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Standard Test Method for Elevated Temperature Tensile Creep Strain, Creep Strain Rate, and Creep Time-to-Failure for Advanced Monolithic Ceramics[1]

This standard is issued under the fixed designation C 1291; 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 test method covers the determination of tensile creep strain, creep strain rate, and creep time-to-failure for advanced monolithic ceramics at elevated temperatures, typically between 1073 and 2073 K. A variety of specimen geometries are included. The creep strain at a fixed temperature is evaluated from direct measurements of the gage length extension over the time of the test. The minimum creep strain rate, which may be invariant with time, is evaluated as a function of temperature and applied stress. Creep time-to-failure is also included in this test method.

1.2 This test method is for use with advanced ceramics that behave as macroscopically isotropic, homogeneous, continuous materials. While this test method is intended for use on monolithic ceramics, whisker- or particle-reinforced composite ceramics as well as low-volume-fraction discontinuous fiber-reinforced composite ceramics may also meet these macroscopic behavior assumptions. Continuous fiber-reinforced ceramic composites (CFCCs) do not behave as macroscopically isotropic, homogeneous, continuous materials, and application of this test method to these materials is not recommended.

1.3 The values in SI units are to be regarded as the standard (see Practice E 380).

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 4 Practices for Force Verification of Testing Machines[2]

E 6 Terminology Relating to Methods of Mechanical Testing[2]

E 83 Practice for Verification and Classification of Extensometers[2]

E 139 Practice for Conducting Creep, Creep-Rupture, and Stress-Rupture Tests of Metallic Materials[2]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[3]

E 220 Test Method for Calibration of Thermocouples by Comparison Techniques[4]

E 230 Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples[4]

E 380 Practice for Use of the International System of Units (SI)[5]

E 639 Test Method for Measuring Total-Radiance Temperature of Heated Surfaces Using a Radiation Pyrometer[6]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

E 1012 Practice for Verification of Specimen Alignment Under Tensile Loading[2]

3. Terminology

3.1 *Definitions*—The definitions of terms relating to creep testing, which appear in Section E of Terminology E 6 shall apply to the terms used in this test method. For the purpose of this test method only, some of the more general terms are used with the restricted meanings given as follows.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *axial strain, ϵ_a, [nd], n*—the average of the strain measured on diametrically opposed sides and equally distant from the specimen axis.

3.2.2 *bending strain, ϵ_b [nd], n*—the difference between the strain at the surface and the axial strain.

3.2.2.1 *Discussion*—In general, it varies from point to point around and along the gage length of the specimen. **[E 1012]**

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[2] *Annual Book of ASTM Standards*, Vol 03.01.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

[5] Discontinued 1997; Replaced by IEEE/ASTM SI-10.

[6] *Annual Book of ASTM Standards*, Vol 15.03.

3.2.3 *creep-rupture test*, *n*— a test in which progressive specimen deformation and the time-to-failure are measured. In general, deformation is greater than that developed during a creep test.

3.2.4 *creep strain, ϵ, [nd]*, *n*— the time dependent strain that occurs after the application of load which is thereafter maintained constant. Also known as engineering creep strain.

3.2.5 *creep test*, *n*—a test that has as its objective the measurement of creep and creep rates occurring at stresses usually well below those that would result in fast fracture.

3.2.5.1 *Discussion*—Since the maximum deformation is only a few percent, a sensitive extensometer is required.

3.2.6 *creep time-to-failure, t_f , [s]*, *n*—the time required for a specimen to fracture under constant load as a result of creep.

3.2.6.1 *Discussion*—This is also known as creep rupture time.

3.2.7 *gage length, l, [m]*, *n*—the original distance between fiducial markers on or attached to the specimen for determining elongation.

3.2.8 *maximum bending strain, ϵ_{bmax}, [nd]*, *n*—the largest value of bending strain along the gage length. It can be calculated from measurements of strain at three circumferential positions at each of two different longitudinal positions.

3.2.9 *minimum creep strain rate, ϵ_{min}, [s^{-1}]*, *n*—minimum value of the strain rate prior to specimen failure as measured from the strain-time curve. The minimum creep strain rate may not necessarily correspond to the steady-state creep strain rate.

