

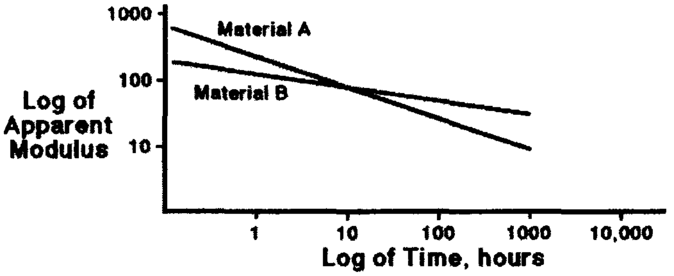

FIG. 3-34—Using creep data for material selection.

Importance of Creep Testing—an Example

Figure 3-34 shows a situation where:

A. The static modulus of "Material A" is greater than that of "Material B", but
B. After a period of time, the roles are reversed. A producer got into serious trouble several years ago by failing to take into account this possibility, as follows:
    1. A customer had determined that they needed a material with a modulus of 3.27 gPa (474,000 psi) in order to ensure the successful performance of a mechanical part which they were planning to manufacture. In view of this, the producer recommended one of its materials on the basis that its Young's modulus was 8.27 gPa (1,200,000 psi); this seemed to provide a safety factor of better than 2:1. The customer went into production with this material.
    2. About three months later, they were deluged with failed parts, to the consternation of all concerned.
    3. Then, someone decided to conduct creep tests on the material. Of course, you may imagine what they learned, much to the sorrow of all—after three months under creep loading, the creep modulus had dropped to only 2.64 gPa (383,000 psi)—far under the required modulus of 3.27 gPa (474,000 psi)!

Stress Relaxation

Stress relaxation is the converse of creep. It occurs in any situation where a plastic item is subjected to a constant

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[8] If the series of plots are from a non-rigid material, they may show inflections, as in Fig. 33*b*. Instead of connecting the break points by this new curve, connect the inflections to establish the asymptotic limit.

strain, rather than to a constant stress. In such a situation, it requires some initial amount of stress to induce the initial strain. Thereafter, however, the stress decreases as the strain is held constant. An example:

A. Many of us do-it-yourselfers have had the experience of replacing a radiator hose on our automobile. We tightened the clamps on the new hose, filled the radiator, then carefully tested the system to be sure that there were no leaks.
B. A few months later, however, we discovered a leak from the end of the new hose. We were surprised to find that the clamp was no longer as tight on the hose as when we installed it. We tightened it again, and thereafter we had no problem.

The reason there was no problem after the second tightening was not because we applied more force—it was because a stress-relaxation curve looks very much like a creep curve. The rate of relaxation is quite high at first, but it slows to a very slow rate after some period of time.

Stress relaxation data are very important to a manufacturer of material to be used for gaskets.

HEAT DEFLECTION TEMPERATURE, "HDT"

Figure 3-35 depicts the equipment setup for ASTM D 648, Test Method for Deflection Temperature of Plastics Under Flexural Load. Both the test method and the results are called "HDT." It is another of those tests that nearly all producers of thermosets feel they must run because everyone else has data on it. However, many people believe that the method has very little merit for the reason that we will explain below.

The specimen is, nominally, a rectilinear bar 127 mm (5 in.) long by 13 mm (0.5 in.) deep by any width [from 3 mm (0.125 in.) to 13 mm (0.5 in.)]. The actual depth and width of the specimen is measured, and the cross-sectional area is calculated.

The specimen is placed in an oil bath. It is supported near each end, and a deadweight load is applied at the center, sufficient to produce a maximum fiber stress of 1820 kPa (264 psi) in the specimen. A dial micrometer, which measures the position of the center of the specimen, is set to "zero." A heater and a stirrer in the oil bath are then activated.

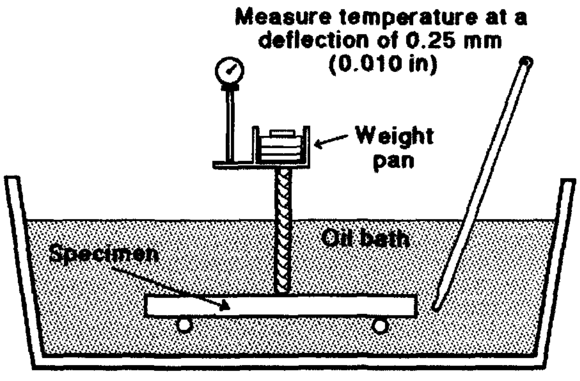

FIG. 3-35—Heat deflection temperature (HDT), ASTM D 648.

The temperature of the oil bath is monitored until the dial indicator shows that the center of the specimen has deflected by 0.25 mm (0.010 in.). The temperature at that time is recorded. This temperature is the HDT.

Rate of Temperature Rise

The rate of temperature rise of the oil must be tightly controlled so as to maintain a uniform rate of rise of 2 ± 0.2°C (3.6 ± 0.35°F) per minute. Maintaining this tolerance is the most important factor in the test. This factor is often highly dependent on what oil (or other liquid) is used. It is imperative that the rate of temperature rise be specially monitored, and adjusted if necessary, when the equipment is first put into service, and at scheduled intervals thereafter.

Productivity

The HDT test method is probably the best candidate for automation of any piece of equipment in a physical testing laboratory. Automated HDT equipment has been available for some time.

If the equipment is not automated, the technician must constantly watch it until the HDT is reached. Some materials will take an hour and a half to do so; others may take only a few minutes. There usually is no movement of the dial indicator until shortly before the HDT is reached—then it moves quite rapidly; you may miss the HDT if you are away from the equipment for as little as one minute at the wrong time!

Multi-Point Data

The main reason for including a description of the HDT method in this volume is to make the following point—for any item of interest, multipoint data are much more reliable than using single-point data.

Note that D 648 is a form of creep testing, but only a single point on the deflection-versus-time curve is measured. Because of this, HDT data should be used very judiciously, if at all. It is probably useful as a quality control tool, but one should not rely on HDT data for developmental work or problem solving.

Consider the example shown in Fig. 3-36. The creep moduli of a "Material C" and of a "Material D" are plotted against temperature. The HDT for each of these plastics is also shown as a point on the creep modulus curve.

A. You ask, "How do we get modulus from HDT?" We have standardized the stress at 1820 kPa (264 psi), and the end point of the test occurs at a deflection of 0.25 mm (0.010 in.), which is a strain of 0.001875 mm/mm (in./in.). [*Modulus equals stress divided by strain, so the modulus* at the end point of the test is always approximately 0.971 gPa (1.408 × 10^5 psi).]

Note that the HDT for Material C is considerably higher than for Material D. Therefore, if we looked only at the HDT values, we would conclude (incorrectly) that Material C is a

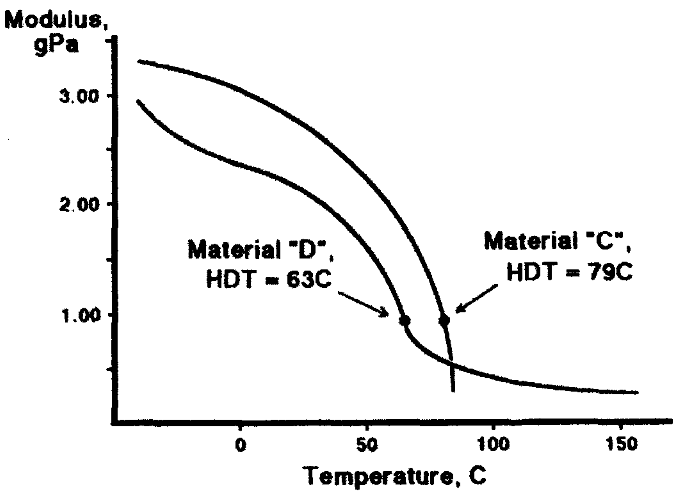

FIG. 3-36—HDT versus creep modulus.

better candidate for use at higher temperatures. However, when we look at the additional information available from the plots of modulus versus temperature, we see that Material D actually maintains more strength at temperatures above the HDT than Material C does.

FATIGUE TESTING

What is *fatigue* of a material? We have all seen cases where a can opener has cut all around the top of a tin can, except for a short section where the top remained hinged to the side. Most of us have found that, if we bend the top back and forth repeatedly, like a hinge, the top will eventually separate from the side. The mechanism that slowly allows the complete separation is fatigue. Most materials, including plastics, will fatigue if subjected to repeated cycles of loading and relaxation or of loading and reversed loading.

The loads used in fatigue testing must be low enough so that the specimen will not break in just one cycle. As with other types of fracture, the failure mechanism starts with the formation of a small crack; under repeated small strains, the crack grows until it results in complete fracture.

