BS ISO 20032:2013

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Method for evaluation of tensile properties of metallic superplastic materials

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National foreword

This British Standard is the UK implementation of ISO 20032:2013. It supersedes BS ISO 20032:2007 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee ISE/101/2, Ductility testing.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Method for evaluation of tensile properties of metallic superplastic materials

Méthode de détermination des caractéristiques de traction des matériaux métalliques superplastiques

Reference number
ISO 20032:2013(E)

Contents

Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2. www.iso.org/directives

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The committee responsible for this document is ISO/TC 164, *Mechanical testing of metals*, Subcommittee SC 2, *Ductility testing*.

This second edition cancels and replaces the first edition (ISO 20032:2007) of which it constitutes a minor revision.

Introduction

Superplastic forming requires the characterization of metallic superplastic materials. The tensile test specified in this International Standard permits the evaluation of superplastic properties, such as superplastic elongation, flow stress, strain rate sensitivity exponent (m-value), stress-strain relation and flow stress-strain rate relation.

Method for evaluation of tensile properties of metallic superplastic materials

1 Scope

This International Standard specifies a method for evaluating the tensile properties of metallic superplastic materials which exhibit what is called "Fine-Grained Superplasticity", without significant work-hardening or dynamic microstructure evolution, by means of a tensile test at constant cross-head velocity, for flat-form test pieces, without an extensometer attached.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 6892-1, *Metallic materials — Tensile testing — Part 1: Method of test at room temperature*

ISO 6892-2, *Metallic materials — Tensile testing — Part 2: Method of test at elevated temperature*

ISO 7500-1, *Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system*

ISO 80000-1, *Quantities and units — Part 1: General*

IEC 60584-1, *Thermocouples — Part 1: Reference tables*

IEC 60584-2, *Thermocouples — Part 2: Tolerances*

3 Symbols, terms and definitions

For the purposes of this document, the symbols, terms and definitions given in Table 1 apply.

Table 1 — Symbols, terms and definitions

Symbol	Term	Definition	Unit
Superplasticity			
—	Superplastic state	Deformation conditions on onset of superplasticity and its continuation	—
Test piece			
—	R-type test piece	Test piece that has the shape of a conventional tensile test piece without its parallel portion	—
—	R portion	Principal portion of the R-type test piece to be elongated, which has an arc-like shape between grips	—
b	Width of parallel-sided portion or minimum width of the R portion	Original width of parallel-sided portion of the S-type test piece or original minimum width of the R portion of the R-type test piece	mm
$b_o[i]$	Original width in division, *i*, of the R portion	Original width in a specific division, *i*, of the R portion	mm
$b[i]$	Width in division, *i*, of the R portion	Width in a specific division, *i*, of the R portion after the interrupted test	mm
A	Superplastic elongation	Elongation after fracture in a superplastic state	%
B_g	Grip portion width	Width of grip portion of the S- or R-type test piece	mm
L_o	Original gauge length	Original distance between gauge marks measured by appropriate apparatus with an accuracy of at least 1 % of the distance or 0,01 mm, whichever is greater	mm
L_u	Final gauge length after fracture	Final distance between gauge marks measured after fracture with fracture surfaces placed together with care, so that the centre line of either fracture surface is on a single straight line	mm
L_c	Parallel length	Original length of parallel portion of the S-type test piece	mm
L_t	Total length of test piece	Original total length of test piece	mm
L_g	Grip portion length	Length of grip portion of test piece	mm
L_R	R portion length	Original length of the R portion	mm
ΔL_R	Elongation of the R portion	Increase in the R portion length at any moment during the test	mm
R	Radius of fillet or R portion radius	Original radius of fillet of the S-type test pieces or original R portion radius of the R-type test piece	mm
S_o	Original cross-sectional area of test piece in the reduced section	Original cross-sectional area of a test piece measured by an appropriate apparatus with an accuracy of ±2 % or better	mm^2
$S_o[i]$	Original cross-sectional area in division, *i*, of the R portion	Original cross-sectional area in a specific division, *i*, of the R portion	mm^2
$S[i]$	Cross-sectional area in division, *i*, of the R portion	Cross-sectional area in a specific division, *i*, of the R portion after the interrupted test	mm^2
t	Thickness of test piece	Thickness of the S- or R-type test piece	mm
$t_o[i]$	Original thickness in division, *i*, of the R portion	Original thickness in a specific division, *i*, of the R portion	mm