3.2.10 *slow crack growth, v, [m/s]*, *n*—subcritical crack growth (extension) which may result from, but is not restricted to, such mechanisms as environmentally assisted stress corrosion, diffusive crack growth, or other mechanisms.

3.2.11 *steady-state creep, ϵ_{ss}, [nd]*, *n*—a stage of creep wherein the creep rate is constant with time.

3.2.11.1 *Discussion*—Also known as secondary creep.

3.2.12 *stress corrosion*, *n*—environmentally induced degradation that initiates from the exposed surface.

3.2.12.1 *Discussion*—Such environmental effects commonly include the action of moisture, as well as other corrosive species, often with a strong temperature dependence.

3.2.13 *tensile creep strain, ϵ_t, [nd]*, *n*—creep strain that occurs as a result of a uniaxial tensile-applied stress.

4. Significance and Use

4.1 Creep tests measure the time-dependent deformation under load at a given temperature, and, by implication, the load-carrying capability of the material for limited deformations. Creep-rupture tests, properly interpreted, provide a measure of the load-carrying capability of the material as a function of time and temperature. The two tests compliment each other in defining the load-carrying capability of a material for a given period of time. In selecting materials and designing parts for service at elevated temperatures, the type of test data used will depend on the criteria for load-carrying capability that best defines the service usefulness of the material.

4.2 This test method may be used for material development, quality assurance, characterization, and design data generation.

4.3 High-strength, monolithic ceramic materials, generally characterized by small grain sizes (<50 μm) and bulk densities near their theoretical density, are candidates for load-bearing structural applications at elevated temperatures. These applications involve components such as turbine blades which are subjected to stress gradients and multiaxial stresses.

4.4 Data obtained for design and predictive purposes should be obtained using any appropriate combination of test methods that provide the most relevant information for the applications being considered. It is noted here that ceramic materials tend to creep more rapidly in tension than in compression **(1, 2, 3)**.[7] This difference results in time-dependent changes in the stress distribution and the position of the neutral axis when tests are conducted in flexure. As a consequence, deconvolution of flexural creep data to obtain the constitutive equations needed for design cannot be achieved without some degree of uncertainty concerning the form of the creep equations, and the magnitude of the creep rate in tension vis-a-vis the creep rate in compression. Therefore, creep data for design and life prediction should be obtained in both tension and compression, as well as the expected service stress state.

5. Interferences

5.1 *Time-Dependent Phenomena*—Other time-dependent phenomena, such as stress corrosion and slow crack growth, can interfere with determination of the creep behavior.

5.2 *Chemical Interactions with the Testing Environment*—The test environment (vacuum, inert gas, ambient air, etc.) including moisture content (for example, % relative humidity (RH)) may have a strong influence on both creep strain rate and creep rupture life. In particular, materials susceptible to slow crack growth failure will be strongly influenced by the test environment. Surface oxidation may be either active or passive and thus will have a direct effect on creep behavior by changing the material's properties. Testing must be conducted in environments that are either representative of service conditions or inert to the materials being tested depending on the performance being evaluated. A controlled gas environment with suitable effluent controls must be provided for any material that evolves toxic vapors.

5.3 *Specimen Surfaces*—Surface preparation of test specimens can introduce machining flaws that may affect the test results. Machining damage imposed during specimen preparation will most likely result in premature failure of the specimen but may also introduce flaws that can grow by slow crack growth. Surface preparation can also lead to residual stresses which can be released during the test. Universal or standardized methods of surface preparation do not exist. It should be understood that final machining steps may or may not negate machining damage introduced during earlier phases of machining which tend to be rougher.

5.4 *Specimen/Extensometer Chemical Incompatibility*—The strain measurement techniques described herein generally rely on physical contact between extensometer components (contacting probes or optical method flags) and the specimen so as to measure changes in the gage section as a function of time. Flag attachment methods and extensometer contact materials must be chosen with care to ensure that no adverse chemical

[7] The boldface numbers in parentheses refer to the list of references at the end of this test method.

reactions occur during testing. Normally, this is not a problem if specimen/probe materials that are mutually chemically inert are employed (for example, SiC probes on Si_3N_4 specimens). The user must be aware that impurities or second phases in the flags or specimens may be mutually chemically reactive and could influence the results.