There are special machines for some specific fatigue testing methods, but the type of machine for general fatigue testing is a servohydraulic machine, which looks much like a UTM, but is more sophisticated and thus more expensive. (Servohydraulic machines may be used as UTMs, but do not try to do fatigue testing on a UTM—UTMs are not designed for it, will not cycle fast enough, and will wear excessively.)

Fatigue testing may be done in any of the four modes of mechanical testing. However, as with creep testing, design engineers for automotive companies and other companies often prefer to have tensile fatigue data because the finite element analysis programs in their computers were written to accommodate tensile fatigue data only.

In the following, we will describe tensile fatigue testing. However, the only difference between testing for tensile fatigue and fatigue in the other three modes is in the specimen geometry and the hardware for holding the specimens.

Test Setup and Operation for Tensile Fatigue

In general, a standard tensile specimen may be used for tensile fatigue, although some people use a specimen even larger than the D 638 Type III specimen. The specimen is gripped similar to the way it is held in a static tensile test. The servohydraulic machine has controls for stretching the specimen to a preset maximum value and then relaxing it to a preset lower value under any of the following procedures:

A. Under load control, the control panel monitors the load on the specimen and reverses the direction of crosshead travel whenever the preset maximum load or the preset minimum load is reached.
B. Under extension control, the control panel monitors the output from an extensometer attached to the specimen and reverses the direction of crosshead travel whenever the preset maximum extension or the preset minimum extension is reached.
C. Under crosshead control, the control panel directly monitors the crosshead position and reverses the direction of crosshead travel whenever the crosshead reaches the preset limits of upward crosshead travel or downward crosshead travel.
D. Combination control:
   1. The control panel may be set to monitor any combination of the above three types of control. For example.
      a. It may be set to monitor the load during upward crosshead travel, then switch to crosshead control for downward travel.

Regardless of which type of control is chosen, the crosshead must not be allowed to return back to its starting point because to do so would allow the specimen to slip in the grips and possibly fall out.

Procedure

The first step is to measure the static tensile strength of the material. Carry out a static test on five specimens according to D 638. Calculate the average stress at break. (For compressive mode, use Test Method D 695. For flexural mode, use Method D 790.)

Let us assume that we are going to run our fatigue test under load control. We place a specimen in the grips and dial into the machine the maximum and the minimum load limit for cycling. The maximum limit that we choose must be such that the specimen will not be stretched to its static breaking strength, otherwise the specimen will break immediately, and the data from that specimen must not be used. One of the things that must be reported with the test data is either:

"R," the ratio of the maximum load limit to the minimum load limit, or
"A," the ratio of the stress amplitude to the mean stress.

We also must set into the machine the frequency of cycling. While metals are often tested at frequencies as high as 600 Hz (cycles per second), the frequency for plastics must be limited to about 3 Hz in order to prevent premature failure due to heat buildup.

After checking to be sure that the cycling counter on the control panel is set to zero, we start the machine and let it cycle until the specimen breaks. Depending on the material and the maximum stress level that we have chosen, this may take anywhere from a few seconds to several weeks (or even months, possibly). We then plot the dialed-in maximum stress versus the log of the number of cycles to failure ("N"), as shown in Fig. 3-37.

We then choose a different maximum load level, a minimum load level to maintain the same "R" value, and repeat the sequence of cycling-to-failure and of plotting the result. We continue to repeat this procedure until we have enough data points to accomplish the following:

A. We draw the best smooth curve that we can that will represent the data points (see Fig. 3-37). We need enough data points to establish a curve out to the number of cycles with which we feel comfortable for the specific purpose of the test.

   1. The value of the static breaking stress should be used as the first data point. To do so, plot the average value of the static breaking stress on the "Y" axis versus N = 1/2 cycle on the "X" axis.

The smooth curve is known as an S-N curve, and it is the usual way of presenting fatigue data. If we carry the curve out far enough, it may establish an asymptotic limit.

Modulus Decay

Some people gather another set of useful data while performing fatigue testing. At several arbitrary numbers of cycles (before the specimen breaks, of course) they stop the cycling temporarily. They attach an extensometer to the specimen (see the earlier section on static tensile testing), connect it to an X-Y chart recorder, and record load and deflection for a single fatigue cycle. Then they resume the fatigue test. From the load/deflection curve, they calculate a modulus and plot it versus the log of the cycles to that point on the same chart as the S-N data. These modulus points establish a curve of modulus decay.

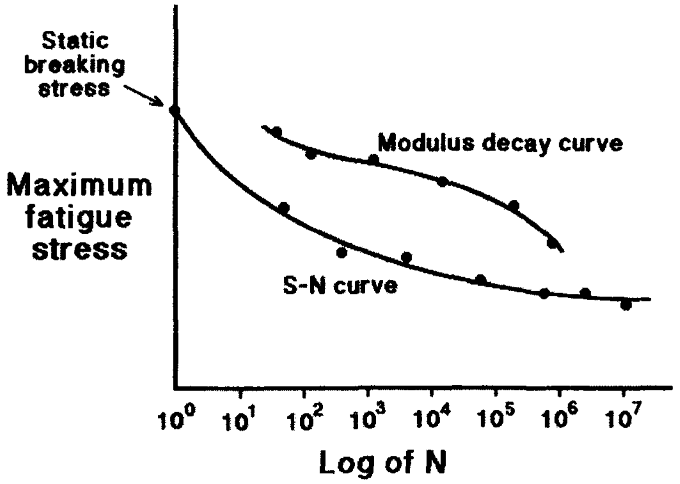

FIG. 3-37—Plotting fatigue data.

ASTM D 671

By comparison with the above, we should also mention ASTM D 671, Test Method for Flexural Fatigue of Plastics by Constant-Amplitude-of-Force. This method is based on a specific commercial apparatus, which flexes the specimen back and forth in cantilever bending, much like bending the lid of the tin can. The specimen is of a unique size and shape, entirely different from that used for other tests.

Although all of the texts on fatigue testing of plastics caution against using frequencies higher than about 5 Hz because of heat buildup, this apparatus operates at 30 Hz and cannot be operated at any other frequency. Section 7.3 of D 671-93 does state, "The temperature at the region of the highest stress in the specimen shall be measured and recorded."

A. Caution—It is not valid to compare data from this test with data from flexural fatigue run in a D 790 configuration because:

   1. The D 790 configuration is a three-point-bending geometry; D 671 is a cantilever beam geometry.
   2. The specimen geometry is entirely different.
   3. The high frequency causes excessive heat buildup.

D 671 is not widely used. In the author's opinion, the chief value of continuing to carry it as an ASTM method is that its Appendix X3 is a good source for descriptions of fatigue testing terms.

WEATHERING

Plastics that are exposed to the outdoor elements over periods of time usually exhibit deterioration of mechanical properties, as well as fading, to some degree. Some fare very badly, while others do remarkably well. A few years ago, one of the senior engineers of Disney World gave a very interesting and informative talk to ASTM Committee D-20. Among other things, he emphasized that the top portions of most of the buildings in the Magic Kingdom are made of reinforced plastics. The engineer was very pleased with their performance, and he made a particular point of stating how well the beautiful blue color of Mickey's Castle had held up over a period of several years.

Water and ultraviolet light are the chief culprits in weathering, with UV being the most severe problem. With regard to water, the purer it is, the more readily it will attack some plastics. There are companies in Arizona and Florida who specialize in conducting outdoor exposure tests for clients. These are very long-term tests, so it is logical to try to find accelerated test procedures that will predict long-term results.

ASTM G 23, Operating Light- and Water-Exposure Apparatus (Carbon-Arc Type) for Exposure of Nonmetallic Materials, has been used for many years. Specimens are mounted inside a large cabinet and exposed to light generated by an arc struck between carbon electrodes. A newer method is ASTM G 26, Operating Light-Exposure Apparatus (Xenon-Arc Type) With and Without Water for Exposure of Nonmetallic Materials. It is basically similar to G 23, but more sophisticated. Each of these items of equipment is commonly called a "weatherometer." Specimens may be exposed round-the-clock by either method.

The Xenon lamp produces a spectrum that is significantly richer in the shorter wavelengths of ultraviolet light than the carbon arc; thus, it is considered to be a more severe test. Test results from the carbon arc are not comparable with results from the Xenon arc. Either piece of equipment costs a few thousand dollars, and both are rather expensive to maintain—both pieces of equipment require daily handholding, and the water exposure eventually corrodes metal parts in the interior of the cabinet.

COMPUTERIZATION OF TESTING EQUIPMENT

There now exist hundreds of items of physical testing equipment whose output is recorded directly by a computer program, which then does whatever calculations are needed and prints out both the raw data and the test results. In many cases, the computer program also controls the testing equipment during the test. Although the information offered below may suggest differently, the author is very much in favor of using such systems to improve both accuracy and productivity. However, the author is also concerned with the likelihood that many people may adopt such systems with unrealistic expectations of them.