Table 1 *(continued)*

Symbol	Term	Definition	Unit
$t[i]$	Thickness in division, *i*, of the R portion	Thickness in a specific division, *i*, of the R portion after the interrupted test	mm
Force			
F_{10}	10 % deformation force	Force at 10 % nominal strain	N
Stress			
K	*K* value	A constant-with-stress dimension, which is defined by Formula (5)	MPa
σ_{10}	10 % flow stress	True stress when 10 % nominal strain is achieved	MPa
σ_f	Flow stress	True stress during superplastic deformation	MPa
σ_N	Nominal stress	A load during deformation divided by the minimum area of the original cross-section in the R portion, which is defined for the R-type test piece	MPa
$\sigma[i]$	True stress	A load during deformation divided by the cross-sectional area in a specific division, *i*, of the R portion, which is defined for the R-type test piece	MPa
Strain			
$\varepsilon[i]$	True strain	True strain given by deformation in a specific division, *i*, of the R portion	—
Period			
τ_{inter}	Period required for interrupted test	Period required from the time when the axial force starts to increase linearly against strain in the elastic deformation stage, until elongation of the R portion, ΔL_R, reaches 3 mm	s
Strain rate and *m*-value			
$\dot{\varepsilon}_N$	Nominal strain rate	Crosshead velocity divided by the original parallel length, L_c for the S-type test piece, and divided by the original gauge length, L_o for the R-type test piece	s^{-1}
$\dot{\varepsilon}$	True strain rate	Increment of true strain per unit time	s^{-1}
$\dot{\varepsilon}[i]$	True strain rate during deformation	True strain rate during deformation in a specific division, *i*, of the R portion	s^{-1}
m	*m*-value	Index representing the strain-rate sensitivity of flow stress in superplastic materials	—

4 Principle

The test consists of straining a test piece by a tensile force, for the purpose of determining the superplastic properties, such as superplastic elongation (A), flow stress (σ_f) and strain-rate sensitivity exponent (*m*-value).

The tensile testing is carried out at elevated temperatures and strain rates.

An S-type test piece is employed to evaluate mechanical properties for general superplastic materials, or at an early stage of deformation.

Due to the limitation of furnace length, S-type test pieces are more suitable for lower-strain superplastic testing. R-type test pieces are more suitable for higher-strain superplastic testing, as most of the strain is developed in a small section at the centre of the specimen.

5 Test piece

The shape and dimensions are given in Figures 1 and 2, and Tables 2 and 3, respectively. The thickness of a test piece shall be the same as the sheet thickness of the material. The dimensional tolerance of a test piece shall be in accordance with ISO 6892-1.

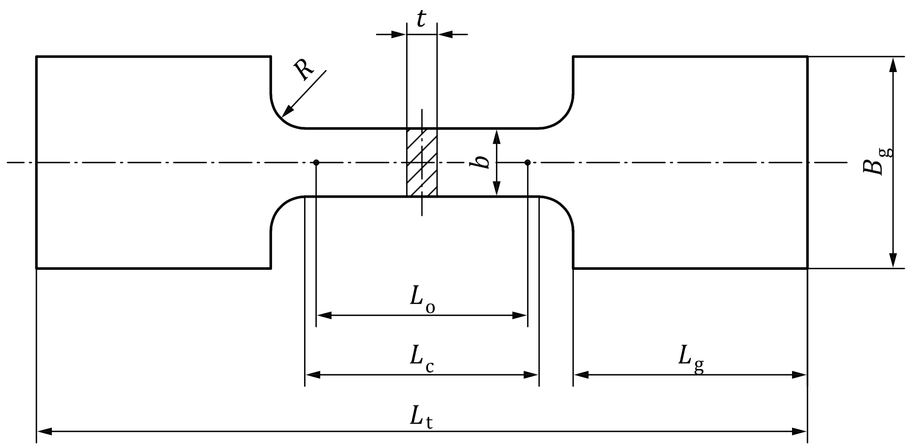


Figure 1 — Shape of flat form test piece (S-type)