5.5 *Specimen Bending*—Bending in uniaxial tensile tests can cause extraneous strains or promote accelerated rupture times. Since maximum or minimum stresses will occur at the surface where strain measurements are made, bending may introduce either an over or under measurement of axial strain, if the measurement is made only on one side of the tensile specimen. Similarly, bending stresses may accentuate surface oxidation and may also accentuate the severity of surface flaws.

5.6 *Temperature Variations*—Creep strain is often related to temperature through an exponential function. Thus fluctuations in test temperature or change in temperature profile along the length of the specimen in real time can cause fluctuations in strain measurements or changes in creep rate.

6. Apparatus

6.1 *Load Testing Machine*:

6.1.1 Specimens may be loaded in any suitable testing machine provided that uniform, direct loading can be maintained. The testing machine must maintain the desired constant load on the specimen regardless of specimen deformation with time, either through dead-weight loading or through active load control. The force measuring system can be equipped with a means for retaining readout of the force, or the force can be recorded manually. The accuracy of the testing machine must be in accordance with Practice E 4.

6.1.2 *Allowable Bending*—Allowable bending, as defined in Practice E 1012, should not exceed 5 %. This is based on the same assumptions as those for tensile strength testing (see Ref 4, for example). It should be noted that unless percent bending is monitored until the end-of-test condition has been reached, there will be no record of percent bending for each specimen. The testing system alignment including the test machine, gripping devices (as described in 6.2), and load train couplers (as described in 6.3), must be verified using the procedure detailed in the appendix such that the percent bending does not exceed 5 at a mean stress equal to one half the anticipated test stress. This verification must be conducted at a minimum at the beginning and the end of each test series. An additional verification of alignment is recommended, although not required, at the middle of the test series. Either a dummy or actual test specimen may be used. Tensile specimens used for alignment verification should be equipped with a recommended eight separate longitudinal strain gages to determine bending contributions from both eccentric and angular misalignment of the grip heads. (Although it is possible to use a minimum of six separate longitudinal strain gages for specimens with circular cross sections, eight strain gages are recommended here for simplicity and consistency in describing the technique for both circular and rectangular cross sections.) If dummy specimens are used for alignment verification, they should have the same geometry and dimensions as the actual test specimens as well as an elastic modulus that closely matches that of the test material to ensure similar axial and bending stiffness characteristics.

6.2 *Gripping Devices*:

6.2.1 Various types of gripping devices may be used to transmit the measured load applied by the testing machine to the test specimens. The brittle nature of advanced ceramics requires a uniform interface between the grip components and the gripped section of the specimen. Line or point contacts and nonuniform pressure can produce Hertzian-type stresses leading to crack initiation and fracture of the specimen in the gripped section. Gripping devices can be classed generally as those employing active and those employing passive grip interfaces as discussed in the following sections. Regardless of the type of gripping device chosen, it must be consistent with the thermal requirements imposed on it by the elevated temperature nature of creep testing. This requirement may preclude the use of some material combinations and gripping designs.

6.2.1.1 *Active Grip Interfaces*—Active grip interfaces require a continuous application of a mechanical, hydraulic, or pneumatic force to transmit the load applied by the test machine to the test specimen. Generally, these types of grip interfaces cause a load to be applied normal to the surface of the gripped section of the specimen. Transmission of the uniaxial load applied by the test machine is then accomplished by friction between the specimen and the grip faces. Thus, important aspects of active grip interfaces are uniform contact between the gripped section of the specimen and the grip faces, and constant coefficient of friction over the grip/specimen interface.

(*1*) For cylindrical specimens, a one-piece split collet arrangement acts as the grip interface **(4, 5)**. Generally, close tolerances are required for concentricity of both the grip and specimen diameters. In addition, the diameter of the gripped section of the specimen and the unclamped, open diameter of the grip faces must be within similarly close tolerances to promote uniform contact at the specimen/grip interface. Tolerances will vary depending on the exact configuration used.

(*2*) For flat specimens, flat-face, wedge-grip faces act as the grip interface. Generally, close tolerances are required for the flatness and parallelism as well as wedge angle of the grip faces. In addition, the thickness, flatness, and parallelism of the gripped section of the specimen must be within similarly close tolerances to promote uniform contact at the specimen/grip interface. Tolerances will vary depending on the exact configuration used.