In most cases, computerization will improve productivity. In a few specific applications, such as the HDT method, the improvement will be dramatic. However, with regard to the great bulk of testing on UTMs, it is common for people to assume that such computerization, in itself, will improve productivity to a greater extent than it really does. And it is a rare person, indeed, who will admit that the system is not living up to expectations, if he or she is the one who recommended its adoption in the first place. In many cases, this person is not even aware that there are problems even after the system has been in operation for a long time.

This section is addressed mainly to those people who may be charged with implementing or supervising a computerized system, particularly for a UTM.

Usually, if you are contemplating the computerization of a UTM, the worst thing you can do is to go to the computer professionals within your company and ask them to undertake the job. The chief problem is not in hooking up the computer to the test equipment; that is relatively easy. The propensity for generating false facts lies in the software (which is, collectively, the programs—that is, the sets of instructions that tell the computer what to do, very specifically, from one step to the next).

Software Problems

The chief problem, which most people do not realize, is that it is impossible for a computer programmer to write foolproof algorithms that will cause the computer program to identify correctly the intended points on the load/deformation curve every time, in all cases. If you are a programmer, don't get bent out of shape at this point—read on! The truth of this is not immediately evident.

What We Used to See on a Chart Recorder

When one looked at the relatively smooth load/deformation curve that was generated on a chart recorder, as was done for dozens of years before the advent of computers, the following features usually seemed obvious to us:

A. The point at which to measure breaking load and deformation.
B. The point at which to measure yield load and deformation.
C. Whether there was, or was not, a straight portion of the curve. If there was, we could easily draw a straight line through it in order to choose a point for calculating tangent modulus.
D. How to correct the curve to eliminate the "toe," so that we had a valid starting point for measuring deformation at break or at yield.

There were often, of course, strange wiggles in the curve that might baffle us momentarily, but we had the mental ability to evaluate the general picture that we were seeing, and so we could easily decide how to analyze each curve.

What the Computer Sees

By comparison, what the computer program actually has to deal with is not a smooth curve (Fig. 3-38), but a series of individual dots that are far more inconsistent than the worst curve generated by a chart recorder. (The inherently slower response time of a chart recorder results in a relatively smooth curve.) The problem for the computer program, then, is to:

A. Identify the dot that best fits the overall pattern in the segment that contains the true break point or yield point.
B. Decide whether the wiggly series of dots at the very beginning of the curve is still part of the toe or whether it is a valid part of the curve.
C. Decide whether it is valid to draw a straight line through the wiggly series of dots after the toe.
    1. If it is, calculate the proper slope of the line for use in calculating tangent modulus. Then, extend the line down to the base and remeasure all of the distortion values from this point.

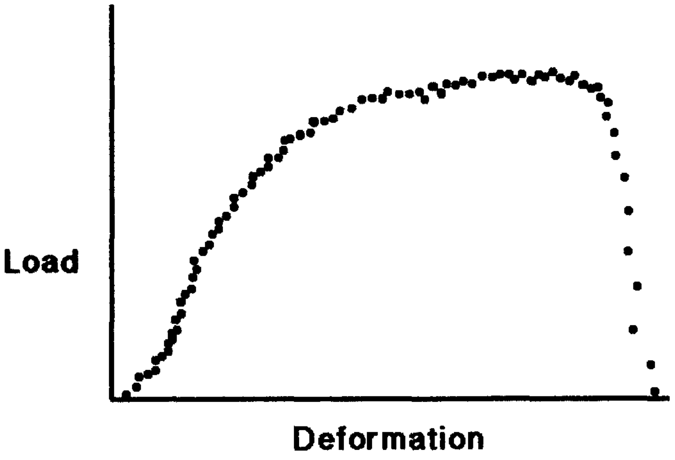

FIG. 3-38—Computerized testing—what the computer "sees."

2. If it is not, reject the calculation of tangent modulus. Use some other means for making the proper toe corrections.

Algorithms (Decision-Making Sequences)

Most computer programmers are not qualified to write satisfactory algorithms to perform the above unless they have a lot of experience in evaluating load/deflection curves. Even with such experience, it is impossible to write algorithms that will, for every possible curve, cause the computer program to perform all of the above tasks perfectly. Remember, the algorithm is dealing only with various combinations of dots, not a smooth, intelligible curve. Some particular wiggle, which the human brain would readily understand and dismiss, may fit the wrong algorithm (or part of an algorithm) in the program. With the enormous variety of possible wiggles that may occur in load/deformation curves, the algorithm is bound to pick a wrong point or a wrong modulus line now and then.

Acquiring a Computerized System

If you are considering installing a computerized system, the best thing you can do is to consult at least two companies who specialize in these applications and buy a software-and-installation-and-training package from one of them. Almost every vendor of testing machines now advertises the availability of computerized systems. Some vendors are more experienced, and more practical, than others. Some may still be relatively inexperienced.

Guidelines in Making Your Selection

A. Be sure that the package includes these features:
   1. The load/deformation curve must be displayed progressively on the computer's monitor as it is being generated during the test.
   2. The display must include an auto-scaling feature so that the entire curve will be visible on the monitor regardless of what the initial settings of full-scale load and deformation are.
   3. The modulus line, the yield point, the break point, and any other point that the computer program has chosen must then be superimposed on the curve on the monitor in order that the technician may evaluate whether the computer program has chosen an obviously incorrect point or modulus line or failed to choose a point that it should have. For the reasons explained in an earlier section, these errors will most assuredly occur occasionally, and there is not always a pattern to them.
   4. There must be an easy procedure available by which the technician may correct an obviously incorrect point.
   5. There must be a feature that allows the technician to enlarge any segment of the curve in order to carry out the above evaluations and corrections.
   6. The computer program must then automatically recalculate any data based on an incorrectly chosen point.
   7. After the curve has been approved by the technician, the entire curve must be stored for an agreed period of time.
   8. There must be provision for retrieving and displaying the entire curve at a future time and making further corrections, if they are warranted.
      a. Ascertain how easy it is to retrieve and re-examine individual curves without having to retrieve several others as well.

B. Ascertain whether the data, but not the curves, may be electronically transferred to another computer easily.

C. Find out how easy it will be to revert to manual testing if the system breaks down, as it will sooner or later. As Murphy said, "Nothing's perfect!"

D. Ask the prospective vendor for a list of customers who have bought their package. Check with some of the people on that list to get their comments.

Anticipate Problems

Be prepared for the computerized system, on balance, to yield less increase in productivity than you thought it was going to. Remember:

A. When the system breaks down, someone has to fix it.
   1. Who will that be if it is a software problem?
   2. Who will that be if it is a hardware problem?
   3. In either case, how long will it take?
   4. Meanwhile, how will you operate?

B. It will take a fair amount of training before your technicians will be able to operate the computerized system competently and comfortably. The technician must become computer-literate to some degree in order to use the system effectively.

C. While the computer relieves the technician of reading data from charts and calculating results, it imposes other restrictions on how he or she must carry out the test procedure. Therefore, you lose some flexibility in how the tests may be adapted for unique situations.

Computer-Generated Data Versus Manually Generated Data

Computer-generated data usually differs from manually generated data to some degree or other. Which is correct? It may be impossible to know.

A. On the one hand, the computer should eliminate mistakes in calculating results and transcribing numbers.

B. On the other hand, from the information in the earlier section on "Software Problems," we must realize that sometimes the point on the curve, which the computer program chose for the calculation, may be slightly different from what the point would be if it were chosen manually.

Instant Stress/Strain Curves

Before computers, it was a very time-consuming chore to convert a load-deformation curve into a stress-strain curve, so actual stress-strain curves were seldom drawn. And, as noted early in this chapter, load-deformation curves may have many different data scales, so they are difficult to compare against each other. Therefore, the user of the test data seldom even saw such a curve.

The computer, however, can instantly generate a stress-strain curve for each test specimen. All of the curves from a complete set of specimens may easily be printed out on a single sheet of paper and issued in conjunction with the test report. This is highly recommended, especially when generating data to be used in a development or "problem-solving" situation. Once a development chemist or technician gets used to what he or she is looking at, one glance at such a set of stress-strain curves may be much more informative than all of the numbers that were generated from their various parts!

