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Standard Guide for Use of Thermocouples in Creep and Stress-Rupture Testing to 1800°F (1000°C) in Air[1]

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INTRODUCTION

This guide provides basic information, options, and guidelines to enable the user to apply thermocouples, temperature measurement, and control equipment with sufficient accuracy to satisfy the temperature requirements for creep and stress-rupture testing of materials.

1. Scope

1.1 This guide covers the use of ANSI thermocouple Types K, N, R, and S for creep and stress-rupture testing at temperatures up to 1800°F (1000°C) in air at one atmosphere of pressure. It does not cover the use of sheathed thermocouples.

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *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:*[2]

E139 Test Methods for Conducting Creep, Creep-Rupture, and Stress-Rupture Tests of Metallic Materials
E207 Test Method for Thermal EMF Test of Single Thermoelement Materials by Comparison with a Reference Thermoelement of Similar EMF-Temperature Properties
E220 Test Method for Calibration of Thermocouples By Comparison Techniques
E230 Specification and Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples
E292 Test Methods for Conducting Time-for-Rupture Notch Tension Tests of Materials
E344 Terminology Relating to Thermometry and Hydrometry
E574 Specification for Duplex, Base Metal Thermocouple Wire With Glass Fiber or Silica Fiber Insulation
E1129/E1129M Specification for Thermocouple Connectors
E1684 Specification for Miniature Thermocouple Connectors

3. Terminology

3.1 *Definitions*—Unless otherwise indicated, the definitions given in Terminology E344 shall apply.

4. Classification

4.1 The following thermocouple types are identified in Tables E230:

4.1.1 *Type K*—Nickel—10 % chromium (+) versus nickel—5 % (aluminum, silicon) (−),

4.1.2 *Type N*—Nickel—14 % chromium, 1.5 % silicon (+) versus nickel—4.5 % silicon—0.1 % magnesium (−),

4.1.3 *Type R*—Platinum—13 % rhodium (+) versus platinum (−),

4.1.4 *Type S*—Platinum—10 % rhodium (+) versus platinum (−).

5. Summary of Guide

5.1 This guide will help the user to conduct a creep or stress-rupture test with the highest degree of temperature precision available. It provides information on the proper application of thermocouples that are used to measure and control the temperature of the test specimen. It also points out sources of error and suggests methods to eliminate them.

6. Significance and Use

6.1 This guide presents techniques on the use of thermocouples and associated equipment for measuring temperature in creep and stress-rupture testing in air at temperatures up to 1800°F (1000°C).

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[1] This guide is under the jurisdiction of ASTM Committee E28 on Mechanical Testing and is the direct responsibility of Subcommittee E28.04 on Uniaxial Testing.

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[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.

6.2 Since creep and stress-rupture properties are highly sensitive to temperature, users should make every effort practicable to make accurate temperature measurements and provide stable control of the test temperature. The goal of this guide is to provide users with good pyrometric practice and techniques for precise temperature control for creep and stress-rupture testing.

6.3 Techniques are given in this guide for maintaining a stable temperature throughout the period of test.

6.4 If the techniques of this guide are followed, the difference between "indicated"[3] temperature and "true"[4] temperature will be reduced to the lowest practical level.

7. Apparatus

7.1 Instrumentation may be individual instruments, a data acquisition system (multipoint recorders or digital type), a computer-based control system, or a combination of these devices. (**Warning**—Since each thermocouple is "grounded" by contacting the specimen, it is necessary that the instrumentation treat each thermocouple as isolated or "floating" from all other thermocouples. Neither leg should be connected to a common ground at the instrumentation end of the system. Also, equipment having a high common mode rejection ratio is necessary because of the proximity of strong electromagnetic fields from the heating elements of the furnace.)

7.2 *Temperature Measurement Instrumentation*—The measurement system should be able to resolve the thermocouple signal to ±0.1°F (0.05°C). The temperature indication should have no more than ±1.0°F (0.5°C) uncertainty for the purposes of this test. In addition, where specific corrections for the calibration of individual thermocouples or a thermocouple lot is required, the capability of the instrumentation system to accommodate these data shall be considered.

7.2.1 *Reference Junction Compensation:*

7.2.1.1 Thermocouples are usually calibrated to a 32°F (0°C) reference temperature. Unless an ice point reference is used, some means must be provided to compensate for the temperature where the thermoelectric circuit connects to the instrument (refer to MNL-12 on Reference Junctions[5]).

7.2.1.2 Reference junction compensation is usually performed within the instrumentation itself. Most devices or electronic data acquisition systems measure the temperature where the thermoelements connect to the input terminals and introduce a compensating emf to simulate the ice point.