Table 2 — Dimensions of S-type test piece

Dimensions in millimetres

Gauge length	**Parallel length**	**Width of parallel- sided portion**	**Radius of fillet**
L_o	L_c	b	R
18	24	6	Not more than 3

The recommended value should be as follows:

$$L_t = 2L_g + L_c + 2R \quad (1)$$

$$B_g = 3b \quad (2)$$

The thickness of the test piece is the same as that of the material to be used.

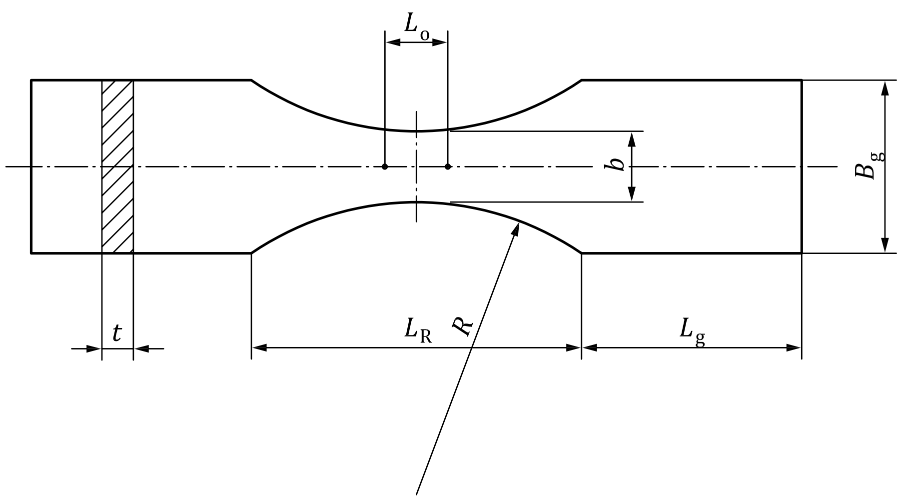


Figure 2 — Shape of R-type test piece

Table 3 — Dimensions of R-type test piece

Dimensions in millimetres

Grip portion width	Minimum width of the *R* portion	R portion length	*R* portion radius	Gauge length
B_g	b	L_R	R	L_o
16	6	30	25	6
L_g should not be less than 20 mm.				

The thickness of the test piece is the same as that of the material to be used.

6 Apparatus

6.1 Testing machine

The testing machine used shall conform to ISO 7500-1. The machine used shall satisfy the condition of constant cross-head velocity. The appropriate class of machine range shall be selected under the agreement between interested parties.

6.2 Clamping device for test pieces

The clamping device shall not be plastically deformed during an elevated temperature test. A recommended method is described in 7.2.

6.3 Heating apparatus

A furnace with a temperature-regulator shall be used for heating the test pieces. During a test, the furnace shall be capable of keeping the test piece temperature over the gauge length uniform and constant within the allowable range of Table 4.

Table 4 — Allowable range of test temperatures

Temperatures in kelvins

Test temperature	≤473	>473 ≤873	>873 ≤1 073	>1 073 ≤1 273	>1 273
Allowable range	Agreed by interested parties	±3	±4	±5	Agreed by interested parties

6.4 Atmosphere

The measurement of the test piece dimensions, especially distances between gauge marks or divisions after the test, shall be enabled and the damage of the device shall be avoided in the environment used. If the test is performed in a controlled atmosphere, the measured force should be corrected by compensating the pressure difference.

6.5 Thermometric apparatus

6.5.1 General

The thermometric apparatus consists of a measuring device and a thermocouple.

6.5.2 Measuring device

The measuring device shall be capable of measuring the temperature of the test piece within the allowable range given in Table 4, over the entire range of the temperature to be measured.

6.5.3 Thermocouple

The thermocouple shall basically conform to IEC 60584-1 and IEC 60584-2. Also, the materials used for the thermocouple shall be capable of withstanding the service temperature and environment.