AUTOMATION OF TESTING EQUIPMENT

The natural extension of the computerization of test equipment is to automate the specimen handling. This is accomplished with a robot, under computer control, which:

A. Picks up a specimen from a rack. The specimens have already been placed in racks in the order in which they are to be tested.
B. Submits the specimen, properly oriented, to an auto-reading micrometer or dial gage for measurement of the width and thickness at the center of the specimen. If weight is a factor to be recorded, the specimen is placed on an auto-recording laboratory balance. These inputs are automatically stored by the computer program.
C. Orients the specimen in the proper position in the test fixture. The computer program then controls the testing machine during the actual test and records the test data.
D. Removes the broken specimens from the test fixture, if necessary, and discards them.

Advantages and Cautions

Obviously, such a system has the potential for significant savings in a high-volume quality control laboratory. Such a system is capable of running tests 24 hours a day, seven days a week, and it doesn't even take coffee breaks. Such a system should certainly be used to improve technicians' productivity.

However, it is a bad idea to set it up for the night shift and let it run itself with no technician to monitor it for the following reasons:

A. Scurrilous load/deflection curves may be generated, from time to time, caused by such things as:
    1. Slippage of the knife edges of an extensometer. This will immediately be obvious to an experienced technician checking the monitor, but no algorithm in the program will recognize it.
    2. Internal flaws in a specimen. Data from such specimens will be flagged by the computer program only if the results fall below specification requirements. Properly assigning the cause to a sub-standard specimen will only occur if the broken pieces are identified and examined.
B. As explained earlier, the algorithms of the computer program are going to pick incorrect points from which to read data from time-to-time. Many of these will be immediately obvious, and easily correctable, to an experienced technician checking the monitor. Without review of the data, some of them will be incorrect. If they go into a database, they will be treated as gospel truth, rather than false facts.
C. Some data points might be identified as possible outliers, if proper statistical evaluation routines are included in the software. However, as we cautioned in Chapter 3, no such data should be rejected unless an actual physical cause for it is identified!
D. Some part of the system may malfunction occasionally. If this is undetected for a period of time:
    1. There is the possibility of damage to untested specimens.
    2. The testing schedule may be set back by several hours:
        a. Critical shipments may be delayed.
        b. The lack of quality control feedback may cause large quantities of off-specification material to continue to be produced before the problem is discovered and corrected.

Development Testing and Non-Routine Curves

In a laboratory that is devoted to product development and customer service testing, the laboratory's most valuable resource is the knowledge and experience of its personnel. In the author's experience, some of the most important information to come out of the testing lab has been when the technician, examining the load/deflection curve, has noticed some unusual profile in the curve that would not have been detected by any of the algorithms of a computer program. In many cases, the technician has immediately invited the concerned development chemist to come and witness the testing of the remainder of the specimens, thus offering valuable insight that the chemist would not have received through routine reporting of test data.

In any development lab, this valuable asset should be preserved, through continued review by a real, live qualified person, of load/deformation curves and test data, whether automated or not!

BIBLIOGRAPHY

Handbook of Plastics Test Methods, 2nd ed., by R. P. Brown, George Godwin Ltd. in association with The Plastics and Rubber Institute.
Handbook of Plastics Testing Technology, by Vishu Shah, John Wiley & Sons, 1984.
Introduction to Polymer Viscoelasticity, by Aklonis, MacKnight and Shen.
Mechanical Properties of Polymers, by L. E. Nielson, Reinhold.
Mechanical Testing of Plastics, 2nd ed., by S. Turner, George Godwin Ltd. in association with The Plastics and Rubber Institute.
Physics of Plastics, by Ritchie; Van Nostrand.
Testing of Polymers, Volumes 1 thru 4, by Schmitz & Brown, Interscience Publisher.
Viscoelastic Properties of Polymers, by Ferry.

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4

Responses to Flame Exposure

by Alvin J. Flint, Jr.[1]

INTRODUCTION

THERE ARE DOZENS OF small-scale test methods that measure response, of one kind or another, to a flame or to some other heat source, or both. However, there is no test that will faithfully predict how a specific material or assembly will respond to an actual fire scenario. The reason is that the behavior of actual fires is not predictable except in very broad terms. Each actual fire represents a complex interaction of:

A. Ignition source.
B. Ease of ignition of the materials present.
C. The oxygen supply.
D. The geometry of the fire site.
E. The flame spread rate and fuel contribution of the heated materials, etc.

We can only approximate some of those actual conditions, using rather large-scale tests. Because large-scale tests are very expensive, dozens of different smaller-scale test methods have been designed by various people, over a period of many years, to try to find a correlation between a small-scale test and a large-scale test. Almost no reliable correlations have been found except for some exceptions that are specific to certain material formulations.

However, unwanted fires are a never-ending threat to all of us in terms of life and health as well as property loss. Therefore, it is necessary to do the best we can, with what technology and tests that we have, to maximize protection for all of us against harm and loss.

This chapter gives a brief description of some of the more widely used tests—ignition, flame-spread, smoke generation, heat release, and toxicity, along with some cautions regarding safety in the laboratory.

We wish to re-emphasize that, regardless of how complete a description of any test method is given here, it is essential that you study the method thoroughly before attempting to carry out any test!

Jurisdictions

Any ASTM combustibility standard that is not specifically applicable to a stated type of material, such as plastics, falls under the jurisdiction of Committee E-5 on Fire Testing. That committee, of course, has members from a tremendous variety of interests—wood, steel, concrete, carpeting, plastics, rubber, textiles, etc. Committees whose jurisdiction is limited to a specific type of material or product, such as D-20 on Plastics, may write combustibility methods that apply only within their material or product jurisdiction. Committee D-20 has written several test methods for combustibility that are specific to plastics.

SAFETY DURING TESTING

In addition to the safety precautions listed in Chapter 3, it is important to enforce the following precautions before anything is burned in the laboratory:

A. Burn materials only in a properly exhausted laboratory hood. All materials that contain carbon in their molecular structure will generate carbon monoxide gas when they are burned in a normal atmosphere. Carbon monoxide is a deadly poison, and it is both odorless and colorless. In many other cases, additional poisonous or noxious gases are also created. Do not breathe products of combustion from any material!
    1. Many of the test methods call for the exhaust system of the hood to be turned off while burning each specimen and turned on again immediately after burning each specimen. However, most of the methods allow the exhaust system to remain on if the specimen is placed inside a metal cabinet, which is placed inside the hood. The cabinet must have a glass door. It also must have ventilation holes in it sides, near the bottom and near the top, to prevent drafts from the exhaust system from passing across the specimen. The author strongly recommends the use of such a cabinet, leaving the exhaust system turned on all the time.
B. Be sure a proper fire extinguisher is readily at hand. Before each shift of testing, check to be sure it is properly charged.
C. Make sure that there are no other flammables lurking in the exhaust hood. This includes both fluids and solids in bottles and cans, as well as loose stuff.
D. Check all gas connections for leaks, using a soap solution or some other detector.
E. Make sure that all steel gas bottles are stored vertically and securely strapped to a solid support that will prevent them from tipping. A loose gas bottle becomes a lethal torpedo if the valve breaks off.

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[1] Formerly, supervisor of the Physical Measurements Laboratory, ICI Polyurethanes, West Deptford, NJ. Now retired. Present address: 109 Hoiland Drive, Shipley Hts., Wilmington, DE 19803-3227.

A LABORATORY TEST VERSUS TWO ACTUAL FIRE HISTORIES

As stated at the beginning of this chapter, there is no test that will faithfully predict how a specific material or assembly will respond to an actual fire scenario. This is borne out in the following two examples.

Actual Fire History No. 1—The Boardstock Factory

Boardstock (also called "foamboard") consists of slabs of rigid cellular plastic, with facings of heavy paper, thin plastic, aluminum foil, or some other thin material adhered to both sides. It is a building construction product. Boardstock is usually about four feet wide by eight feet long by one-half inch to three inches thick.

About 1967, a fire broke out around 2:30 a.m. in a boardstock manufacturing plant operated by Atlas Chemical Industries in Pennsauken, NJ. It took firemen three hours to bring the blaze under control. The building was not equipped with a protective automatic sprinkler system, and the building itself was a total loss. The heat inside was so intense that steel girders, which had formerly supported the roof, softened and sagged down as far as they could go.

When the building finally cooled down enough for people to enter, everyone was astonished—the sagged steel girders were resting on stacks of boardstock, still largely intact! Only the outsides of the stacks were charred!

Based on the author's memory of the event, he recollects that this was particularly astounding because the foamed material in the core of this particular product was rated only "self-extinguishing," rather than "non-burning," according to ASTM D 1692.