6.2.1.2 *Passive Grip Interfaces*—Passive grip interfaces transmit the load applied by the test machine to the test specimen through a direct mechanical link. Generally, these mechanical links transmit the test loads to the specimen by means of geometrical features of the specimens such as button-head fillets, shank shoulders, or holes in the gripped head. Thus, the important aspect of passive grip interfaces is uniform contact between the gripped section of the specimen and the grip faces.

(*1*) For cylindrical specimens, a multi-piece split collet arrangement acts as the grip interface at button-head fillets of the specimen **(6)**. Because of the limited contact area at the

specimen/grip interface, soft, deformable metallic collets may be used to transfer the axial load to the exact geometry of the specimen. In some cases, tapered collets may be used to transfer the axial load to the shank of the specimen rather than into the button-head radius **(6)**. Generally, moderate tolerances on the collet height must be maintained to promote uniform axial-loading at the specimen/grip interface. Tolerances will vary depending on the exact configuration used.

(*2*) For flat specimens, pins or pivots act as grip interfaces at either the shoulders of the specimen shank **(7, 8)** or at holes in the gripped specimen head **(9, 10)**. Generally, close tolerances of shoulder radii and grip interfaces are required to promote uniform contact along the entire specimen/grip interface as well as to provide for non-eccentric loading. Generally, very close tolerances are required for longitudinal coincidence of the pin and the hole centerlines.

6.3 *Load Couplers*:

6.3.1 Various types of devices (load train couplers) may be used to attach the active or passive grip interface assemblies to the testing machine. The load train couplers, in conjunction with the type of gripping device, play major roles in the alignment of the load train and thus subsequent bending imposed on the specimen. Load train couplers can be classified generally as fixed or non-fixed as discussed in the following sections. Note that the use of well-aligned fixed or self-aligned non-fixed couplers does not automatically guarantee low bending in the gage section of the tensile specimen. Generally, well-aligned fixed or self-aligning non-fixed couplers provide for well-aligned load trains, but the type and operation of grip interfaces as well as the as-fabricated dimensions of the tensile specimen can add significantly to the final bending imposed on the gage section of the specimen. Regardless of the type of load couplers chosen, they must be consistent with the thermal requirements imposed on them by the elevated temperature nature of creep testing. These requirements may preclude the use of some material combinations and load train designs.

6.3.2 *Fixed Load Train Couplers*—Fixed couplers may incorporate devices that require either a one-time, pretest alignment adjustment of the load train which remains constant for all subsequent tests or an in-situ, pretest alignment of the load train which is conducted separately for each specimen and each test. Such devices **(11, 12)** usually employ angularity and concentricity adjusters to accommodate inherent load train misalignments. Regardless of which method is used, alignment verification must be performed as discussed in 6.1.2.

6.3.3 *Non-Fixed Load Train Couplers*— Non-fixed couplers may incorporate devices that promote self-alignment of the load train during the movement of the crosshead or actuator. Generally, such devices rely upon freely moving linkages to eliminate applied moments as the load train components are loaded. Knife edges, universal joints, hydraulic couplers, and air bearings are examples **(7, 11, 13, 14, 15)** of such devices. Although non-fixed load couplers are intended to be self-aligning and thus eliminate the need to evaluate the bending in the specimen for each test, the operation of the couplers must be verified as discussed in 6.1.2.

6.4 *Heating Apparatus*:

6.4.1 The apparatus for and method of heating the specimens must provide the temperature control necessary to satisfy the requirements specified in 6.4.2 without manual adjustments more frequent than once in each 24-h period after load application. It must also satisfy the requirements of the testing environment in 6.4.3.

6.4.2 *Temperature*—The furnace must be capable of maintaining the tensile specimen temperature constant with time to 2 K. The temperature readout device must have a resolution of 1 K or less. The furnace system must be such that thermal gradients are minimal in the tensile specimen so that no more than a 5-K differential exists in the specimen gage length at temperatures up to 1773 K.

6.4.3 *Environment*—The furnace may have an air, inert, or vacuum environment as required. If an inert or vacuum chamber is used, and it is necessary to direct load through bellows, fittings, or seal, then it must be verified that force losses or errors do not exceed 1 % of the applied force.