When a temperature measurement instrument other than a thermocouple is used, it shall be as accurate as or more accurate than a thermocouple.

7 Procedure

7.1 General

Test procedures are described in 7.2 to 7.8. Additions that are not described in this clause shall be in accordance with ISO 6892-1 and ISO 6892-2.

7.2 Method for clamping the test piece

The test piece shall be clamped so that only axial force is applied to the test piece during the test. No axial compression and only a small (<5 % of the σ_{10}) axial tension shall be applied to the clamped test piece to maintain straightness during heating to the specified temperature and holding at this temperature before testing.

7.3 Measurement of the test temperature

The measurement of the test temperature shall be conducted by using thermocouples as defined in 6.5.3. The temperature to be measured should be that of the test piece. Such measurement may be flexible if the correlative temperature between the surface of the test piece and a certain position in the furnace was initially evaluated.

For more precise temperature measurement, at least two thermocouples should be used at separate locations.

Interested parties shall agree on the time of heating the test piece to the test temperature and the holding time at the test temperature before starting the test, provided that such agreement shall be made with full assurance of uniform temperature distribution over the test piece.

The test temperature shall be maintained within the allowable range specified in Table 4 during the test.

7.4 Application of the force

The test shall be performed with a constant cross-head velocity.

7.5 Method for dimensional measurement of test pieces

The dimension of the gauge portion of the test piece shall be measured by adequate measuring equipment with a precision of at least 1 % of a specified dimension, or 0,01 mm, whichever is greater. Each end of the original gauge length shall be marked by means of fine marks or scribed lines, but not by notches which could cause premature fracture.

7.6 Method for determining the superplastic elongation

The gauge length prior to the test shall be 18 mm for the S-type, or 6 mm for the R-type test pieces.

The superplastic elongation shall be determined by Formula (3). The value shall be rounded off to two significant figures in accordance with ISO 80000-1.

$$A = \frac{L_u - L_o}{L_o} \times 100 \tag{3}$$

where

A is the superplastic elongation (%);

L_u is the final gauge length after fracture (mm);

L_o is the original gauge length (mm).

The 10 % flow stress shall be given by Formula (4).

$$\sigma_{10} = \frac{1{,}1 F_{10}}{S_o} \tag{4}$$

where

σ_{10} is the 10 % flow stress (MPa);

F_{10} is the force at 10 % nominal strain (N);

S_o is the original cross-sectional area of the test piece (mm^2).

7.7 Determination of strain rate sensitivity exponent (*m*-value)

At a given temperature and microstructure, the relation between flow stress, σ_f, and true strain rate, $\dot{\varepsilon}$, during superplastic deformation is given in Formula (5). Strain hardening is not taken into account for the formula. This International Standard should be applied to the materials in which the strain hardening effect can be neglected using Formula (5).

$$\sigma_f = K \dot{\varepsilon}^m \tag{5}$$

The true strain rate, $\dot{\varepsilon}$, is given by

$$\dot{\varepsilon} = \frac{1}{1 + \varepsilon_N} \dot{\varepsilon}_N = \frac{1}{1 + \varepsilon_N} \frac{V}{L_c} \tag{6}$$

where

V is the cross-head velocity (mm/s);

ε_N is the nominal strain (-);

L_c is the parallel length (mm).

Then, exponent m is expressed as in Formula (7)

$$m = \frac{d(\ln \sigma_f)}{d(\ln \dot{\varepsilon})} \tag{7}$$

The *m*-value in Formula (7) can be obtained from the slope of the line in a double logarithmic plot of stress and strain rate. In general, the *m*-value depends on deformation conditions and materials in the range from 0 to 1, and a higher *m*-value leads to larger superplastic elongation.

On one test piece, conduct tests at 5 or more velocities (i.e. jump tests) to obtain the relationship between 10 % flow stress at each cross-head velocity and the corresponding true strain rate, which should be plotted on a log-log scale. This procedure may be repeated on several test pieces to determine the uncertainty in the *m*-value. Linear regression should be applied to this relationship by the method of least squares. Then round off the *m*-value, as the slope of a straight line, to the first decimal place in accordance with ISO 80000-1.

7.8 Determination of the *m*-value with the R-type test piece

The strain rate sensitivity exponent, *m*-value, shall be determined from the force and test-piece dimensions immediately before interruption as follows. The tensile test shall be interrupted when the elongation of the R portion reaches the specified values at two or more different cross-head velocities. The minimum and maximum cross-head velocities shall be selected in such a manner that the difference between them amounts to between twofold and tenfold. Furthermore, it is specified that the amount of elongation of the R portion shall be normally 3 mm ±0,5 mm, that can be regarded as the elongation in the initial deformation. For each test, the true stress $\sigma[i]$ and true strain rate $\dot{\varepsilon}[i]$ at each division of a scale (see Figure 4) shall be determined as described in a) to g).

a) In the middle region of the R portion, five divisions shall be set at intervals of 3 mm in the tensile-axis direction. The test piece shall be placed with its tensile axis horizontal as shown in Figure 3. The division at the centre on the minimum cross-section of the R portion is named as division [0], those at the left side as divisions [−6] and [−3] and those at the right side as divisions [+3] and [+6].

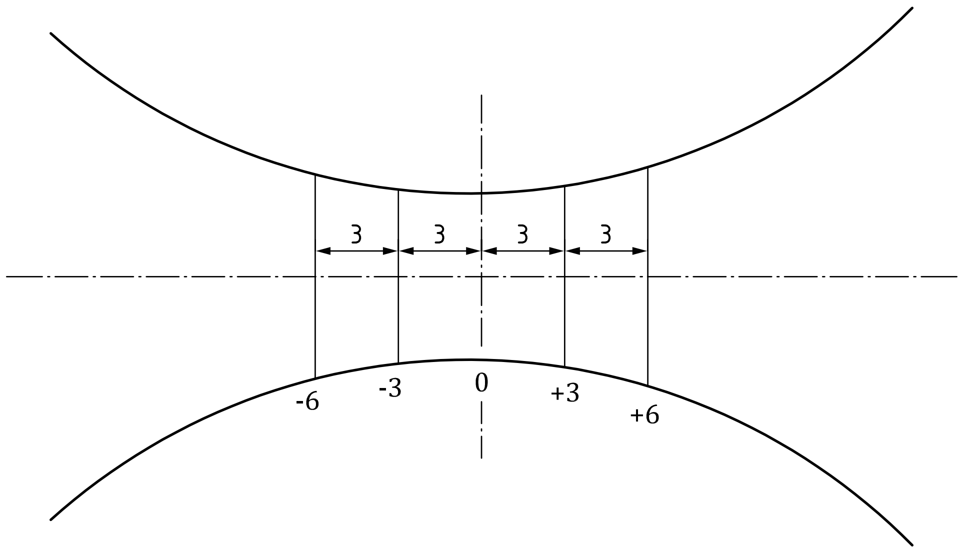


Figure 3 — Divisions set of R-portion

b) The R portion width, $b_0[i]$(i = −6, −3, 0, +3, +6) and thickness $t_0[i]$ at respective divisions on the R portion before the test shall be measured. From measured values, cross-sectional areas $S_0[i]$ at respective divisions shall be calculated.

c) Each test shall be stopped when the elongation of the R portion, ΔL_R reaches 3 mm. The force, *F*, at this point and a period, τ_{inter}, required from the time when the axial force starts to increase linearly against strain in the elastic deformation stage until the test is stopped, shall be recorded.

d) The R portion width $b[i]$ and thickness $t[i]$ at respective divisions after the test shall be measured. For the measurement, adequate measuring equipment, which can accurately measure values at respective divisions, shall be used. From these values, the cross-sectional areas $S[i]$ at respective divisions shall be calculated.

e) The true stress $\sigma[i]$ and true strain $\varepsilon[i]$ at each division shall be calculated as follows:

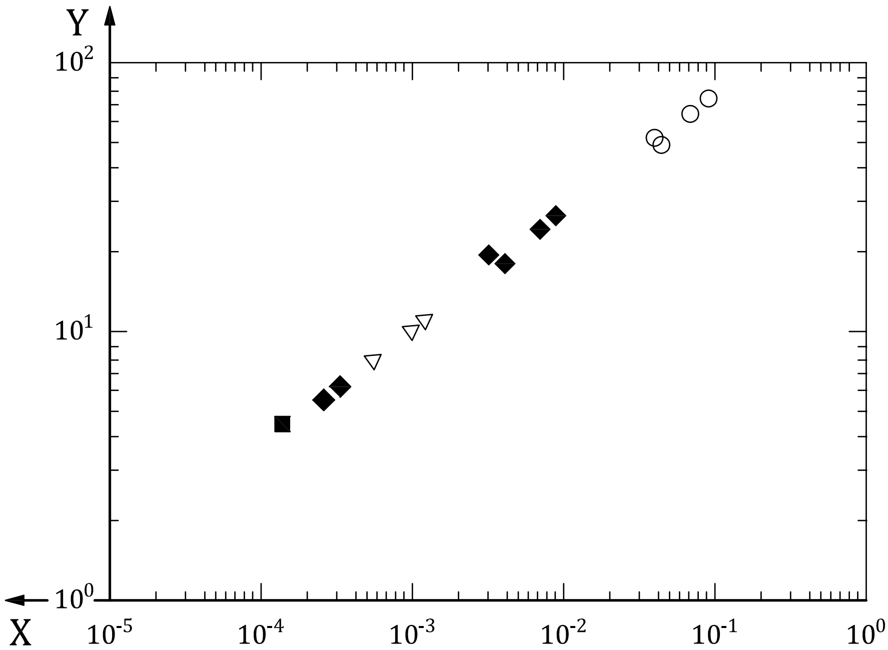
$$\sigma[i] = \frac{F}{S[i]} \tag{8}$$

$$\varepsilon[i] = \ln \frac{S_o[i]}{S[i]} \tag{9}$$

f) The true strain rate $\dot{\varepsilon}[i]$ at each division shall be derived from the true strain $\varepsilon[i]$ and period, τ_{inter} required for the interrupted test using Formula (10).

$$\dot{\varepsilon}[i] = \frac{\varepsilon[i]}{\tau_{inter}} \tag{10}$$

g) For the test results obtained at two or more cross-head velocities, the linear-regression analysis shall be conducted on a log-log plot of true stress σ[*i*] against true strain rate $\dot{\varepsilon}[i]$ at each division. The *m*-value shall be derived from the slope of the line, up to two decimal places (see Figure 4). On this occasion, in order to treat the data at different cross-sections equally, twofold weighting on the datum of division [0] shall be imposed in the determination of a regression line.



Key

X true strain rate, $\dot{\varepsilon}$ (s^{-1})

Y true stress, σ (MPa)

Cross-head velocity (ms^{-1})

■ $5{,}0 \times 10^{-6}$

▽ $1{,}7 \times 10^{-5}$

◆ $1{,}7 \times 10^{-4}$

○ $1{,}7 \times 10^{-3}$

Figure 4 — Example of the relationship between true stress and true strain rate in a superplastic 7475 alloy

8 Test report

The test report shall include the following information, as required by the agreement between the interested parties.

a) **Test materials**

   1) Manufacturer

   2) Designation of materials

   3) Kind or mark

   4) Manufacturing lot number

b) **Dimensions of the test piece**

c) **Description of test equipment**

d) **Test conditions**

   1) Test temperature

   2) Cross-head velocity

   3) Test atmosphere

   4) Heating rate

   5) Holding time prior to testing

   6) Sampling position and direction of test pieces

e) **Test results**

   S-type test piece

   1) Superplastic elongation

   2) 10 % flow stress

   3) *m*-value

   4) Relationship between nominal stress and nominal strain

   5) Relationship between 10 % flow stress and strain rate

   R-type test piece

   1) Superplastic elongation

   2) Maximum nominal stress

   3) Relationship between nominal stress and displacement

   4) *m*-value (in the true strain rate range)

f) **Additional items**

   It is desirable to include the following information.

   1) Chemical composition of materials

   2) Fabrication of materials

3) Heat treatment of materials

4) Grain size of materials

5) Mechanical properties of materials at room temperature

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