Test Method D 1692

This was the laboratory bench test method that was most commonly used by all foam manufacturers at that time. It used a specimen 152 mm (6 in.) long by 50 mm (2 in.) wide by 13 mm (0.5 in.) thick, which could not have any "skins" or other coatings on it. An ink line was drawn across the width of the specimen 25.4 mm (1 in.) from each end. The specimen was placed on a piece of steel hardware cloth and lighted at one end by a Bunsen burner with a wing-tip flame. The spread of the flame front along the specimen was then observed:

A. If the flame reached the second ink line, the material was reported as "burning according to ASTM D 1692."
B. If none of five specimens burned to the second ink line, but the flame reached the first ink line, the material was reported as "self-extinguishing according to ASTM D 1692."
C. If none of five specimens burned to the first ink line, the material was reported as "non-burning according to ASTM D 1692."

Actual Fire History No. 2—The Boardstock Do-it-Yourselfer

Shortly after the above fire, a man bought some boardstock that was labeled "Self-extinguishing according to ASTM D 1692." Reasoning that "self-extinguishing" meant that it was safe to install it in a room in one's home, he installed it. He did not put any protective barrier in front of it, as he did not know that approved installation procedures called for an additional facing to cover the boardstock.

Later on, there was a fire in his home. His two children were killed. He sued on the basis of the "self-extinguishing" label. He collected, and the judge saw fit to make statements in regard to D 1692 that were derogatory, to say the least. Naturally, as the material was a form of plastic, the case was widely publicized.

As a result of this case, ASTM adopted a policy to the effect that terms such as "self-extinguishing," "non-burning," "nonflammable," "fire retardant," or any other descriptive term that could possibly be misinterpreted must not be used to describe any material in an ASTM standard. All test methods dealing with response to heat or flame must produce a result that must be described in numerical terms only, such as "a burning rate of 2.3 mm/min," or "a burning distance of 3.6 mm." At the same time, ASTM also discontinued D 1692 (but see next section).

WHAT COMBUSTIBILITY TESTS CAN TELL US

If small-scale tests cannot predict how a material will behave in a real fire, what good are they? Any test method associated with fire or combustibility is a useful method if it is capable of producing data that are useful for making valid comparisons between the flame response of different materials or different formulations.

Even with the much-maligned D 1692 test, in fact, this was the case. The method provided a clear basis for distinguishing a very bad formulation from a very good one—so much so that manufacturers have continued to use the method on a non-certifying basis. In fact, the method has now been rewritten, as D 4986, with the test results being reported in numeric terms, of course.

Table 1 categorizes some of the test methods that are the most commonly used in the plastics industry. In the next few sections, we will provide some guidance in regard to most of them. We will approach them in a different sequence from that of the table, however.

In general, one should compare test results only among specimens of approximately the same thickness, as different thicknesses generally yield significantly different data.

TESTING THERMOPLASTICS—A PROBLEM

Thermoplastics tend to melt, drip, sag, shrink, etc. when exposed to an igniter flame or to a radiant heat source. Some producers of thermosets tend to feel that these phenomena

TABLE 1—Some Commonly Used Combustibility Tests.

	Ignition	Flame Spread	Heat Release	Smoke	Specific to Plastics? NC	Solid	Cellular	Flexible	Films
LARGE-SCALE TESTS									
Full-Room Burn		X	?		X	X	X	X	
Factory Mutual Corner		X	?		X	X	X		
ASTM E 84 Steiner 25-ft. Tunnel		X	(SEE TEXT)		X	X	X		
Others					X				
MEDIUM-SCALE TESTS									
ASTM E 162 Radiant Panel		X	(SEE TEXT)		X	X	X		
ASTM D 3675 Radiant Panel							X	X	
SMALL-SCALE TESTS									
ASTM D 1929 Stechkin Furnace	X					X	X	X	X
ASTM D 3713	X					X			
ASTM D 635 & Ul 94		X				X		X	X
ASTM D 2863 Oxygen Index	?	?				X	X	X	X
ASTM D 3801 & Ul 94	?					X		X	X
ASTM D 5048						X			
ASTM D 3014 Butler Chimney		X					X		
ASTM D 4986 (formerly D1692)		X					X	X	
MVSS 302		X					X	X	
California Bulletin 117		X					X	X	
HEAT RELEASE RATE (FUEL CONTRIBUTION)									
ASTM E 906 (smith, OSU)			X		X	X	X		
ASTM E 1354			X		X	X	X		
SMOKE TESTS									
ASTM D 2843 XP-2 Chamber				X		X	X	X	X
ASTM E 662 NBS Chamber				X	X	X			
TOXICITY TESTS									
Pittsburgh test					X				
ASTM E 1678					X				

SEE ALSO:
ASTM D 3814, Standard Guide for Locating Combustion Test Methods for Plastics
ASTM D 5025, Standard Specification for a Laboratory Burner

	Ignition	Flame Spread	Heat Release	Smoke	Specific to Plastics? NC	Solid	Cellular	Flexible	Films
CORROSIVITY TESTS									
ASTM D 5485				X		X	X		

bias combustibility test data in favor of thermoplastics when they are compared with data for thermosets. For this reason, some test methods exclude thermoplastics from their scope. In the case of the other test methods, these phenomena create obvious problems in running the test. In general, if the material recedes away from an igniter flame in any way, the technician should "chase" the specimen by moving the burner to keep the igniter flame in contact with the specimen. Thermoplastics producers may feel that this practice tends to favor thermosets.

With some methods, a strip of cotton is placed beneath the specimen. If flaming drops from the burning specimen ignite the cotton, it must be so stated in the test report. In some methods, this behavior is reflected in how the material is classified.

LARGE-SCALE FIRE TESTS

ASTM E-84

ASTM E 84, Test Method for Surface Burning Characteristics of Building Materials, is probably the best known of all of the larger combustibility tests in existence, as it is cited in most building codes throughout the United States. Its scope includes all building materials, not just plastics. It is usually referred to as "the 25-foot tunnel" or, sometimes, just "the tunnel test," even though there are several other smaller tunnel tests: a 2-ft tunnel, a 4-ft tunnel, an 8-ft tunnel, etc. It is also often called "the Steiner tunnel," in honor of Dr. Al Steiner, who developed the method at the National Bureau of Standards (now NIST) many years ago.

The test chamber is, indeed, a tunnel. Its sides and base are made of fire brick. The specimen, which must be 25 ft long,[2] comprises the ceiling of the test chamber. The usual specimen width is about 500 to 560 mm (20 to 22 in.). Two gas burners spout igniter flames against the bottom of the specimen at one end of the tunnel. The flames are 1.37 meters (4.5 feet) long. Glass viewing ports are built into one of the walls of the tunnel at 300-mm (one-foot) intervals. The tunnel operator watches the progress of the flame front along the specimen as it burns, recording intervals of time and distance. These distances are then plotted versus time on coordinate paper, and the area under the distance/time plot is calculated.

Before running specimens, the tunnel must be calibrated by exposing set-up specimens of red oak flooring and inorganic reinforced cement board. The area under the distance/time plot for the red oak is assigned a flame spread index of "100." The area under the distance/time plot for the cement board is assigned a flame spread index of zero. When an actual specimen is run, the area under its distance/time plot is compared to this 0-to-100 scale to assign a flame spread index to the specimen. The scale may also be extrapolated upward above 100 for specimens whose flame spread performance is worse than that of red oak.

The "magic number" for this test is 25, as most building codes that refer to this test method require the building products to have a flame spread index no higher than 25.

Other Large-Scale Tests

We merely wish to make the reader aware that there are other standard methods for conducting large-scale fire tests, all of which come under the jurisdiction of ASTM Committee E-5 on Fire Testing. If the reader wishes more information about them, they are contained in Vol. 04.07 of the *Annual Book of ASTM Standards*. The volume also contains a Standard Guide for Room Fire Experiments, E 603. Note that this is a guide only—there is not, as yet, any standard method for room fire experiments.

Full-Room Burn Experiment

Some years ago, the author witnessed a full-room burn experiment at what was then named the National Bureau of Standards (now named the National Institute for Standards Technology—NIST).

One entire wall of the room was a large glass window, through which we could clearly observe the entire room. The other walls were covered with wallpaper, and there was a simulated window with curtains. The room was furnished with a sofa, stuffed chairs, etc. to simulate a typical family room. There was a small pile of crisscrossed pieces of wood (called a "crib") at the base of the corner to serve as an ignition source.

For a few minutes after the crib was lighted, the resulting fire seemed rather innocuous; the small fire front traveled slowly up the corner walls. Eventually, it ignited the curtains, but even this was not frightening until "flashover" occurred. When it did, it was terrifying! In a matter of about 1 to 2 seconds, all of the covered walls, the ceiling, and the furniture were suddenly completely ablaze! It was an unforgettable experience!

Factory Mutual Corner Test

There is another large-scale test method that should be mentioned—the Factory Mutual Corner Test. In this method, very large specimens are mounted to comprise the interior walls of a corner assembly that is 16 feet high and several feet wide. A fuel crib is ignited at the base of the corner, as in the full room burn, and the ensuing flame spread is measured.