6.5 *Temperature Measuring Devices*:

6.5.1 The method of temperature measurement must be sufficiently sensitive and reliable to ensure that the temperature of the specimen is within the limits specified in 6.4.2. Depending on the temperature range being used, this can be accomplished with either calibrated thermocouples or pyrometers.

6.5.2 *Thermocouples*:

6.5.2.1 *Calibration*—The thermocouple(s) must be calibrated in accordance with Method E 220 and Tables E 230.[8] For longer tests at higher temperatures, this must be done both before the test is initiated and after the test is completed in order to determine the extent of thermocouple degradation and possible thermal drift during the test.

6.5.2.2 *Accuracy*—The measurement of temperature must be accurate to within 5 K. This includes the error inherent to the thermocouple and any error in the measuring instruments.[9,10]

6.5.2.3 *Extension Wire*—The appropriate thermocouple extension wire must be used to connect a thermocouple to the furnace controller or temperature readout device, or both. Special attention must be accorded to connecting the extension wire with the correct polarity.

6.5.2.4 *Degradation*—The integrity and degree of degradation of used bare thermocouples must be verified before each test. At certain temperatures, oxidation and elemental diffusion of the thermocouple alloys will affect the electromotive force (EMF) of the thermocouple junctions. As a consequence, the EMF of a bare, used thermocouple will no longer correspond to the calibration values determined in the pristine condition. The indicated temperature will therefore be less than the actual temperature. This is a particular problem when the same thermocouple is used for both monitoring and control of temperature. Previously used bare thermocouples must be

[8] Thermocouples should be periodically checked since calibration may drift with usage or contamination.

[9] Resolutions should not be confused with accuracy. Beware of instruments that readout to 1°C (resolution), but have an accuracy of only 10 K or ½ % of full scale (½ % of 1200 K is 6 K).

[10] Temperature measuring instruments typically approximate the temperature-EMF tables, but with a few degrees of error.

replaced (with newly welded and annealed, or cut-back, rewelded, and annealed thermocouples) when calibration at the test temperature reveals an error of >2K. It is preferable to use fully sheathed thermocouples in order to minimize degradation.

6.5.3 *Pyrometers*:

6.5.3.1 *Calibration*—The pyrometer(s) must be calibrated in accordance with Test Method E 639.

6.5.3.2 *Accuracy*—The measurement of temperature must be accurate to within 5 K. This shall include the error inherent to the pyrometer and any error in the measuring instruments.[9],[10]

6.6 *Extensometers*:

6.6.1 The strain measuring equipment must be capable of being used at elevated temperatures. The sensitivity and accuracy of the strain-measuring equipment must be suitable to define the creep characteristics with the precision required for the application of the data.

6.6.2 *Calibration*—Extensometers must be calibrated in accordance with Practice E 83.

6.6.3 *Accuracy*—Extensometers with accuracies equivalent to the B-1 classification of extensometer systems specified in Practice E 83 are suitable for use in high-temperature testing of ceramics. Results of analytical and empirical evaluations at elevated temperatures show that mechanical extensometers **(16)** can meet these requirements. Optical extensometers using flags have gage length uncertainties that will generally prevent them from achieving class B-1 accuracy **(17)**. Empirical evaluations at elevated temperature **(18)** show that these extensometers can yield highly repeatable creep data, however.

6.7 *Timing Apparatus*—For creep rupture tests, a timing apparatus capable of measuring the elapsed time between complete application of the load and the time at which fracture of the specimen occurs to within 1 % of the elapsed time shall be employed.

7. Test Specimens and Sample

7.1 *Specimen Size*:

7.1.1 *Description*—The size and shape of test specimens must be based on the requirements necessary to obtain representative samples of the material being investigated. The specimen geometry shall be such that there is no more than a 5 % elastic stress concentration at the ends of the gage section. Typical shapes include square or rectangular cross-section dogbones and cylindrical button-head geometries, and are shown in Appendix X1. It is recommended, in accordance with Practice E 139 and in the absence of additional information to the contrary, that the grip section be at least four times larger than the larger dimension of either width or thickness of the gage section.