7.2.1.3 The input connections shall be isothermal and shielded from sudden changes of temperature.

7.2.2 *Recalibration*—The accuracy of the temperature measurement equipment may be affected by component aging, environment, handling, or wear. Therefore, a periodic recalibration of the measuring instrumentation with a checking instrument is necessary. The checking instrument should be of higher accuracy than the measurement system, and to ensure conformity to national standards, it should be calibrated with a known primary test standard, traceable to the National Institute of Standards and Technology.

7.3 *Temperature Control Equipment Requirements*—A temperature controller or temperature control system should be selected on the basis of stability (variations of ±1°F (0.5°C) or less), and accuracy (uncertainty of ±1.5°F (0.7°C) or less). Generally, a control system with proportional band, automatic reset, and slow approach to final set point features should be used. When employing an automatic feedback control system, the tuning constants or control algorithm shall be optimized, not only to maintain the test specimen at the set point without excessive deviations, but to eliminate or limit the amount of overshoot upon initial heating.

NOTE 1—The same precautions regarding reference junction compensation in the control device apply as in 7.2.1.

7.3.1 *Configuration*—The control configuration may take one of several forms:

7.3.1.1 The center thermocouple is connected to a control loop that strives to maintain the temperature of the center of the reduced section at set point. The upper and lower thermocouples are used to measure the temperatures at the ends of the reduced section. Means shall be provided to adjust the heating power above and below the center to equalize the temperatures.

7.3.1.2 The bottom and top thermocouples may be connected to control loops that regulate the power to the upper and lower heaters independently. Thus, the end temperatures are maintained automatically. The center thermocouple is used only as a monitor.

7.3.2 *Control System Recalibration and Reliability*—The control system should be subjected to routine recalibration, as circumstances and type of equipment dictate. The checking procedure should include calibration of the controller and a sensitivity check. A calibration circuit, as shown in Appendix X1, should be employed.

7.4 *Heating Equipment*—Furnaces should be appropriately sized or adjusted relative to the workload and heat losses to provide a zone of uniform temperature across the specimen. Because creep and stress-rupture testing is usually done at constant temperature and with an unchanging furnace load, the main requirement is a well-insulated furnace, capable of achieving the desired temperatures. The top and bottom openings should be closed to limit convection losses, but the furnace should not be sealed airtight.

8. Hazards

8.1 The duration of a creep test ranges from a few hours to several hundred hours at elevated temperatures, at least partially unattended by operators. Such tests are normally ended before test specimen failure. Stress-rupture tests may operate at higher stresses, higher temperatures, and for shorter times than creep tests, but they normally continue until the specimen has achieved its required life or has failed.

8.2 The stability of the emf of the thermocouples and the rapid response of the control system to any changes of temperature over the period of the test are crucial to maintain the specimen within the allowable temperature band.

[3] As defined in Practice E139 and Test Methods E292.

[4] As defined in Practice E139.

[5] Manual on the Use of Thermocouples in Temperature Measurement, ASTM MNL-12.

8.3 *Thermocouple Requirements*—The requirements for thermocouples used for measurement are somewhat different from thermocouples used for control purposes (especially with automatic feedback control systems). Of course, both requirements may be met with one set of thermocouples that is judiciously chosen and placed.

8.3.1 *Measurement*—Thermocouples used for measurement are designed to represent the temperature of the specimen along its reduced section.[4] Since only two or three thermocouples are used, they shall be located at places on the specimen that represent the average temperature of their respective sections. This can be determined by a test program, where more than the usual number of thermocouples are mounted along the reduced section to establish the temperature profile.

8.3.2 *Control*—Thermocouples used for control are designed and placed to be sensitive to changes of or impending changes to the temperature of the specimen. Control thermocouple wire should be as thin as possible. However, the wire should not be so thin that oxidation or strain would cause emf errors or failure during the test period.

NOTE 2—Locating a control thermocouple next to the heater as a means to limit fluctuations of temperature is not advisable. A controller with a wide proportional band and automatic reset is capable of compensating for the thermal lag of most furnace designs.

9. Thermocouples

9.1 *Basic Information*—Information on basic thermocouple characteristics and performance is available from ASTM publications such as MNL-12.[5] Stability of the emf over the period of test is the most crucial requirement for a control thermocouple, whereas accuracy of the measurement thermocouple is paramount for successful correlation of test results.

9.1.1 Factors affecting thermocouple selection are: atmosphere, temperature of exposure, duration of testing, and response time. These factors should be considered to determine type (K, N, R, or S), wire size, insulation, and installation methods.