IGNITION TEST METHODS

This seems to be the item of least interest in the field of combustibility testing—a fact that has always puzzled the author. He knows of only two test methods that are described as measuring ignition properties of plastics.

The older one is ASTM D 1929, Test Method for Ignition Properties of Plastics. It is based on an apparatus called the Stechkin furnace. It is primarily intended to identify two end-points, as stated in Section 3 of the method:

A. *Flash-ignition temperature*—the lowest initial temperature, of air passing around the specimen, at which a sufficient amount of combustible gas is evolved to be ignited by a small external pilot flame.

B. *Self-ignition temperature*—the lowest initial temperature, of air passing around the specimen, at which (in the absence of an ignition source) the self-heating properties of the specimen lead to ignition, or ignition occurs of itself.

  - Very few people request D 1929 test data, and it is quite difficult to find someone who has the equipment to run the test.

The other known ignition method is ASTM D 3713, Test Method for Measuring Response of Solid Plastics to Ignition by a Small Flame. Specimens are the same size as for D 3801 (which is an older flame test method, described further on in this chapter)—13 by 127 mm (0.5 by 5.0 in.), with a maximum thickness of 12.7 mm (0.50 in.).

A set of specimens is subjected to a standard flame, applied in uniformly increasing 5-second increments to a maximum of 60 seconds. A new specimen is used at each increment. If an endpoint[3] occurs, the time of the flame application is decreased until ten specimens tested consec-

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[2] It is usually impractical, if not impossible, to fabricate and ship a single piece 25 ft long; specimens are usually shipped to the test site in three sections, whose ends are then abutted when placed in position atop the tunnel. The three pieces do not need to be the same length—in fact, it is best to make one of the pieces as long as possible in order for the first seam in the completed specimen to be as far away from the igniter flames as possible.

[3] An endpoint is defined as an occurrence of any of the following:

A. A specimen burns for more than 30 seconds after the burner is removed, or

B. The material drips, with or without ignition of cotton, either during the flame application or within 30 seconds after the burner is removed, or

C. The combustible material in the specimen is totally consumed within 30 seconds after the burner is removed.

utively, at the same duration of flame, pass the test. This duration, along with the specimen thickness and the letter(s) identifying the mode of response, is reported as the ignition response index (IRI).

ASTM D 2863, OXYGEN INDEX (A SMALL-SCALE TEST)

ASTM D 2863, Test Method for Measuring the Minimum Oxygen Concentration to Support Candle-Like Combustion of Plastics (Oxygen Index), is also referred to as the "oxygen candle" because the specimen is oriented like a burning candle.

This is probably the most widely used of the small-scale tests, primarily because it is just about the only combustibility test in existence that is capable of giving excellent repeatability. Coefficients of variation (see Chapter 2) in the 3 to 5% range are normal! The test method was originally developed within the General Electric Company and then offered to ASTM Committee D-20, which adopted it as a standard test method.

It is difficult to classify this test as to whether it is a flame-spread method or an ignition test. What is measured is the concentration of oxygen that will just support the continued burning of the specimen.

A. The test, and the test result, is often incorrectly called the "LOI," for "limiting oxygen index." (This was a pet peeve of the chairman of the task group that wrote the original ASTM method, published in 1970). George Carlin could have fun with this, as the term "oxygen index," in itself, is a limiting term in view of the procedure by which it is measured.

The specimen size for self-supporting plastics is 3 by 6.5 by 70 mm (0.12 by 0.25 by 2.8 in.). The specimen is held vertically on top of a stem-like holder, which is inside an open-top glass column of 75-mm (3-in.) minimum diameter and 450-mm (17.7-in.) minimum height.

Oxygen and nitrogen (or oxygen and air) are each piped through flow regulators and flow-measuring devices (do not use manometers—they are not sensitive enough). The flows are then combined at a "Y" joint in order to mix the gases. This common line then brings the gas mixture in at the bottom of the glass column, from where it rises through a bed of small glass beads to complete the mixing of the gases.

The flow rates of the two gases are set and measured at some arbitrary initial ratio, from which one may calculate the percentage of oxygen in the mixture. The top of the specimen is ignited by a small gas flame at the end of an igniter tube, which is introduced through the open top of the test column. As soon as the top of the specimen is burning merrily, the igniter is removed. The specimen is then merely observed to see whether it will continue to burn to the designated end point, as follows:

Specimen Type	Form of Material	Minimum Burning Criteria
A	Physically self-supporting	3 min or 75 mm
B	Alternate for self-supporting flexible plastics	3 min or 75 mm
C	Cellular plastics	3 min or 75 mm
D	Film or thin sheet	Past the 100-mm reference mark

If the specimen burns to the applicable end point, we reduce the oxygen concentration for the next specimen. If the specimen does not burn to the applicable end point, we increase the oxygen concentration for the next specimen. We continue to repeat the procedure with additional specimens until it is evident as to what oxygen concentration will barely allow specimens to reach the applicable burning criterion. That is the "oxygen index" value.

Calibration

In the older version of the method, Section 8.1 of the method required that the flow-measuring system be calibrated using a wet test meter in accordance with Method D 1071. However, D 1071 does not tell you how to use a wet test meter! In any case, the volume of the total gas flow may vary widely without affecting test results, so it is not important that the total gas flow be measured precisely.

The thing that must be known very precisely is the ratio of oxygen flow to nitrogen flow (or airflow) at any combination of flow readings. Therefore, the author recommends that the flow meter for the oxygen be calibrated directly against the flow meter for the nitrogen (or air) over as wide a range as will be used.

BURNER FLAMES

In regard to flame application for other small-scale combustibility tests, many of the test methods go into great detail in setting forth requirements for burner type and size, what gas should be used, and the size, shape, and temperature of the flame. The requirements vary widely among the various test methods. There is disagreement among testing people as to the importance of those aspects of the igniter flame. Some people can cite studies that show that none of those factors is very important. Others can cite studies that seem to prove that all of those factors may affect the test data.

Because of the above concerns, Committee D-20 has worked for several years to reach agreement on requirements for a "Standard Laboratory Burner and Flame Calibration Method." Such a burner is now described in ASTM D 5025, Specification for a Laboratory Burner Used for Small-Scale Burning Tests on Plastic Materials, and the calibration method is now described in ASTM D 5207, Practice for Calibration of 20- and 125-mm Test Flames for Small-Scale Burning Tests on Plastic Materials.

In any case, however, do whatever the method that you're using says you should do!

ASTM D 3801 AND UL94 (A SMALL-SCALE TEST)

ASTM D 3801, Test Method for Measuring the Comparative Extinguishing Characteristics of Solid Plastics in a Vertical

Position, was proposed by IBM Corporation and adopted by ASTM Committee D-20 in 1980.

Specimens are 13 by 127 mm (0.5 by 5.0 in.). Maximum thickness is 12.7 mm (0.50 in.). The specimen is held vertically at its top. A burner flame is applied to its bottom for 10 seconds, then withdrawn; as soon as flaming of the specimen ceases, the burner flame is reapplied for another 10 seconds. The data to be reported are:

A. Duration of flaming time after first flame impingement.
B. Duration of flaming time after second flame impingement.
C. Duration of flaming plus growing times after second flame impingement.

For UL94, the material is rated as V0, V1 , or V2, depending on how long the specimen burned and whether flaming drops ignited the cotton placed below the specimen, using the same criteria as for ASTM D 3801.

METHODS THAT SPECIFICALLY MEASURE FLAME SPREAD

This type of testing was the primary type of combustibility testing in which most laboratories were interested until about 1970. Of course, it continues to be very important, along with testing for smoke evolution, heat release, and toxicity, which will be described farther along in this chapter.

Medium-Scale Tests

ASTM E 162 Radiant Panel Test

We are mentioning ASTM E 162, Test Method for Surface Flammability of Materials Using a Radiant Heat Energy Source, only to make you aware that most people who have tried to use the method have found that the repeatability and the reproducibility are very bad.

On paper, it seems to have the makings of an excellent method. A 150 by 460-mm (6 by 18-in.) specimen is ignited at the top while exposed, at an angle, to radiant heat from a gas-fired panel. A flame spread index is calculated, but it is not comparable to data from the 25-ft tunnel.

ASTM D 3675 Radiant Panel Test

ASTM D 3675, Test Method for Surface Flammability of Flexible Cellular Materials Using a Radiant Heat Energy Source, is essentially the same test method as the above E 162 radiant panel method.