7.1.2 *Dimensions*—Suggested dimensions for tensile creep specimens that have been successfully used in previous investigations are given in Appendix X1. Cross-sectional tolerances are 0.05 mm. Parallelism tolerances on the faces of the specimen are 0.03 mm. Various radii of curvature may be used to adjust the gage section or change the mounting configuration. Although these radii are expected to be larger, resulting in a smaller stress concentration, wherever possible, resort should be made to a finite element analysis to determine the locations and intensities of stress concentrations in the new geometry.

7.2 *Specimen Preparation*—Depending on the intended application of the data, use one of the following specimen preparation procedures:

7.2.1 *Application-matched Machining*— The specimen must have the same surface preparation as that specified for a component. Unless the process is proprietary, the report must be specified about the stages of material removal, wheel grits, wheel bonding, and the amount of material removed per pass.

7.2.2 *Customary Procedure*—In instances where a customary machining procedure has been developed that is completely satisfactory for a class of materials (that is, it induces no unwanted surface damage or residual stresses), then this procedure shall be used. It shall be fully specified in the report.

7.2.3 *Standard Procedure*—In instances where 7.2.1 or 7.2.2 are not appropriate, then 7.2.3 will apply. This procedure will serve as the minimum requirements, but a more stringent procedure may be necessary.

7.2.3.1 *Grinding Process*—All grinding using diamond-grit wheels must be done with an ample supply of appropriate filtered coolant to keep workpiece and wheel constantly flooded and particles flushed. Grinding must be done in at least two stages, ranging from coarse to fine rates of material removal. All machining must be done in the surface grinding mode, and be parallel to the specimen long axis (several specimens are shown in the appendix). Do not use Blanchard or rotary grinding.

7.2.3.2 *Material Removal Rate*—The material removal rate must not exceed 0.03 mm (0.001 in.) per pass to the last 0.06 mm (0.002 in.) per face. Final and intermediate finishing must be performed with a resinoid-bonded diamond grit wheel that is between 320 and 600 grit. No less than 0.06 mm per face shall be removed during the final finishing phase, and at a rate of not more than 0.002 mm (0.0001 in.) per pass. Remove approximately equal stock from opposite faces.

7.2.3.3 *Precaution*—Materials with low fracture toughness and a high susceptibility to grinding damage may require finer grinding wheels at very low removal rates.

7.2.3.4 *Chamfers*—Chamfers on the edges of the gage section are preferred in order to minimize premature failures due to stress concentrations or slow crack growth. The use of chamfers and their geometry must be clearly indicated in the test report (see 10.1.1).

7.2.4 *Button-head Specimen-Specific Procedure*—Because of the axial symmetry of the button-head tensile specimen, fabrication of the specimens is generally conducted on a lathe-type apparatus. The bulk of the material is removed in a circumferential grinding operation with a final, longitudinal grinding operation performed in the gage section to ensure that any residual grinding marks are parallel to the applied stress. Beyond the guidelines stated here, more specific details of recommended fabrication methods for cylindrical tensile specimens can be found elsewhere **(4)**.

7.2.4.1 *Computer Numerical Control (CNC) Precaution*—Generally CNC fabrication methods are necessary to obtain consistent specimens with the proper dimensions within the required tolerances. A necessary condition for this consistency

is the complete fabrication of the specimen without removing it from the grinding apparatus, thereby avoiding building unacceptable tolerances into the finished specimen.

7.2.4.2 *Grinding Wheels*—Formed, resinoid-bonded, diamond-impregnated wheels (minimum 320 grit in a resinoid bond) are necessary to fabricate critical shapes (for example, button-head radius) and to minimize grinding vibrations and subsurface damage in the test material. The formed, resin-bonded wheels require periodic dressing and shaping (truing), which can be done dynamically, to maintain the cutting and dimensional integrity.

7.2.4.3 *Subsurface Damage*—The most serious concern is not necessarily the surface finish (on the order of R_a= 0.2 to 0.4 μm) which is the result of the final machining steps. Instead, the subsurface damage is critically important although this damage is not readily observed or measured, and therefore, must be inferred as the result of the grinding history. More details of this aspect have been discussed in Ref. **(4)**. In all cases, the final grinding operation ("spark out") performed in the gage section must be along the longitudinal axis of the specimen to ensure that any residual grinding marks are parallel to the applied stress.