9.2 *Precautions*—The emf output of a thermocouple is affected by inhomogeneities in the region of a temperature gradient. Inhomogeneities are produced by cold work, contamination, or metallurgical changes produced by temperature itself. Therefore, thermocouples should be handled carefully without unnecessary stretching, bending, or twisting the wires. Bending around small radii should be avoided entirely, especially where the wires may lie in a temperature gradient. Where necessary, a minimum amount of bending may be performed carefully around bend radii at least 20 times the diameter of the wire.

9.3 *Types K and N Thermocouples:*

9.3.1 *Suitability*—Types K and N thermocouples are useful for creep and rupture testing because they provide a relatively high emf and are relatively stable over the low and middle temperatures of the testing range.

9.3.2 *Limitations*—Conventional Type K, and to a lesser extent, Type N thermocouples undergo emf drift as the result of metallurgical changes during use. Therefore, that portion of wire that has been exposed to temperatures above 500°F (260°C) or 1600°F (870°C) for Types K and N thermocouples, respectively, should be discarded after one use.

TABLE 1 Thermocouple and Extension Wire Tolerances and Calibration Uncertainties

Type	Temperature Range	Tolerance		Typical Calibration Uncertainty[A]
		Standard[B]	Special	
Thermocouples, °F				
K, N	32 to 2000	4 or 0.75 %	2 or 0.4 %	1.8 to 2.2
S, R	32 to 2000	2.7 or 0.25 %	1 or 0.1 %	1.1 to 1.2
Extension Wire, °F[C]				
KX, NX	32 to 400	4	[D]	0.5 to 2
RX, SX	32 to 400	9	[D]	2 to 4
Thermocouples, °C				
K, N	0 to 1100	2.2 or 0.75 %	1.1 or 0.4 %	1 to 1.2
R, S	0 to 1100	1.5 or 0.25 %	0.6 or 0.1 %	0.6 to 0.7
Extension Wire, °C[C]				
KX, NX	0 to + 200	2	[D]	0.3 to 1
RX, SX	0 to + 200	5	[D]	1 to 2

[A] Calibration uncertainty in an actual test depends on number of test points, media, and reference standard used during the calibration (see Test Method E220).

[B] Tolerances and uncertainties are plus or minus the indicated values expressed in degrees Fahrenheit or degrees Celsius or as a percentage of the value of the measured temperature, whichever is greater (see Tables 1, 2, and 3 in Specification E230).

[C] Worst case, where the temperature of the transition point differs from the instrument's reference junction by 360°F (200°C).

[D] No special tolerance limits have been established, but materials with tolerances closer than the standard limits may be available.

9.3.3 *Assessment*—Type K or N thermocouples should be reused only after their suitability for a particular test program is proven by a body of test data. Stability tests are advised, using Type R or S thermocouples as references.

9.4 *Types R and S Thermocouples:*

9.4.1 *Suitability*—Types R and S are highly resistant to oxidation and are therefore stable for these tests at the higher temperatures of the range. They provide the highest reproducibility and repeatability of the several thermocouple types but are initially more costly. Because they do not deteriorate during normal use, it is possible to reuse them. When Types R and S wires are no longer suitable for service, they still retain a significant portion of their initial cost in salvage value.

9.4.2 *Limitations*—The limiting factor for reuse of Type R or S thermocouples is error introduced by strain or contamination. Wires of Types R and S are mechanically weaker than Types K and N, so they must be adequately supported to avoid straining them. Sufficient slack should be provided so that the wires do not strain or tear when the specimen elongates. Contamination of the wire[6] may be caused by oils, grease, or other chemicals, and from metallic vapors from the test specimen during heating.

[6] "Creep and Rupture Test Pyrometry," Charles R. Wilks, ASTM Special Technical Publication No. 178—Panel Discussion on Pyrometric Practices, Presented at the 58th Annual Meeting, June 30, 1955.

TABLE 2 Relative Stability of Thermocouples

Thermocouple Type		
R or S	N	K
Most stable	Intermediate	Least stable

9.4.3 *Assessment*—A Type R or S thermocouple should not be used indefinitely. One way to determine the end of its useful life is to place a new thermocouple from the same lot beside the old one and compare outputs. Replace the older thermocouple when the change exceeds the uncertainty indicated on Table 1. Special precaution is advised to avoid errors arising from the use of compensating extension wires. See Table 1.

9.5 *Stability*—Studies have been made to determine emf stability with time of the thermocouple types covered by this guide. Results of those investigations show that the Types R and S thermocouples may be expected to have greater stability under favorable conditions than either Type K or N. Other work indicates Type N is more stable than Type K.[7] Relative stabilities are summarized in Table 2.

9.6 *Calibration:*

9.6.1 *Method*—Calibration of thermocouples should be done on representative lot samples with Test Method E220. Recalibration of used thermocouples that may contain inhomogeneities is not recommended unless it can be performed in place and under actual conditions of use.

9.6.2 *Thermocouple and Extension Wire Tolerance Grades*—Thermocouples are commercially available in two tolerance grades, as given in Specification E230. In lieu of ordering special or better tolerances, it is advisable that the calibration of a specific lot of wire be determined. This should be done by actual calibration of representative samples to determine the specific emf temperature relation of each wire lot. An estimate of the uncertainties contributed by wire selection and testing is included in Table 1. Extension wire Types KX and NX are commercially available in two tolerances, but only the standard tolerances of Specification E230 are stated here. Compensating extension wires for Types R and S are available in several tolerances but only the standard tolerances of Specification E230 for Types RX and SX are included here.

9.6.2.1 The values of thermocouple tolerances given here represent initial values, applicable only to new thermocouple material in the as-manufactured condition. The emf of a thermocouple during use may change.

9.6.2.2 The stability of special emf tolerance wire is not necessarily greater than standard emf tolerance wire. Special emf tolerance is attained by mating selected pairs of the two wires. Emf stability is an inherent characteristic of the thermocouple alloys relative to the environment in which they are used.

9.6.2.3 Because of an expected emf shift after initial heating to high temperature and the development of inhomogeneities in the wire, it is not recommended that individual Type K or Type N thermocouples be calibrated and subsequently used to make critical temperature measurements. It is preferable to calibrate representative thermocouples from sections of each spool of wire instead and make the measurements with unheated thermocouples made from those spools.

9.6.3 *Other Tolerances and Compensations*— Requesting tighter tolerances than special grade from the thermocouple provider is possible but not advisable. It may incur higher costs and difficulties with availability. A more practical approach would be to calibrate samples from the lot, and apply corrections (manually or automatically) to the instrumentation to match the emf-temperature curve of the lot over the range of test.

9.7 *Insulation:*

9.7.1 Any degradation or decomposition of the electrical insulation between the thermocouple wires can introduce errors in the indicated temperature. Pre-insulated wire is acceptable if it is used within the temperature constraints given in Specification E574. The basic kinds of materials for this use are fiberglass, fibrous silica, or fibrous ceramic. Impregnants should be avoided, since they may contaminate the thermocouple or test specimen. (**Warning—**Silica or insulations containing boron are not suitable for use with platinum or its alloys at high temperatures.)

9.7.2 *Ceramic Sleeves and Beads*—Hard-fired unglazed ceramic insulators are preferred to insulate the thermocouple at the higher temperatures. Mullite (alumina + silica) insulators may be used with either Type K or Type N thermoelements. Type R and S thermoelements require high purity insulators (96 % minimum alumina).

9.7.3 *Reuse of Insulators*—Insulators may become soiled or contaminated by careless or inappropriate storage or handling. Because soiled or contaminated insulators can produce conduction, only clean insulators (new or used) should be used for this work.

9.8 *Thermocouple Junction Fabrication:*

9.8.1 *Wire Preparation*—Cut the thermoelement wires to length. Strip the wires with tools that do not nick the surface. Avoid excessive stretching and cold working the wire. Discard ends damaged by gripping. Wear latex, plastic, or clean cloth gloves to avoid contaminating the wires with dirty hands.

9.8.2 *Welded Junctions*—The diameter of the weld bead should not exceed four times the diameter of a single thermoelement wire.

9.8.2.1 Fusion of Types K and N by the tungsten-inert-gas (TIG) process is preferred. Processes based on electric arc or other non-contact fusion principles as well as electric resistance welding may be used. A clean, sound weld junction shall be produced. Fluxes, filler materials, or mercury processes should not be used.

9.8.2.2 For Type R or S junctions, an oxygen-gas torch with an excess-oxygen flame is suitable, however, this process is not recommended for Types K or N materials.

9.8.3 *Mechanical Junctions*—Compression fittings are acceptable, provided their durability for the intended use is demonstrated. When using compression-type junctions, it is convenient to insert the thermoelement ends into the fitting from opposite directions so that each end extends sufficiently

[7] "The Nicrosil versus Nisil Thermocouple: Properties and Thermoelectric Reference Data," NBS Monograph 161, April 1978, U.S. Dept. of Commerce, NIST, Gaithersburg, MD. Also see RR:E20-1001, available from ASTM.