Small-Scale Tests

ASTM D 635 (and UL94)

Until a few years ago, there was a very small difference in procedure between ASTM D 635, Test Method for Rate of Burning and/or Extent and Time of Burning of Self-Supporting Plastics in a Horizontal Position, and Underwriters' Laboratories' UL94 test method with the specimen positioned in the horizontal position. (UL94 also includes a vertical orientation.) The difference was just enough so that test results from the two methods were not quite comparable. Happily, the two methods have been comparable for the past several years.

Both methods use a specimen 125 mm (5 in.) by 12.5 mm (0.5 in.) by material thickness. Two lines are scribed across the specimen at 25 mm (0.98 in.) and 100 mm (3.94 in.) from one end of the specimen. The specimen is held by a laboratory clamp at the end nearest the 100-mm line, with its longitudinal axis horizontal and its transverse axis inclined at 45°.

Flame from an ASTM D 5025 burner is applied to the other end for 30 seconds. If the specimen warps, melts, or shrinks away from the flame, the flame must be moved to keep it in contact with the specimen. The burner is removed after 30 seconds or when the flame front reaches the 25-mm line, whichever comes first.

This is a very simple test, except for figuring out how to report the test results. In regard to this, the reader should study D 635 very carefully, including notes and annexes or appendices.

As stated in the "safety" section early in this chapter, the author strongly recommends the use of a specimen-shielding cabinet, leaving the exhaust hood turned on all the time while running this test and the test methods named below.

ADDITIONAL SMALL-SCALE FLAME-SPREAD TEST METHODS

The following test methods use various specimen sizes and orientations, as well as different ways of mounting specimens. However, in general, they all use variations of the techniques already described in regard to D 1692 and D 635:

> ASTM D 3014, Standard Test Method for Flame Height, Time of Burning, and Loss of Mass of Rigid Thermoset Cellular Plastics in a Vertical Position. The method is commonly called the "Butler Chimney" test.
>
> California Bulletins: The state of California has issued several combustibility test methods, named as "Bulletins." California Bulletin 117, for instance, describes a test method for flexible cellular plastics intended for use in upholstered furniture.
>
> DOT Motor Vehicle Safety Standard No. 302 (MVSS 302). This is a horizontal test for flexible cellular plastics. It was dreamed up by the U.S. Department of Transportation. It has a great similarity to D 1692 (now D 4986), but D 4986 is much easier to understand. The very brief instructions in MVSS 302 leave a lot to the imagination.

RECOMMENDATIONS

In view of the lack of precision and the lack of correlations among most of the combustibility methods, how should one proceed to evaluate the combustibility of a new formulation? The author strongly discourages taking any action based on data from only one combustibility method. The author recommends choosing at least two small-scale methods to start with (three or four is even better) and taking the results of all of them into account on a subjective basis before investing money in a larger-scale test. It is recommended that Oxygen Index be one of the small-scale tests. Many people also have found the data from D 3801 to be quite useful.

SMOKE GENERATION

In the early 1970s, the concern about flame spread testing became extended to an equal concern about the amount of smoke produced by burning materials. At first, the emphasis was on measuring the amount of smoke particulates. There was a gravimetric method, ASTM D 4100, Method for Gravimetric Determination of Smoke Particulates from Combustion of Plastic Materials. However, gravimetric methods never became widely used. Accordingly, Committee D-20 has discontinued D 4100.

Early on, it was generally recognized that the greatest danger from smoke in a real fire situation is that the smoke is likely to obscure exit signs and cause psychological responses such as disorientation and panic, thus preventing people from escaping. It also was recognized that the obscuration characteristics of the smoke depended on several factors other than the weight of the particulates. Therefore, the accepted way to measure smoke evolution of a material is by measuring its visual obscuration effect. The two most widely used methods for measuring smoke generation are:

A. ASTM D 2843, Test Method for Density of Smoke from the Burning or Decomposition of Plastics. The method was originated by Rohm and Haas Company and was approved by ASTM Committee D-20 as a standard test method in 1970. It is commonly called "the XP-2 chamber."

B. ASTM E 662, Test Method for Specific Optical Density of Smoke Generated by Solid Materials. The method originated at the National Bureau of Standards (now NIST) and was offered to ASTM Committee E-5 around 1971. Its approval was delayed for several years by arguments, round robins, and stonewalling. It was finally approved as a standard test method in 1979. The National Fire Protection Association had already approved it around 1975. It is commonly called "the NBS chamber."

D 2843 is limited to plastics. E 662 is a general method, and it is used to test plastics, as well as many other materials. In both methods, a specimen is placed inside a metal chamber. The chamber is closed, and the specimen is exposed to heat. A beam of light is directed across the chamber; the amount of light that is able to pass through the smoke is measured by a photoelectric cell.

Data from one method are not comparable with data from the other. There are important differences between the two methods:

A. Specimen size:
   D 2843: 25.4 by 25.4 mm (1 by 1 in.).
   E 662: 76.2 by 76.2 mm (3 by 3 in.).

B. Heat exposure:
   1. E 662: The specimen is supported vertically in a holder with a cement board backing. It is subjected to a radiant heat flux of 2.5 watts per square centimeter (2.2 Btu per square foot), provided by an electric furnace. If desired, flame-induced ignition may be provided by six small gas flamelets at the base of the specimen. Flaming exposure always is accompanied by the above radiant heat exposure—never as an alternative to it.
      a. The size of the flamelets is controlled very precisely by adjusting the flow rate of the propane gas. The gas passes through a flowmeter in order to monitor its rate of flow. It is essential that the propane flowmeter be carefully calibrated from time to time. The author experienced a case where a government facility was consistently measuring D_m values (see Subparagraphs D.2.b. below) of about 100 for polyether sulfone, but the author's laboratory was consistently obtaining values of about 25.

         Initially, despite a great deal of cooperative investigation, neither party could discern any reason for the difference. Suddenly, we both realized the likely solution—the government facility's propane flowmeter had not been recalibrated for several years, whereas the author's equipment was quite new. After recalibration of the government facility's flowmeter, the two laboratories got excellent agreement.
      b. When the igniting flamelets are not used, some specimens flame spontaneously, while others only degrade and char.
      c. With some formulations, flaming exposure produces more smoke; with other formulations, non-flaming exposure produces more smoke. There is no way to predict which way it will go. When testing new formulations, the author strongly recommends using both flaming and non-flaming modes of exposure.
      d. There is a narrow trough at the bottom of the specimen holder to collect any thermoplastic material that may flow downward during heat exposure. In the flaming mode, two of the flamelets are directed downward into the trough.
   2. D 2843: There is no radiant heat exposure. The specimen rests horizontally on a stainless steel screen and is ignited by a small burner flame.
      a. Until some time after 1987, D 2843 did not address the problem of thermoplastic materials, which melt and drip away from the igniter flame. The current revision provides for such material to be collected below the specimen holder and for a second igniter flame to impinge on it.

C. Orientation of light path:
   1. D 2843: The light path is horizontal. Because of this, any stratification of the smoke inside the chamber will cause an erroneous degree of light absorption.
   2. E 662: The light path vertical.

D. Data:
   1. D 2843: The instrumentation of the photoelectric cell is such that the output is in terms of light absorbed, rather than light transmitted. The per cent light absorbed is recorded at 15-second intervals for 4 minutes. It is then plotted on linear coordinates.
      a. The highest point on the curve is reported as maximum smoke density in terms of per cent light absorbed.
      b. The total smoke produced during the 4 minutes is measured as the total area under the curve. This is divided by the total area of the chart (within the chart limits of 100% and 4 minutes) and multiplied by 100 to give a value called the smoke density rating in per cent.
   2. E 662: The per cent light transmittance is automatically plotted on a chart recorder. The test is terminated 3

minutes after the minimum light transmittance value is reached, or after 20 minutes, whichever occurs first.

a. The data on per cent light transmittance are converted to specific optical density, D_s, which is inversely proportional to the amount of light transmitted.
    (1) The calculation of D_s also takes into account the volume of the chamber, the exposed area of the specimen, and the length of the light path. For this reason, it was originally thought that D_s values from the test could be used to predict smoke levels in theoretical fire situations in rooms of known size. However, as with so many other aspects of the physical testing field, the logical theory broke down in the face of actual practice.

b. The D_s value for the minimum light transmittance is reported as D_m.
    (1) Until 1983, the method required that, after all of the smoke had been evacuated from the chamber at the end of the test, the light transmittance be measured and D_s be calculated. This D_s was called "D_c", a correction for the smoke that had accumulated on the windows of the light path system. It was subtracted from D_m to give D_m(corr), which was reported instead of D_m. However, analysis of round robin data showed that the D_m values were more uniform than the D_m(corr) values; it was then realized that the argument for using D_m(corr) was flawed. D_m(corr) is no longer used.

c. The time, in minutes, for the smoke to accumulate to the minimum light transmittance point is reported as t_{D_m}.
    (1) This is reported because, for some materials, the rate of smoke generation is very low for several minutes, and then it increases dramatically. Others start generating large amounts of smoke almost immediately. Often, the D_m values of the two materials will be about the same. Obviously, though, if we were in a burning room, we would hope that the first material had been used, rather than the second, to give us more time before the EXIT signs become obscured.

d. It would be desirable to have a single term that would roughly evaluate the size and shape of the total smoke/time curve. There have been several proposals for accomplishing this, but none have been written into E 662. The author developed such a term and used it within his laboratory for several years. He called it smoke generation rate (SGR). It has the advantage of being much simpler to calculate than most of the other proposals:

$$\text{SGR} = D_{s1.5} + D_{s4} + (D_m/t_{D_m})$$

where: $D_{s1.5}$ is D_s at 1.5 minutes after starting the test,
D_{s4} is D_s at 4 minutes after starting the test, and
D_m and t_{D_m} are described above.

The 25-foot tunnel and the radiant panel test equipment include photoelectric cells for the purpose of measuring the smoke evolution of the burning material. However, the values for Smoke Index from these two test methods are generally regarded as being so imprecise that very few people pay any attention to them.

AMOUNT OF HEAT GENERATED BY BURNING MATERIALS

The 25-foot tunnel and the radiant panel test equipment also include thermocouples for the purpose of measuring the fuel contribution of the burning material. However, the values for the Fuel Contribution Index from these two test methods are also generally regarded as being so imprecise that very few people pay any attention to them.

Rate of Heat Release

What has gained far more acceptance in regard to fuel contribution is ASTM E 906, Test Method for Heat and Visible Smoke Release Rates for Materials and Products. It is based on an apparatus developed at Ohio State University by Dr. Edwin Smith, who offered it to ASTM Committee E-5 on Fire Testing for consideration. It was adopted as a standard method by E-5 in 1983. It is still often referred to as "the Smith apparatus."

The apparatus is very expensive (something over $50,000 in 1992), and relatively few laboratories have it. In the past, the reproducibility between laboratories has not been good. However, recent improvements in some aspects of the equipment have improved the reproducibility.

Meanwhile, another method for measuring rate of heat release was originated by Dr. Vytenis Babrauskas at NIST. It is commonly referred to as "the cone calorimeter," although this is a partial misnomer. Actually, the cone calorimeter is the radiant heat device that is used to heat the specimen. The actual measurement of heat release is done indirectly, by monitoring the rate of oxygen consumption in the exposure chamber during the thermal breakdown of the specimen. The equipment, also, is very expensive. The method has been adopted by Committee E-5 as ASTM E 1354.

TESTING FOR COMBUSTION TOXICITY

This is proving to be an especially controversial field of testing. There are two general approaches to it:

A. *The Chemical Analysis Approach*—The earliest approach was to expose a specimen of material to an ignition source and then identify and measure the quantity of the gases that were generated, by passing them through various analytical instruments.
    1. A lot of data were collected for awhile, but this approach seems to have pretty much stalled with the realization that these analytical data are useless unless we known how the gases will affect humans. Therefore it has largely been supplanted by the second approach, which is to use bio-assay techniques.

B. *The Bio-Assay Approach*—There are two basic ideas connected with the bio-assay approach:

1. The only practical way to find out what effect evolved gases will have on humans is to study their effect on laboratory animals.
2. Burning materials do not give off single gases—they evolve complex mixtures of gases, and various combinations of gases may produce synergistic effects on animals. Therefore, the bio-assay experiments do not use pure gases. Instead, laboratory animals are exposed to whatever mixture of gases may evolve from a burning specimen.

The specimen is exposed to a heat source or an ignition source, or both. The evolved gases are passed into a chamber containing test animals. In the United Kingdom, monkeys are widely used, but mice or rats are the usual choice in the United States. Any animals that survive the immediate test exposure are observed for a standard period (such as two weeks) after the test. The specimen exposure is varied from one test method to another.

The procedure follows the LD_{50} ("LD" is short for "lethal dose") approach—a test result consists of determining the specimen exposure level that will cause the death of 50% of the animals within the standard time period.

A. Many people believe that a more useful end point for LD_{50} would be incapacitation rather than death. The basis for this idea is that the toxic gases from a fire do not need to kill a person outright—if the person is incapacitated, he will probably die from further gas exposure or from the heat. Or, he may escape from the fire scene and then have delayed toxic reactions.

During the early 1980s, five different proposed toxicity test methods were introduced by various people for consideration by the Toxicity Section of ASTM Committee E-5. All of them were variations of the general procedure described above. In 1996, ASTM E 1678, Test Method for Measuring Smoke Toxicity for Use in Fire Hazard Analysis, was approved. This standard is not based on the method adopted by the state of New York, which is described below.

State of New York Toxicity Test Requirement

Meanwhile, in 1986, the State of New York passed a law that requires that certain building products be subjected to a toxicity test that was developed at the University of Pittsburgh. But the law does not include any performance requirements! The data are merely filed, and the data are available to the public. The test may be conducted only by a laboratory acceptable to the New York Secretary of State.

The fact that the University of Pittsburgh test was adopted by New York does not mean that it is a better method than any of the five that were already under consideration in Committee E-5. It was submitted to Committee E-5 as a sixth contender after it was adopted by New York. Under this scrutiny, several aspects of the test procedure and equipment were challenged by people who are very knowledgeable in the field. As far as the author knows, those suggested changes that were most important were never made.

Carbon Monoxide Poisoning

You are probably aware that the burning of some plastics causes the generation of hydrogen cyanide gas, and you were probably shocked and scared when you learned it. But did anyone ever tell you that other materials (wool, for instance) also evolve hydrogen cyanide in large quantities? They do, indeed!

As stated at the beginning of this section, a lot of work has been done in quantifying the amounts of the various gases that are given off by the burning of a wide range of materials (including mashed potatoes, yet!). In reviewing lists based on these analyses, the author was struck by one common result—in every case where carbon-based materials were burned (wood, plastic, paper, textiles, etc), carbon monoxide was evolved in far greater quantities than any other gas.

People seem to lose sight of the fact that carbon monoxide is a deadly poison, and it is hard for them to believe that it is odorless and, thus, extremely dangerous. Most people think that they will be able to smell it if they find themselves in a fire situation, because they know that it is present in automobile exhaust fumes and they have all smelled the exhaust fumes. They do not realize that they are smelling other combustion products—not the carbon monoxide.

Quite a number of years ago, the author was told that Johns Hopkins Hospital had undertaken a project to perform autopies on people who had died in fires. The autopsies were carried out on approximately 100 victims, which is a large enough sampling for the study to be statistically valid. It was found that, in the vast majority of cases, the cause of death was carbon monoxide inhalation rather than exposure to flames.

In view of the above, the author wonders whether further combustion toxicity testing is really worthwhile when we already know that carbon monoxide presents the greatest danger in any fire scenario, and that its presence is unavoidable.

Corrosivity

The above chemical analysis projects showed that some materials evolve gases that are highly corrosive, such as hydrogen chloride (HCl). There has been at least one instance of a fire in which there were no deaths or injuries, but corrosion damage to equipment was monumental.

Since 1990, ASTM Committee D-9 has been evaluating the combustion corrosivity of cable insulation materials, and a test method has been published—ASTM D 5485.

Since 1992, ASTM Committee E-5 has been considering the problem of testing for combustion corrosivity. A proposed standard was under ballot as of late 1996.

Subject Index

ABOUT THE EDITOR

STEPHEN BURKE DRISCOLL

Since 1968 Stephen Burke Driscoll has been a Professor in The Department of Plastics Engineering at The University of Massachusetts Lowell (formerly The Lowell Technological Institute and the University of Lowell). He teaches undergraduate and graduate students polymeric materials engineering, additives for plastics, physical properties and polymer characterization, including dynamic mechanical rheological testing, and commercial development of polymers.

He joined ASTM in 1978 and is a founding member and Chair of the D20.10.15 Section on Dynamic Mechanical Properties of Plastics. He has also chaired D20.10.14 on Long Term Properties of Plastics. Within the D20 Committee on Plastics, he has completed three two-year terms as elected Vice-Chair of Test Methods and has recently been elected Vice-Chair of Materials. He has served ASTM as an appointed three-year member of The Committee on Technical Committee Operations (COTCO) and in 1998 will complete his three-year assignment as Chair of COTCO.

In addition to authoring/presenting more than 100 papers at various technical society meetings, he has authored several standard test methods (STMs) on dynamic mechanical properties and has contributed chapters to four ASTM STPs on RIM, polymeric materials and products, high modulus fiber composites, and instrumented impact testing. He has twice served the United Nations Industrial Development Organization (UNIDO) as a Consulting Fellow on Rheology in India.

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