7.2.5 *Handling Precautions*—Care must be exercised in storing and handling of specimens to avoid the introduction of random and severe flaws, such as might occur if the specimens were allowed to impact or scratch each other. Specimens should be stored separately in cushioned containers to minimize the occurrence of these problems.

7.3 *Specimen Sampling and Number*—Samples of the material to provide test specimens must be taken from such locations so as to be representative of the billet or lot from which it was taken. Although each testing scenario will vary, generally, a minimum of 24 specimens is required for the purpose of completely determining the creep and creep rupture behavior across a significant temperature and stress range. Typically, six specimens are run at each temperature of interest over the entire range of applied stresses of interest. Initial tests are used to define the range of temperature where creep is the dominant deformation mechanism, and the remainder are used to acquire more precise creep and creep-time-to-failure data. Variations from this number are permitted as necessary to meet limitations on the amount of material or other mitigating factors. A smaller number of specimens is permissible in cases where the ranges of applied stress or temperature, or both, are more narrow.

8. Procedures

8.1 *General*:

8.1.1 *Specimen Dimensions*—Determine the thickness, diameter, and width of the gage section of each specimen to within 1 % of its absolute value. In order to avoid damage in a critical area, carefully make the measurement using a flat, anvil-type micrometre. Ball-tipped or sharp anvil micrometres are not recommended because they can cause localized cracking. Use the measured dimensions to calculate the force required to achieve the desired stress in the gage section.

8.1.2 *Determination of Gage Length*—Determine the gage length of the specimen by points of attachment of the extensometer system being used. It should be as close to the length of the uniform cross section of the specimen as possible within the temperature variations stated in 6.4.2. It can be determined by any suitable optical or contact extensometry method. A number of such systems are available commercially. Make calibrations according to the appropriate manufacturer's instructions and check periodically using independent means.

8.1.2.1 *Mounting Flags to the Specimen*:

(*1*) *Optical Method*—Attach two or more flags of dimensions suitable for the gage width and thickness chosen, to the specimen gage length. Fig. 1 shows typical flags used for the specimens shown in Fig. X1.2 of the Appendix. They can be made from the test material itself or sintered SiC. The depth of the flag (dimension *d* in Fig. 1) should be kept as small as possible.

(*2*) *Contacting Method*—Setting of the initial gage length for a contacting extensometer depends on the extension measurement method (capacitance-based or strain gage-based), and the manufacturer's procedures for setup must be followed. Position the extensometer probes with rounded knife-edge tips in contact with the specimen and hold in place with a light (0.1 to 1.0 N) contact force. A schematic of a contacting extensometer system is shown in Fig. 2. At elevated temperatures, oxidation at the probe/specimen interface minimizes slippage.

8.1.2.2 *Mounting the Specimen in the Furnace*—Mount specimens in the load train prior to heating the furnace. After the specimens are mounted in the load train, apply a small preload to maintain the load train alignment during subsequent heat-up to the test temperature. The preload should introduce a stress of no more than 5 MPa in the gage section. For specimens using contacting extensometry, make the extensometry settings prior to heating the furnace. The contacting probes may be left in contact with the specimen during heat-up or brought into contact with the specimen after it has reached the test temperature, depending on the testing setup.

8.1.2.3 *Heating to the Test Temperature*:

(*1*) *Specimens with Flags*—Specimens with flags may be heated to the test temperature in stages. The first stage, if required, takes the temperature to approximately 700 K to burn off the room temperature cement. The soak time at this temperature is about 1 h. The second stage takes the specimen to the test temperature at a rate of approximately 300 to 500 K/h, but may be as fast as 1000 K/h. The soak time at the test temperature is determined experimentally, and must be long enough to allow the entire system to reach thermal equilibrium. The total time for heating and soaking should be less than 24 h. State heating rates and soak times in the report.

(*2*) *Specimens Using Contacting Extensometers*—Specimens that utilize contact extensometry may be either heated from room temperature to the test temperature in a single stage and constant heating rate of up to 1000 K/h or may be heated from a preheat furnace temperature to the final test temperature. If the furnace is heated from room temperature to the test temperature, a soak time should be determined experimentally, and must be long enough to allow the entire system to reach thermal equilibrium. The total time for heating and soaking should be less than 24 h. State heating rates and soak times in the report.

8.1.2.4 *Use of Thermocouples*: