Designation: D7690 − 11 (Reapproved 2017)

Standard Practice for Microscopic Characterization of Particles from In-Service Lubricants by Analytical Ferrography[1]

This standard is issued under the fixed designation D7690; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the identification by optical microscopy of wear and contaminant particles commonly found in used lubricant and hydraulic oil samples that have been deposited on ferrograms. This practice relates to the identification of particles, but not to methods of determining particle concentration.

1.2 This practice interfaces with but generally excludes particles generated in the absence of lubrication, such as may be generated by erosion, impaction, gouging, or polishing.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D4057 Practice for Manual Sampling of Petroleum and Petroleum Products

D4175 Terminology Relating to Petroleum Products, Liquid Fuels, and Lubricants

D7684 Guide for Microscopic Characterization of Particles from In-Service Lubricants

G40 Terminology Relating to Wear and Erosion

3. Terminology

3.1 *Definitions:*

3.1.1 *abrasion, n*—wear by displacement of material caused by hard particles or hard protuberances. **D4175**

3.1.2 *abrasive wear, n*—wear due to hard particles or hard protuberances forced against and moving along a solid surface. **G40**

3.1.3 *adhesive wear, n*—wear due to localized bonding between contacting solid surfaces leading to material transfer between the two surfaces or loss from either surface. **G40**

3.1.4 *break-in, n*—See *run-in*. **D4175, G40**

3.1.5 *break in, v*—See *run in*. **G40**

3.1.6 *catastrophic wear, n*—rapidly occurring or accelerating surface damage, deterioration, or change of shape caused by wear to such a degree that the service life of a part is appreciably shortened or its function is destroyed. **G40**

3.1.7 *corrosion, n*—chemical or electrochemical reaction between a material, usually a metal surface, and its environment that can produce a deterioration of the material and its properties. **D4175**

3.1.8 *corrosive wear, n*—wear in which chemical or electrochemical reaction with the environment is significant. **G40**

3.1.9 *debris, n—in tribology*, particles that have become detached in a wear or erosion process. **G40**

3.1.10 *debris, n—in internal combustion engines,*solid contaminant materials unintentionally introduced in to the engine or resulting from wear. **D4175**

3.1.11 *fatigue wear, n*—wear of a solid surface caused by fracture arising from material fatigue. **G40**

3.1.12 *fretting, n—in tribology*, small amplitude oscillatory motion, usually tangential, between two solid surfaces in contact.

3.1.12.1 *Discussion*—Here the term *fretting* refers only to the nature of the motion without reference to the wear, corrosion, or other damage that may ensue. The term *fretting* is often used to denote fretting corrosion and other forms of

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[1] This practice is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.96.06 on Practices and Techniques for Prediction and Determination of Microscopic Wear and Wear-related Properties.

Current edition approved May 1, 2017. Published July 2017. Originally approved in 2011. Last previous edition approved in 2011 as D7690 – 11. DOI: 10.1520/D7690-11R17.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

fretting wear. Usage in this sense is discouraged due to the ambiguity that may arise. **G40**

3.1.13 *fretting wear, n*—wear arising as a result of fretting. (See *fretting*.) **G40**

3.1.14 *friction, n*—resistance to sliding exhibited by two surfaces in contact with each other. Basically there are two frictional properties exhibited by any surface; static friction and kinetic friction. **D4175**

3.1.15 *impact wear, n*—wear due to collisions between two solid bodies where some component of the motion is perpendicular to the tangential plane of contact. **G40**

3.1.16 *lubricant, n*—any material interposed between two surfaces that reduces the friction or wear between them. **D4175**

3.1.17 *lubricating oil, n*—liquid lubricant, usually comprising several ingredients, including a major portion of base oil and minor portions of various additives. **D4175**

3.1.18 *pitting, n—in tribology*, form of wear characterized by the presence of surface cavities the formation of which is attributed to processes such as fatigue, local adhesion, or cavitation. **G40**

3.1.19 *rolling, v—in tribology*, motion in a direction parallel to the plane of a revolute body (ball, cylinder, wheel, and so forth) on a surface without relative slip between the surfaces in all or part of the contact area. **G40**

3.1.20 *rolling contact fatigue, n*—damage process in a triboelement subjected to repeated rolling contact loads, involving the initiation and propagation of fatigue cracks in or under the contact surface, eventually culminating in surface pits or spalls. **G40**

3.1.21 *run-in, n—in tribology*, initial transition process occurring in newly established wearing contacts, often accompanied by transients in coefficient of friction, or wear rate, or both, which are uncharacteristic of the given tribological system's long term behavior. (Synonym: *break-in*, *wear-in*.) **D4175**, **G40**

3.1.22 *run in, v—in tribology*, to apply a specified set of initial operating conditions to a tribological system to improve its long term frictional or wear behavior, or both. (Synonym: *break in*, v, and *wear in*, v.) See also *run-in*, n) **G40**

3.1.23 *rust, n—of ferrous alloys*, a corrosion product consisting primarily of hydrated iron oxides. **D4175**

3.1.24 *scoring, n—in tribology*, severe form of wear characterized by the formation of extensive grooves and scratches in the direction of sliding. **D4175**, **G40**

3.1.25 *sliding wear, n*—wear due to the relative motion in the tangential plane of contact between two solid bodies. **G40**

3.1.26 *soot, n—in internal combustion*, engines, sub-micron size particles, primarily carbon, created in the combustion chamber as products of incomplete combustion. **D4175**

3.1.27 *spalling, n—in tribology*, the separation of macroscopic particles from a surface in the form of flakes or chips, usually associated with rolling element bearings and gear teeth, but also resulting from impact events. **G40**

3.1.28 *three-body abrasive wear, n*—form of abrasive wear in which wear is produced by loose particles introduced or generated between the contacting surfaces.

3.1.28.1 *Discussion*—In tribology, loose particles are considered to be a "third body." **G40**

3.1.29 *triboelement, n*—one of two or more solid bodies that comprise a sliding, rolling, or abrasive contact, or a body subjected to impingement or cavitation. (Each triboelement contains one or more tribosurfaces.)

3.1.29.1 *Discussion*—Contacting triboelements may be in direct contact or may be separated by an intervening lubricant, oxide, or other film that affects tribological interactions between them. **G40**

3.1.30 *two-body abrasive wear, n*—form of abrasive wear in which the hard particles or protuberances which produce the wear of one body are fixed on the surface of the opposing body. **G40**

3.1.31 *viscosity, n*—ratio between the applied shear stress and rate of shear. It is sometimes called the coefficient of dynamic viscosity. This value is thus a measure of the resistance to flow of the liquid. The SI unit of viscosity is the pascal second (Pa.s). The centipoise (cP) is one millipascal second (mPa.s) and is often used. **D4175**

3.1.32 *wear, n*—damage to a solid surface, usually involving progressive loss or displacement of material, due to relative motion between that surface and a contacting substance or substances. **G40**, **D4175**

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *abrasive wear particles, n*—long wire-like particles in the form of loops or spirals generated due to hard, abrasive particles present between wearing surfaces of unequal hardness.

3.2.1.1 *Discussion*—Sometimes called cutting wear particles.

3.2.2 *analytical ferrography, n*—technique whereby particles from an oil sample deposited by a ferrograph are identified to aid in establishing wear mode inside an oil-wetted path of a machine.

3.2.3 *bichromatic microscope, n*—optical microscope equipped with illumination sources both above and below the microscope stage such that objects may be viewed either with reflected light, or with transmitted light, or with both simultaneously.

3.2.4 *black oxides of iron, n*—generally small, black clusters with pebbled surfaces showing small dots of blue and orange color. These are nonstoichiometric compounds containing a mixture of Fe_3O_4, Fe_2O_3 and FeO.

3.2.5 *contaminant particles, n*—particles introduced from an extraneous source into the lubricant of a machine or engine.

3.2.6 *chunks, n*—free metal particles >5 µm with a shape factor (major dimension to thickness ratio) of <5:1.

3.2.7 *corrosive wear debris, n*—extremely fine partially oxidized particles caused by corrosive attack.

3.2.8 *dark metallo-oxide particles, n*—partially oxidized ferrous wear particles indicating high heat during generation most likely due to lubricant starvation.

3.2.9 *entry, n*—entry area of the ferrogram, region where the sample first touches down onto the glass surface of the ferrogram and where the largest ferrous particles are deposited.

3.2.10 *ferrograph, n*—apparatus to magnetically separate and deposit wear and contaminant particles onto a specially prepared glass microscope slide.

3.2.11 *ferrogram, n*—specially prepared glass microscope slide that has ferrographically deposited particles on its surface.

3.2.12 *fibers, n*—long, thin, nonmetallic particles.

3.2.13 *friction polymers, n*—these are characterized by small metal particles embedded in an amorphous matrix.

3.2.14 *nonferrous metal particles, n*—free metal particles composed of any metal except iron. All common nonferrous metals behave nonmagnetically except nickel.

3.2.15 *nonmetallic particles, n*—particles comprised of compounds, organic material, glasses, etc., that have bound electrons in their atomic structure.

3.2.16 *nonmetallic amorphous particles, n*—particles without long range atomic order that are transparent and that do not appear bright in polarized light.

3.2.17 *nonmetallic crystalline particles, n*—particles with long range atomic structure that appear bright in polarized light. These may be single crystals but are most likely polycrystalline or polycrystalline agglomerates.

3.2.18 *platelets, n*—flat, free metal wear particles that are longer and wider than they are thick. They have a major dimension-to-thickness ratio in the range of approximately 5:1 to 10:1 or more.

3.2.19 *red oxide particles, n*—rust particles present as polycrystalline agglomerates of Fe_2O_3 appearing orange in reflected white light. These are usually due to water in the lubricating system.

3.2.20 *red oxide sliding particles, n*—sliding wear particles that appear gray in reflected white light, but are dull reddish-brown in white transmitted light.

3.2.21 *reworked particles, n*—large, very thin, free metal particles often in the range of 20 µm to 50 µm in major dimension with the frequent occurrence of holes consistent with the explanation these are formed by the passage of a wear particle through a rolling contact.

3.2.22 *rolling contact fatigue particles, n*—flat platelets, with their length more or less equal to their width, with smooth surfaces, random, jagged and irregularly shaped circumferences and a major dimension-to-thickness ratio in the range of approximately 5:1 to 10:1 or more.

3.2.23 *rubbing wear particles, n*—particles generated as a result of sliding wear in a machine, sometimes called mild adhesive wear. Rubbing wear particles are free metal platelets with smooth surfaces, from approximately 0.5 µm to 15 µm in major dimension and with major dimension-to-thickness ratios from about 10:1 for larger particles and to about 3:1 for smaller particles. Any free metal particle <5 µm is classified as a rubbing wear particle regardless of shape factor unless it is a sphere.

3.2.24 *severe sliding wear particles, n*—severe wear particles displaying surface striations and straight edges.

3.2.25 *severe wear particles, n*—free metal particles >15 µm, and with major dimension-to-thickness ratios between 5:1 and 30:1.

3.2.26 *spheres, n*—metal spheres may be the result of incipient rolling contact fatigue or they may be contaminant particles from welding, grinding, coal burning and steel manufacturing. Spheres may also be caused by electro-pitting.

3.2.27 *wear particles, n*—particles generated from a wearing surface of a machine.

4. Summary of Practice

4.1 Periodic in-service lubricant samples are collected from a machine or engine as part of a routine condition monitoring program. A ferrogram is prepared from the sample to separate particles from sample fluid. The ferrogram is subsequently examined using an optical microscope to identify the types of particles present to aid in identifying the wear mode occurring in the oil-wetted path of the machine.

4.2 In usual practice of a routine condition monitoring program, a ferrogram is not prepared for every sample taken, but may be prepared when routine tests such as spectrochemical analysis, particle counting or ferrous debris monitoring indicate abnormal results.

4.3 The user of this practice employs consistent terminology to achieve accepted and understandable interpretations when communicating instructions and findings based on ferrographic analysis.

5. Significance and Use

5.1 The objective of ferrography is to diagnose the operational condition of the machine sampled based on the quantity and type of particles observed in the oil. After break-in, normally running machines exhibit consistent particle concentration and particle types from sample to sample. An increase in particle concentration, accompanied by an increase in size and severity of particle types is indicative of initiation of a fault. This practice describes commonly found particles in in-service lubricants, but does not address methodology for quantification of particle concentration.

5.2 This practice is provided to promote improved and expanded use of ferrographic analysis with in-service lubricant analysis. It helps overcome some perceived complexity and resulting intimidation that effectively limits ferrographic analysis to the hands of a specialized and very limited number of practitioners. Standardized terminology and common reporting formats provide consistent interpretation and general understanding.

5.3 Without particulate debris analysis, in-service lubricant analysis results often fall short of concluding likely root cause or potential severity from analytical results because of missing information about the possible identification or extent of damaging mechanisms.

5.4 Ferrographic analysis, as described in this practice, provides additional particle identification capabilities beyond methods described in Guide D7684 for the following reasons:

(1) The ferrographic particle separation method is magnetic thus making it possible to readily distinguish between ferrous and nonferrous wear particles.

(2) Ferrography separates ferrous (magnetic) particles by size.

(3) Deposition is on a glass substrate so that particles may be examined using transmitted light as well as reflected light allowing particle types to be identified that cannot be identified when examination is done using only reflected light.

(4) Ferrograms may be heat treated providing important distinctions between ferrous alloy types (steel versus cast iron), further distinctions among various nonferrous alloys and distinctions between inorganic and organic particles.

5.5 Caution must be exercised when drawing conclusions from the particles found in a particular sample, especially if the sample being examined is the first from that type of machine. Some machines, during normal operation, generate wear particles that would be considered highly abnormal in other machines. For example, many gear boxes generate severe wear particles throughout their expected service life, whereas just a few severe wear particles from an aircraft gas turbine oil sample may be highly abnormal. Sound diagnostics require that a baseline, or typical wear particle signature, be established for each machine type under surveillance.

6. Apparatus

6.1 *Required Components:*

6.1.1 *Ferrograph or Ferrogram Maker*—Apparatus for magnetically separating particles from fluids.

6.1.2 *Bichromatic Microscope*—An optical microscope is required with dry metallurgical objective lenses and equipped with a reflected light source and a transmitted light source so that objects may be viewed from both above and below the microscope stage. This permits objects to be viewed either with reflected light, or with transmitted light, or with both simultaneously. Bichromatic microscopes for ferrogram examination are required to be equipped with three objective lenses to give varying degrees of magnification. The low magnification objective lens is typically 10×, the medium magnification objective lens may be 40× or 50× and the high magnification objective lens may be 80× or 100×. Ten power (10×) ocular (eyepiece) lenses are used such that total magnification achieved is 100× at low magnification, 400× or 500× at medium magnification and 800× or 1000× at high magnification. The numerical apertures of the objective lenses need to be high to maximize illumination of particle surfaces when viewed in reflected light. It is required to be able to polarize either light path to facilitate particle identification. Polarized light aids in the identification of nonmetallic particles. A red filter is required to be optionally placed in the reflected light path and a green filter is required to be optionally placed in the transmitted light path. The simultaneous use of red reflected and green transmitted light aids in the distinction between metallic and nonmetallic particles. One of the ocular lenses should be fitted with a calibrated scale so that length of objects may be measured. The stage drive of the microscope should be fitted with calibrated divisions so that thickness of objects may be measured as the stage is raised or lowered.

6.1.3 *Blank Ferrogram Glass Substrates*—A supply of specially prepared microscope slides with nonwetting barriers to contain sample flow in the central portion of the substrate are required.

6.1.4 *Precision Pipettor*—A pipettor capable of delivering a precise volume of 1 mL of viscous fluid is required.

6.1.5 *Mixing Vials*—Clean vials, usually 12 mL capacity, are needed to mix sample with solvent prior to processing by the ferrograph.

6.2 *Optional Components:*

6.2.1 *Hot Plate*—A hot plate capable of achieving surface temperatures of 540 °C is required if it is desired to heat treat ferrograms to further identify the metallurgy of metal particles.

6.2.2 *Surface Thermometer*—A surface thermometer capable of measuring to 540 °C is needed for heat treating ferrograms.

6.2.3 *Tongs*—Tongs are needed to remove the heated ferrogram from the hot plate.

6.2.4 *Camera*—The microscope may be equipped with a suitable camera for taking photomicrographs for reporting and documenting purposes.

7. Reagents

7.1 *Heptane*—The recommended solvent is heptane, but other solvents may be used if they meet the following criteria:

7.1.1 The solvent must be a good oil solvent.

7.1.2 The solvent cannot be too volatile. If the solvent evaporates too quickly the strings of particles on the ferrogram surface will be pulled out of place by the movement of the quickly drying solvent.

7.1.3 If the solvent evaporates too slowly, excessive time will be spent waiting for the ferrogram to dry.

7.1.4 The solvent needs to be residue and particle-free. Prepare a ferrogram using only solvent and examine it under the microscope to make sure the ferrogram surface is clean. From a practical viewpoint, it will be almost impossible to prepare a blank ferrogram, that is, one that is totally free of particles. Therefore, some judgment should be exercised regarding an acceptable cleanliness level. A few small nonmetallic particles are tolerable in that they would not interfere with evaluation of machine condition. On the other hand, if the blank ferrogram has metallic particles deposited on it, then steps need to be taken to eliminate the source of contamination. It may be necessary to filter the solvent through a submicron membrane filter to remove particulate contaminants or to let the solvent remain undisturbed for overnight or longer so that particles settle to the bottom of the bottle or container. Withdrawing solvent from near the top of the undisturbed container will likely yield particle free solvent.

8. Sampling and Sample Handling

8.1 *Sample Acquisition*—The objective of sampling is to obtain a test specimen that is representative of the entire quantity. Thus, laboratory samples should be taken in accordance with instructions in Practice D4057.

9. Procedure

9.1 *Ferrogram Preparation:*

9.1.1 *Sample Preparation*—Laboratory samples should be shaken or agitated to ensure a representative sample is taken from the sample bottle.

9.1.2 In-service lubricating oil samples must be diluted with solvent to lower their viscosity before the sample is allowed to flow onto the substrate. If the solvent is particle free, it does not matter how much solvent is used to dilute the oil sample. The purpose of the dilution is to make the solvent/sample mixture have a viscosity such that it flows onto the ferrogram at an approximate rate of 0.4 mL⁄min. Experience indicates that an ISO 68 oil (an oil having a viscosity of 68 centistokes (cSt) at 40 °C), when diluted in the ratio of 3 parts oil sample to one part heptane, will flow at approximately 0.4 mL⁄min.

9.1.2.1 If the viscosity of the solvent/sample mixture is too high, the particles will be retarded in their migration through the fluid toward the magnet pole pieces. This will have the effect of allowing large ferrous particles to penetrate further along the length of the ferrogram than would normally be the case. Worse, however, from an operational viewpoint, is that the fluid will be so viscous that it will form a large crown and spill over the non-wetting barrier stripe on the ferrogram surface necessitating the ferrogram preparation process to repeated. If the solvent/sample viscosity is too low, the ferrous (magnetic) particles will migrate too quickly toward the pole pieces and many small particles will be deposited at the entry region of the ferrogram (where the fluid first touches down on the ferrogram) along with the large ferrous particles. Furthermore, the fast flow rate may cause the fluid to spill over at the exit end of the ferrogram instead of flowing into the drain tube. Therefore, some judgment is required to dilute the sample properly.

9.1.2.2 In general, oils with ISO grades up to 68 will flow properly if diluted in the ratio of 3 parts sample to one part solvent. More viscous oils require more solvent, 3 parts sample to 2 parts solvent is recommended.

9.1.2.3 To a large extent, the effect of viscosity on the deposition pattern is self-compensating. The higher the viscosity, the longer it takes for the solvent/sample mixture to flow, and the longer it takes for the particles to flow through the fluid due to the viscous resistance the particles experience. Likewise, when the viscosity is low, the sample flows down the substrate more quickly, and the particles move more quickly toward the magnet assembly because the viscous resistance they experience is correspondingly less. Therefore, the resulting deposition pattern on the ferrogram is more or less the same even though the solvent/sample viscosity varies to some degree.

9.1.3 Remove the blank ferrogram glass substrate from its plastic cover and position it so that the marking dot on the glass surface is in the lower left-hand corner of the ferrograph magnet channel. The purpose of the marking dot is to identify the side of the glass having the non-wetting barrier stripe.

9.1.4 Withdraw the spring-loaded position pin on the left side of the magnet assembly. Place the glass substrate on the magnet assembly. Position the upper end of the glass substrate so that it rests on the small step at the back of the magnet assembly slot. Allow the exit end of the glass substrate to rest on the magnet assembly surface.

9.1.4.1 This causes the glass substrate to be elevated at the entry end relative to the exit end. The purpose is to reduce the magnetic field strength at the entry end so that small particles are not deposited as quickly as they might otherwise be. This gives better separation between large and small magnetic particles as they are deposited on the substrate.

9.1.5 Gently release the positioning pin so that the glass substrate is held firmly in place against the right edge of the magnet channel.

9.1.6 Complete ferrogram preparation following specific manufacturer's instructions.

9.1.6.1 This will entail allowing the prepared solvent/sample mixture to flow slowly across the glass surface of the ferrogram slide during which time ferromagnetic particles will be deposited on the glass surface in an orderly fashion according to size. Weakly magnetic and nonmagnetic particles will be deposited randomly along the length of the ferrogram. Soot particles, as found in diesel engine lubricating oil samples, are repelled by the magnetic field of the ferrograph and flow off the ferrogram to waste. After the prepared sample has flowed completely across the ferrogram surface, the remaining sample on the ferrogram surface is rinsed using an appropriate solvent, per specific manufacturer's instructions. After rinsing, remaining solvent is allowed to dry and the separated wear and contaminant particles become firmly adhered to the glass surface of the ferrogram.

9.1.7 After the surface is completely dry, withdraw the spring-loaded positioning pin and lift the ferrogram off the magnet assembly. The ferrogram is now ready for microscopical examination.

9.1.7.1 Use caution—the ferrogram must be lifted straight up off the magnet assembly. If the ferrogram is slid along the magnet assembly, the magnetic field will twist and distort the strings of ferrous particles on the ferrogram surface. To lift the ferrogram off smoothly, it is recommended that the exit end be lifted up first while the front end still rests on the small step at the back of the top plate slot. Once the back end has been raised approximately 2 cm, the entire ferrogram can be lifted away from the magnet assembly.

9.2 *Ferrogram Analysis Procedure:*

9.2.1 Place the ferrogram onto the stage of the microscope and begin inspection of the particles thereon. Table 1 summarizes the suggested procedure for analysis of a ferrogram. Step 1 suggests viewing of the ferrogram at low magnification with red reflected and green transmitted light. At this time, it may be determined whether the deposit on the ferrogram is too heavy for proper particle identification. If the deposit at the ferrogram entry is so heavy that particles are piled on top of one another it will be very difficult to determine the types and relative amounts of particles present. Some piling up is tolerable, but strings of ferrous particles should be separated and ideally particles should be deposited in a single layer. If too many particles are present, it is recommended that the sample be diluted 9:1 with particle free oil and a new ferrogram be prepared with 3 mL of the diluted sample. This will result in a ferrogram prepared from 0.3 mL of sample, rather than the standard 3 mL sample volume. It may happen that the ferrogram prepared from 0.3 mL of sample again has too many

TABLE 1 Suggested Procedure for Analysis of a Ferrogram

Step	Magnification	Reflected	Transmitted	Comments
1	100× (low)	Red	Green	View the entry region to determine if too many particles are deposited on the ferrogram. If so, a new ferrogram needs to be prepared from diluted sample. Otherwise, proceed to Step 2.
2	100× (low)	Red	Green	Look for severe wear particles at entry by presence of bright red particles. Rubbing wear particles are too small to be resolved at this low magnification and appear black. Therefore, if only rubbing wear particles are present on the ferrogram, no red particles will be observed. Large wear particles will appear bright red against an otherwise green background. Scan length of ferrogram looking for severe nonferrous wear particles, nonmetallic particles, or a heavy deposit at the exit end typical of corrosive wear.
3	400× or 500× (medium)	White	Green	Examine the entry deposit making a preliminary judgment as to the specific types of wear particles present such as severe wear, rubbing wear, chunks, etc. A preliminary judgment of dark metallo-oxides must be confirmed at high magnification because particles that are not flat will appear dark. Scan the length of the ferrogram looking for nonferrous metal particles and other distinctive features such as nonmetallic particles, friction polymers, fibers, etc.
4	800× or 1000× (high)			Most particle types can be recognized at medium magnification, but high magnification provides critical details necessary to complete the analysis. Spheres, fine cutting wear particles and small spots of temper color on the surfaces of particles indicative of high heat during generation can be distinguished only at high magnification. Because of the high numerical aperture of the highest magnification lens that provides good light gathering ability, jagged free metal particles may be distinguished from dark metallo-oxides. Friction polymers are recognized by the presence of fine metal particles in an amorphous matrix whereas nonmetallic amorphous particles do not contain fine metal particles. Red reflected and green transmitted light is useful for identifying friction polymers because of the greater contrast provided.
5	100× (low)	OFF	POL	Use polarized transmitted light to identify nonmetallic crystalline particles. These will appear bright in an otherwise dark field.
6	400× or 500× (medium)	POL	OFF	Use polarized reflected light to determine surface characteristics of particles. Oxidized surfaces of metal particles will depolarize light and thus appear bright. Small nonmetallic particles which may not have been seen at 100× can now be detected. Use high magnification with polarized reflected light if surface characteristics are of particular interest.
7	As Required			Take photos prior to heat treating ferrogram. A polarized light photo may be useful if it is desired to distinguish between organic and inorganic particles. Organic particles will not be as bright after heat treatment. A second photo using the same exposure will show this difference. It may also be useful to photograph strings or ferrous particles before heat treatment as well as any suspected Pb/Sn alloy particles
8				Heat treat ferrogram. (Optional)
9	As Required			Reexamine ferrogram after heat treating. Take photos as necessary

particles in which case a further 9:1 dilution should be made from the already diluted sample and another ferrogram prepared. In this case, the resulting ferrogram will have been made from 0.03 mL of sample. In rare circumstances, the sample may have to be diluted yet again. Once it is determined that the ferrogram under examination has an acceptably dilute deposit, proceed to Step 2 on Table 1.

9.2.2 At low magnification using red reflected and green transmitted illumination, large wear particles will appear bright red because of their highly reflective surfaces. Wear particles are metallic except for some few special machines that use nonmetallic components in the oil-wetted path, such as diesel engines with ceramic pistons. Metallic particles always block light, even in exceedingly thin sections. Therefore, metals may be recognized because they block transmitted light. Very often, metal wear particles are present as free metal (not compounded with other elements, such as oxygen) and will therefore have bright, lustrous reflective surfaces. Nonmetallic particles (see 3.2.15) appear partially green because they permit the transmission of light.

9.2.2.1 Metallic particles, even when extremely thin, block the passage of light. Conversely, nonmetallic materials, at least in thin sections such as would be found in oil samples in the few to tens of µm thick range, allow passage of light and therefore appear partially transparent when examined using transparent light. Nonmetallic polycrystalline particles, such as sand (primarily SiO_2), disrupt polarized light and therefore appear bright in an otherwise dark field. Nonmetallic amorphous materials, such as glass or many organic materials, do not disrupt polarized light and therefore remain dark.

9.2.2.2 For common machines, engines and hydraulic systems, most components are composed of iron (ferrous) alloys, that is, steel or cast iron. These particles are magnetic and are separated preferentially on a ferrogram. The largest ferrous particles are deposited near the entry area of the ferrogram, that is, where the sample first touches down onto the glass surface of the ferrogram. As the sample travels down the length of the ferrogram, the ferrous particles deposited become smaller and smaller. By the exit end of the ferrogram, they are submicron in size. Ferrous particle are deposited in strings by the magnetic field of the ferrograph, with their ends touching and their longest dimension perpendicular to the direction of flow along the ferrogram. See Fig. 1.

9.2.2.3 Nonferrous metal wear particles are identified by their nonmagnetic deposition pattern on ferrograms. Instead of aligning with the magnetic field in strings as do ferrous particles, nonferrous particles are deposited with random orientation and may be found along the entire length of the ferrogram regardless of size. By examining the length of the ferrogram at low magnification with red reflected and green transmitted light, large nonferrous metal wear particles will be obvious because they will appear bright red against an otherwise green background.

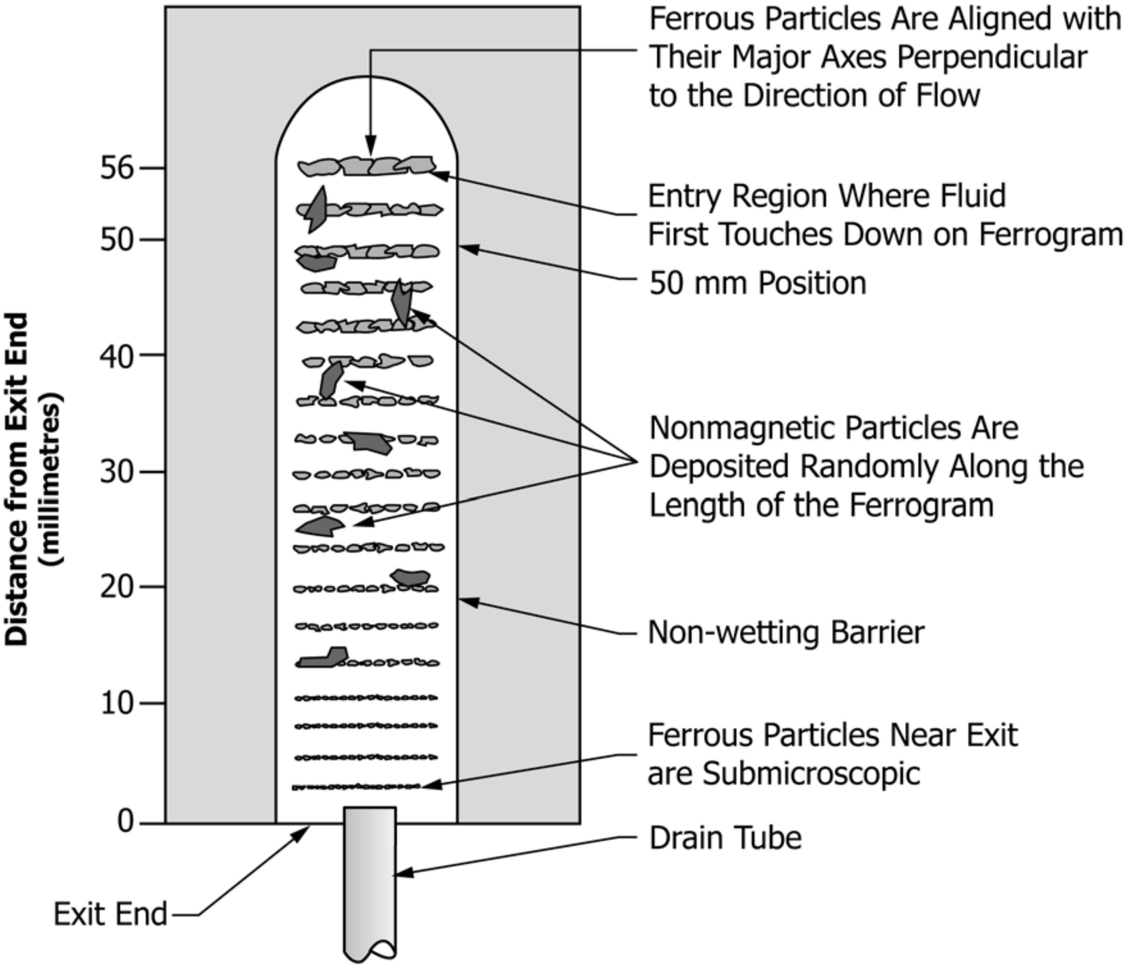

FIG. 1 Deposition Pattern on a Ferrogram

9.2.2.4 Ferrous alloys, such as many stainless steels, may be nonmagnetic, but these alloys are practically never used in oil-wetted tribological contact because they tend to gall and are, therefore, unsuitable wear materials.

9.2.2.5 Examination of the ferrogram at low magnification using red reflected and green transmitted light will indicate whether there are large metal particles present on the ferrogram. Small metal particles will not be large enough to appear bright red at low magnification. Large ferrous particles will be present at or near the ferrogram entry area and large nonferrous particles may be deposited anywhere along the length of the ferrogram. If bright red particles are seen at low magnification, then large metal particles are present, often an indication of abnormal wear modes.

9.2.3 Proceed to Step 3, examination at medium magnification with white reflected and green transmitted light. At the ferrogram entry area, ferrous particles will be aligned in strings. Reference may be made to Table 2 and classification may begin regarding the types of free metal ferrous wear particles are found on the ferrogram. Classification of wear particles is done according to size and shape as summarized in Table 2. Fig. 2 is used, both as a worksheet while examining a ferrogram and also as a means of reporting results to others, along with optional photomicrographs.

9.2.3.1 It is implicitly understood that the wear particles being classified are ferrous since ferrography is a ferromagnetic separation technique. If nonferrous wear particles are present, the type(s) may be indicated in the comments section

TABLE 2 Distinction Among Free Metal Particles

Particle Type	Size (major dimension)	Shape Factor (major/minor dimension)
Rubbing Wear Particles	<15 µm in major dimension	Thin, >5:1, usually about 10:1
Rubbing Wear Particles	<5 µm in major dimension	Any shape except curved or curled
Abrasive Wear Particles	Any size	Long, thin, curled or curved, ribbon like
Severe Wear Particles	>15 µm in major dimension	>5:1 but <30:1
Chunks	>5 µm in major dimension	<5:1
Reworked (Laminar) Particles	>15 µm in major dimension	>30:1

Sample No. ____________
Ferrogram Number ____________
Organization ____________
Equipment Type ____________
Sample Date ____________
Volume of Undiluted Sample to Make Ferrogram ________ mL

Date ____________
Equipment Serial No. ____________
Total Operating Hours ____________
Oil Type ____________
Time or Distance on Oil ____________

Types of Particles	None	Few	Moderate	Heavy
Rubbing Wear Particles				
Severe Wear Particles				
Abrasive Wear Particles				
Chunks				
Reworked (Laminar) Particles				
Spheres				
Dark Metallo-Oxide Particles				
Red Oxide Particles				
Corrosive Wear Debris				
Nonferrous Metal Particles				
Nonmetallic Crystalline Particles				
Nonmetallic Amorphous Particles				
Friction Polymers				
Fibers				
Other (Specify)				

Considered Judgment of Wear Situation:	Normal	Caution	Critical

Comments:

FIG. 2 Ferrogram Analysis Report Sheet

of Fig. 2. At least one or both tribological surfaces in nearly all machines are ferrous (steel or cast iron), thus it is rare that a sample contains only nonferrous wear particles.

9.2.3.2 The classification scheme suggested by Table 2 depends solely on size and shape.

9.2.4 During break-in of a tribosurface, a unique layer is formed at the surface. Break-in is the transition from the "as finished" condition to a smooth low wearing surface. Mechanical work at the surface under the influence of load in the presence of lubricant causes the formation of a thin layer (~ 1 µm thick for steel) of short range crystalline order. This layer exhibits great ductility and may flow along the surface a distance hundreds of times its thickness. Rubbing wear particles are generated by exfoliation of parts of this layer. As long as only rubbing wear particles are observed, the surfaces from which they came may be assumed to be in a smooth stable condition. Disassembly of reciprocating engines that were producing only rubbing wear particles show extremely smooth, mirror like surfaces.

9.2.5 Rubbing wear particles are sometimes called "normal rubbing wear" particles. Objections have been raised that wear of any type should not be considered "normal," although in the context of the design of a specific machine, the presence of rubbing wear particles may be the most benign wear condition that can be expected. Some mechanical designs, such as the shaft of a steam turbine rotating on a journal bearing, generate a full-film wedge of lubrication that effectively separates the two wearing surfaces such that virtually no wear particles are generated. Such mechanical systems are known to run for years without appreciable wear. However, incorporating full-film lubrication between all wearing surfaces in machines of practical design is not a reality, so some level of wear must be tolerated. Therefore, when rubbing wear particles are observed, the surfaces that generated them will eventually wear out. The salient question is whether the machine under observation will continue to operate for its intended lifetime. In this context, rubbing wear particles may be considered normal.

9.2.5.1 Small black oxides of iron are often observed in samples from reciprocating engines. These are in the same size range as rubbing wear particles and as long as their size remains small they are thought to come from surfaces in smooth stable condition. Black oxides of iron are not called out as a separate particle category on Fig. 2.

9.2.5.2 Severe sliding wear begins when stresses increase due to load, speed, or increase in friction, or a combination of these factors. Surface stresses cause cracks to form in the subsurface and to be propagated in the direction of sliding. Repeated cycles over the same surface cause cracks to coalesce such that particles break free. Sliding wear particles exhibit surface striations, have straight, often parallel edges, and typically have a length to thickness ratio of 10:1 or greater. As conditions become more severe within this wear mode, particles become larger, the ratio of large to small particles increases and the striations and straight edges on particles become more prominent. Increasing conditions of load, speed and friction eventually cause catastrophic wear in which complete surface break down occurs and extremely large particles are generated, usually accompanied by screeching noise as fractures propagate rapidly through the subsurface. See Reda, et al,[3] for more information on the regimes of sliding wear.

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[3] Reda, A. A., Bowen, E. R., and Westcott, V. C., "Characteristics of Particles Generated at the Interface Between Sliding Steel Surfaces," *Wear*, 34, 1975, pp. 261-273.

9.2.5.3 For sliding surfaces of approximately equal hardness the presence of fine abrasive contaminants, such as sand, dust or dirt in the lubrication system, causes a significant increase in the generation of rubbing wear particles. A ferrogram will also reveal the contaminant particles. Close examination of the rubbing wear particles often indicate they are somewhat crescent shaped in this situation. If the oil is cleaned and the ingression of contaminants prevented, the concentration of rubbing wear particles will decrease to levels typical for that type of machine indicating the internal wearing surfaces are again in a smooth, stable condition.

9.2.5.4 For rolling contacts of approximately equal hardness the presence of fine abrasive contaminants, such as sand, dust or dirt in the lubrication system, also causes a significant increase in the generation of rubbing wear particles as is the case for sliding contacts. However, even though surface damage may heal to some extent upon removal of contaminants from the lubricant, the passage of contaminants through the rolling contact increases tensile stress at some depth below the surface likely initiating cracks that ultimately lead to fatigue spalling.

9.2.6 Severe wear particles, for the purpose of ferrographic analysis, are defined as being >15 µm in major dimension and having a length to thickness ration between 5:1 and 30:1. If they are thicker, then they are classified as chunks. If a particle is very thin, sometimes with holes, implying it has been flattened by a rolling contact, it is classified as an reworked particle. The term "reworked particle" is new to ferrography practice, being introduced recently to replace the term "laminar particle" which many think inappropriate.

9.2.6.1 Having determined that severe wear particles are present, it is possible to distinguish if these were generated by a sliding or rolling contact. Severe sliding wear particles are longer than wide, tend to have straight edges and often show lengthwise surface striations. Surfaces from which severe sliding wear particles are generated show evidence of scoring. Severe wear particles from rolling contact fatigue are smooth flat platelets, more or less as long as wide with jagged irregular edges. Rolling contact fatigue particles are thicker than sliding wear particles and may sometimes be in the chunk category, where thickness is less than five times length. Particles from combined rolling and sliding, such as are generated from meshing gear teeth, may show combinations of these characteristics. Gear wear particles from the pitch line where the contact is rolling look like rolling contact fatigue particles and particles from the tips or roots of the gear teeth look like sliding wear particles. This may aid in determining the site of wear when examining gear oil samples. The comments section of Fig. 2 is used to indicate if severe wear is predominantly rolling or sliding.

9.2.7 Abrasive wear particles, sometimes called cutting wear particles, are readily distinguished by their long, thin, curved, curled and ribbon-like appearance. In most cases, these are generated by three-body abrasive wear in which hard abrasive particles become embedded in the softer of the two tribological components and abrasive wear particles are cut from the harder of the two sliding surfaces. More rarely, two-body abrasive wear occurs, such as when a misaligned or fractured machine part penetrates its wearing pair, generating long, curved particles. These tend to be larger than those produced by ingression of hard abrasive contaminant particles such as sand.

9.2.8 Chunks are >5 µm in major dimension and are more or less equiaxed with a major dimension to minor dimension ratio <5:1. Particles are classified as chunks regardless of surface texture and may be smooth or craggy. The presence of chunks indicates surface damage is occurring in the machine being sampled.

9.2.9 Reworked particles are large and thin and are most likely due to thicker wear particles having been squeezed through a rolling contact. Not only are reworked particles an indication that large particles are present in the machine being sampled, but their passage through a rolling contact is likely to initiate subsurface cracking that eventually results in rolling contact fatigue.

9.2.10 Spheres may be ferrous, nonferrous or nonmetallic depending upon how the were generated. When viewed in white reflected and green transmitted light, metallic spheres will have a white bright center surrounded by a dark ring. The white bright center appears larger as magnification is increased. Ferrous spheres are deposited in strings along with other ferrous particles aligned by the magnetic field of the ferrograph. Nonferrous metal spheres may be deposited anywhere along the length of the ferrogram. Ferrous spheres have been reported as a precursor to rolling contact fatigue and will be present with a rather tight size distribution and are typically less than 5 µm. Ferrous spheres are readily generated by extraneous sources, such as welding, grinding, and machining. Ferrous spheres are plentiful as aerosols in steel mills. Ferrous spheres are also present in fly ash from coal burning. Fly ash also contains numerous glass spheres. These are transparent and appear green in white reflected and green transmitted light. Ferrous and glass spheres from welding, grinding, machining, steel mills and coal burning all have a wide size distribution, from submicron to tens of micrometers. Step 4, Table 1, recommends using the highest available magnification to clarify the size of spheres as well as determining other fine features of the particles being examined.

9.2.11 Dark metallo-oxide particles are partially oxidized ferrous wear particles. These may be recognized because they behave ferromagnetically and therefore align themselves in strings along with other ferrous particles. These particles have metallic cores and consequently block transmitted light. Dark metallo-oxide particles are produced under conditions of poor lubrication and are an indication of severe surface deterioration.

9.2.12 Red oxide particles or rust particles are composed of Fe_2O_3, and are not ferromagnetic, although strongly paramagnetic. Red oxides have positive attraction to a magnetic field, but not nearly as strongly as do ferromagnetic materials such as steel, cast iron, and black oxides. Consequently, they are deposited on a ferrogram in a more random manner than ferromagnetic particles. Red oxide particles are usually present in the form of crystalline agglomerates and are often hydrated.

Red oxide particles are caused by water in the lubricating oil system and by corrosion. Red oxide particles are also generated by fretting wear.

9.2.12.1 Sometimes the red oxide of iron is present as red oxide sliding wear particles. These are Fe_2O_3 and are formed by poorly lubricated sliding wear. They are not ferromagnetic, although strongly paramagnetic and therefore deposit between strings of wear particles and their major axis may not be aligned in the same direction as ferromagnetic particles. They have more or less the same shape as severe sliding wear particles, that is, they are flat and longer than they are wide. When viewed in white reflected and green transmitted light, they display a gray, reflective surface, but if examined only with transmitted light they will reveal themselves as transparent thus confirming their nonmetallic composition. A separate category for these particles is not provided on Fig. 2 as they occur only occasionally perhaps because rather narrow tribological conditions are necessary for their generation. A category of "Other, specify" is the last choice on Fig. 2 and should be used if red oxide sliding wear particles are identified.

9.2.13 White nonferrous metal wear particles, for example, aluminum, chromium, silver, magnesium, zinc, lead, tin and titanium, are essentially indistinguishable from one another without further testing such as by X-ray fluorescent spectroscopy in conjunction with scanning electron microscopy, or by heat treating the ferrogram, or by treatment with acids or bases.

9.2.13.1 Copper alloy metal wear particles may be identified by their nonferrous deposition pattern on a ferrogram and by their characteristic yellow color. The only other common metal with yellow color is gold and few machine parts are gold or gold coated, except for certain exotic applications.

9.2.14 Corrosive wear particles are generated when lubricating oil becomes acidic. Corrosive wear particles are recognized by a heavy deposit at the exit end of the ferrogram. Most of these particles are below the lower limit of resolution of the microscope and thus may be described as submicron.

9.2.14.1 In cases of severe corrosion, large oxidized flakes may be generated from metallic surfaces. In ferrography, such flakes are classified as either red oxide particles (rust) or nonmetallic crystalline particles. In ferrography, the category of corrosive wear particles is reserved for very fine particles as described above,

9.2.15 Nonmetallic crystalline particles are recognized by their partial transparency in transmitted light and their brightness in polarized transmitted light as suggested by Step 5, Table 1. These are typically due to the ingression of dust or dirt into the lubricating oil system. Abrasive wear particles are often associated with the presence of nonmetallic crystalline particles. Silica (SiO_2) particles are commonly found in sand, dust and dirt and appear on ferrograms as nonmetallic crystalline particles. Step 6, Table 1, suggests using polarized reflected light at medium magnification to determine if the surfaces of wear particles are oxidized as may occur under conditions of poor lubrication.

9.2.16 Nonmetallic amorphous particles, such as glass and many organic materials, do not disrupt polarized light and therefore remain dark when viewed in transmitted polarized light. Nonmetallic amorphous particles may be glass or they may be organic, such as polymerized material formed as the oil degrades. Heat treating of the ferrogram may serve to clarify this distinction as organic particles will char, shrivel or burn at sufficiently high temperature. Glass will be unaffected.

9.2.17 Friction polymers are recognized by metal wear particles embedded in a flat amorphous matrix. Friction polymers are thought to be created by high stress on lubricant in a critical contact and are apparently the result of polymerization of oil molecules to form a large coherent structure.

9.2.18 Fibers are long, thin nonmetallic particles and may be from filters that are tearing or shredding. Paper of various types is often used in oil filters. Cellulose fibers, the main constituent of the cell walls of plants such as wood, paper, cotton and hemp for example, have a ribbon-like structure and appear very bright and multicolored in polarized transmitted light. Other fiber types may also be present. Glass fibers (fiber glass) are recognized by their round cross-section. Only the edges of glass fibers appear bright in polarized light. Asbestos is a generic name for several mineral fiber types. These are distinguished from other fibers by their fine size and when viewed under the microscope they appear to split into ever finer fibers.

9.2.19 Other particle types are sometimes found on ferrograms but are not specifically categorized on Fig. 2 because of their infrequent occurrence. Among these are red oxide sliding wear particles, break-in wear particles, coal particles, carbon flakes, asbestos fibers, molybdenum disulfide particles and black oxide particles. Further information regarding wear particle identification, along with color photomicrographs, may be found in the *Wear Particle Atlas (Revised)*.[4]

9.2.20 Step 7, Table 1, suggests taking photomicrographs of various particles of interest for documentation and reporting purposes. Taking photomicrographs at this time is recommended if the optional step of heat treating the ferrogram will be exercised.

9.3 *Optional Procedure for Heat Treating a Ferrogram to Aid in Metallurgical Identification:*

9.3.1 *Purpose*—This is an optional step that is used primarily to distinguish between broad classes of ferrous metallurgy, namely low alloy steel and cast iron. Heating the ferrogram forms a uniformly thick oxide layer on ferrous particles that result in temper colors caused by destructive interference of incident white light. Most white colored nonferrous metal particles are unaffected by heat treatment, but lead, tin and lead/tin alloy particles are grossly affected because the heat treatment temperature is above the melting temperature of lead and tin. Consequently, this procedure is useful for identifying lead/tin metallurgy as may be found in the wear of journal bearings. Heat treating also permits distinction between nonmetallic crystalline particles and nonmetallic organic particles. In the practical application of ferrography, the practitioner often has knowledge of the materials of construction from the device the sample was taken and heat treating of the ferrogram may not be needed. On the other hand, heat treating may give

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[4] Anderson, D. P., "Wear Particle Atlas (Revised)," Report NAEC-92-163, prepared for the Naval Air Engineering Center, Lakehurst, NJ, June 28, 1982, (approved for public release; distribution unlimited).

valuable information regarding which components are wearing abnormally, especially in the case of reciprocating engines, in which the crank shaft is most often steel and the ring/cylinder metallurgy is cast iron.

9.3.2 *Procedure*—Table 1 requires heat treating the ferrogram on a hot plate for 90 s at 330 °C. A surface thermometer is used to measure the surface temperature of a hot plate. Tongs are used to place the ferrogram on the heated surface and to remove the ferrogram after 90 s. Once removed from the hot plate surface, the ferrogram will cool in 5 s to 10 s and may then be reexamined under the microscope.

9.3.3 Upon heat treatment of the ferrogram, steel particles appear blue and cast iron particles appear straw (yellow) to bronze colored. Lead/tin alloys shrink and have a mottled blue/straw appearance.

NOTE 1—Lead/tin particles will first be recognized as nonferrous by their random deposition pattern on the ferrogram (see 9.2.2.3). Other white nonferrous metals used in oil-wetted contacts including aluminum, chromium, silver and titanium are unaffected. Organic particles will char, melt, shrivel or vaporize upon heat treatment, whereas inorganic particles such as silica found in sand, dust and dirt remain unaffected. Step 9, Table 1, suggests taking additional photomicrographs for reporting and documenting purposes.

9.3.4 Further explanation and details of this technique may be found in Anderson[4] and Barwell,[5] including instructions for heat treating to higher temperatures that my be required only in rare circumstances.

NOTE 2—It is not the intent of this practice to establish or recommend normal, cautionary or critical alert limits for any machinery or fluids. Such limits should be established in conjunction with advice and guidance from the machinery manufacturer or maintenance group.

10. Keywords

10.1 contaminant particles; condition monitoring; ferrography; filter patch; in-service lubricants; membrane filtration; optical microscopy; wear; wear particles

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[5] Barwell, F. T., Bowen, E. R., and Westcott, V. C., "The Use of Temper Colors in Ferrography," *Wear*, 44, 1977, pp. 163–171.