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Standard Guide for Use of Thermocouples in Elevated-Temperature Mechanical Testing[1]

This standard is issued under the fixed designation E633; 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.

INTRODUCTION

This guide provides basic information, options, and guidelines to enable the user to apply thermocouples, temperature-measuring systems, and temperature-control systems with sufficient accuracy to satisfy the temperature requirements for elevated-temperature mechanical testing of materials.

1. Scope*

1.1 This guide covers the use of ANSI thermocouple Types listed in Specification E230/E230M for elevated-temperature mechanical testing at temperatures typical in testing of metals.

NOTE 1—Typical conditions for mechanical testing are specified temperatures up to 1800 °F (982 °C) in air at one atmosphere of pressure.

1.1.1 This guide focuses on most commonly used base metal and noble metal ANSI type K, N, R, and S thermocouples.

1.1.2 Other ANSI thermocouple types that are listed in Specification E230/E230M may be used if the specific precautions and maximum temperatures for their use are followed.

1.1.3 It does not cover the use of sheathed thermocouples.

1.2 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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]

E6 Terminology Relating to Methods of Mechanical Testing
E21 Test Methods for Elevated Temperature Tension Tests of Metallic Materials
E111 Test Method for Young's Modulus, Tangent Modulus, and Chord Modulus
E139 Test Methods for Conducting Creep, Creep-Rupture, and Stress-Rupture Tests of Metallic Materials
E209 Practice for Compression Tests of Metallic Materials at Elevated Temperatures with Conventional or Rapid Heating Rates and Strain Rates
E220 Test Method for Calibration of Thermocouples By Comparison Techniques
E230/E230M Specification for Temperature-Electromotive Force (emf) Tables for Standardized Thermocouples
E292 Test Methods for Conducting Time-for-Rupture Notch Tension Tests of Materials
E328 Test Methods for Stress Relaxation for Materials and Structures
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
E2448 Test Method for Determining the Superplastic Properties of Metallic Sheet Materials

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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.

***A Summary of Changes section appears at the end of this standard**

3. Terminology

3.1 Definitions of terms common to mechanical testing that appear in Terminology E6 apply to this standard, including accuracy, calibration, indicated temperature, metrological traceability, reduced parallel section, temperature-measuring system, specified temperature, and verification.

3.2 Some specific definitions of terms relating to thermometry and hydrometry that appear in Terminology E344 apply to this guide. These terms are compensating extension wire, emf, extension wire, ice point, measuring junction, reference junction, reference temperature, sheathed thermocouple, thermocouple, thermocouple emf, thermoelectric properties, thermoelement, and true value of a temperature.

4. Classification

4.1 The following thermocouple types are identified in Tables E230/E230M:

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 an elevated-temperature mechanical property 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 elevated-temperature mechanical testing under typical conditions. Test durations range from minutes for tension, compression, Young's modulus, and superplastic property tests to thousands of hours for creep, stress-rupture, creep rupture, notch tension, and stress-relaxation tests.

6.2 Since elevated-temperature mechanical 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 elevated-temperature mechanical 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 temperature and true temperature, as used in Test Methods E21, E111, E139, E292, E328, E2448, and Practice E209 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.

7.1.1 Since each thermocouple is "grounded" by contacting the specimen, the instrumentation shall treat each thermocouple as isolated or "floating" from all other thermocouples. Neither the extension wire nor thermoelement shall be connected to a common ground at the instrumentation end of the temperature-measuring system.

7.1.2 Instrumentation shall have a high common mode rejection ratio because of the proximity of strong electromagnetic fields from the heating elements of the furnace.

7.2 *Temperature Measurement System*—The resolution of the temperature-measuring system should be ±0.1 °F (0.05 °C) or better. The uncertainty of the indicated temperature should be no more than ±1.0 °F (0.5 °C) In addition, where specific corrections for the calibration of individual thermocouples or a thermocouple lot are required, the capability of the temperature-measuring system to accommodate these data shall be considered.

7.2.1 *Reference Junction Compensation:*

7.2.1.1 Unless an ice point reference junction is used, provide some means to compensate for the temperature where the thermoelectric circuit connects to the instrumentation end of the temperature-measuring system.

NOTE 2—Thermocouples are usually calibrated to a 32 °F (0 °C) reference temperature. Refer to MNL-12 on Reference Junctions.[3]

NOTE 3—Reference junction compensation is usually performed within the instrumentation of the temperature-measuring system itself. Most temperature-measuring systems measure the temperature where the thermoelements or extension wires connect to the input terminals and introduce a compensating emf to simulate the ice point reference junction.

7.2.1.2 The input terminals shall be isothermal and shielded from sudden changes of temperature.

7.2.2 *Calibration and Verification*—The temperature-measuring system shall be periodically verified so that the temperature errors are consistent with the specified requirements for the accuracy of the indicated temperature.

7.2.2.1 The accuracy of the verification instrument shall be higher than that of the temperature-measuring system, and its calibration should exhibit metrological traceability to the International System of Units (SI).

7.2.2.2 The period between calibration or verification of the temperature-measuring system should be based on age, use, and the drift exhibited between calibrations.

NOTE 4—The accuracy of the temperature-measuring system can be affected by component aging, environment, handling, or wear. Frequent verifications can help determine the period between calibrations.

7.2.2.3 Calibrations and verifications should include multiple points and extend over the intended range of use.

[3] Manual on the Use of Thermocouples in Temperature Measurement, Fourth Edition, 1993. Sponsored by ASTM International Committee E20 on Temperature Measurement. ASTM International MNL-12–4th. DOI: 10.1520/MNL12–4TH-EB.

7.3 *Temperature-Control System 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 temperature-control system with proportional band, automatic reset, and slow approach to final set point 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.

7.3.1 Follow the requirements of 7.2.1 regarding reference junction compensation in the temperature-control system.

7.3.2 *Configuration*—The configuration of the temperature-control system shall be capable of meeting the indicated temperature variation and configuration requirements of the test method that cites E633.

NOTE 5— Many different configurations are used successfully. For example, in one configuration, the center thermocouple is connected to a control loop that strives to maintain the temperature of the center of the reduced parallel section at the set point. The upper and lower thermocouples measure the indicated temperatures at the ends of the reduced parallel section. Independent means are provided to adjust the heating power above and below the center to equalize the temperatures.

In another configuration, the upper and lower thermocouples are connected to control loops that regulate the power to the upper and lower heaters of a two-zone furnace independently. The center thermocouple measures the indicated temperature. Furnaces with three independently controlled zones are also used successfully.

7.3.3 *Temperature-Control System Calibration, Verification, and Reliability*—The temperature-control system should be routinely verified, as circumstances and type of equipment dictate. The verification procedure should include verification, and if necessary, calibration of the temperature-control system and a sensitivity check. A calibration circuit, as shown in Appendix X1, should be employed.

7.4 *Heating Equipment*—Furnaces should be well insulated and appropriately sized or adjusted relative to the workload and heat losses to provide a zone of uniform temperature across the specimen. The top and bottom openings should be closed to limit convection losses, but the furnace should not be sealed airtight.

8. Thermocouples

8.1 The stability of the thermocouple emf and the rapid response of the temperature-control system to any changes of temperature over the period of the test shall be suitable to maintain the specimen within the specified temperature band.

8.2 *Thermocouple Requirements:*

8.2.1 *Measurement*—Thermocouples used for measurement shall be capable of representing the temperature of the specimen along its reduced parallel section. 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.

NOTE 6—A profile test program, where more than the usual number of thermocouples are mounted along the reduced parallel section can help to establish the temperature profile along the test specimen.

8.2.2 *Control*—Thermocouples used for control shall be selected and placed to be sensitive to changes of or impending changes to the temperature of the specimen.

8.2.2.1 Control thermocouple thermoelements should be as small in diameter as possible. However, the diameter should not be so small that oxidation or strain causes thermocouple emf errors or failure during the test period.

8.2.2.2 A control thermocouple should not be located next to the heater as a means to limit fluctuations of temperature.

NOTE 7— A temperature-control system with a wide proportional band and automatic reset is capable of compensating for the thermal lag of most furnace designs

8.2.3 If permitted by the test method standard that cites E633, the measurement and control requirements may be met with one set of thermocouples that is judiciously chosen and placed.

8.3 *Basic Information*—Atmosphere, temperature of exposure, duration of testing, and response time shall be considered to determine type (K, N, R, or S), thermoelement size, insulation, and installation methods.

NOTE 8—Information on basic thermocouple characteristics and performance is available from ASTM publications such as MNL-12.[3] 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.

8.4 *Precautions*—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 bending is necessary, the bend radius should be at least 20 times the diameter of the wire.

NOTE 9—The thermocouple emf 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.

8.5 *Types K and N Thermocouples:*

8.5.1 *Suitability:*

NOTE 10—Types K and N thermocouples are useful for elevated-temperature mechanical testing because they provide a relatively high emf and are relatively stable over the low and middle temperatures of the testing range.

8.5.2 *Limitations*—Any portion of Type K or Type N thermoelement 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.

NOTE 11—Conventional Type K, and to a lesser extent, Type N thermocouples undergo emf drift as the result of metallurgical changes during use.

8.5.3 *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, using Type R or S thermocouples as references should be conducted to establish their suitability.

8.6 *Types R and S Thermocouples:*

8.6.1 *Suitability*—Because they do not deteriorate during normal use, Type R and Type S thermocouples may be reused.

NOTE 12—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. 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.

8.6.1.1 The calibration of Type R or Type S thermocouples that have been exposed to or in contact with chromium-containing alloys should be verified before they are reused.

NOTE 13—Type R and Type S thermocouples degrade significantly at temperatures above 1650 °F (900 °C) when attached to chromium-containing alloys because the chromium vaporizes and contaminates the thermocouple. The net result at 2200 °F (1200 °C) can be an error of up to 14 °F (8 °C).

8.6.2 *Limitations*—Type R and Type S thermocouples shall 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. Care should be exercised to avoid contaminating the thermocouple[4] by oils, grease, or other chemicals, and from metallic vapors from the test specimen during heating.

NOTE 14—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.

8.6.3 *Assessment*—A Type R or S thermocouple should not be used indefinitely.

NOTE 15—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.

8.6.3.1 Special precaution should be taken to avoid errors arising from the use of compensating extension wires. See Table 1.

8.7 *Stability*—For measurements where thermocouple emf stability is critical, Type K and Type N thermocouples should not be used.

NOTE 16—Studies of the thermocouple emf stability show that the thermocouple emf of Types R and S thermocouples is more stable under favorable conditions than the thermocouple emf of either Type K or N. Other work indicates Type N thermocouple emf is more stable than Type K thermocouple emf.[5] Relative stabilities are summarized in Table 2.

8.8 *Calibration:*

8.8.1 *Method*—Calibration of thermocouples should be done on representative lot samples with Test Method E220. Used thermocouples that can contain inhomogeneities should not be recalibrated unless the calibration can be performed in place and under actual conditions of use.

8.8.2 *Thermocouple and Extension Wire Tolerance Grades*—Instead of ordering special or better tolerances, the calibration of a specific lot of wire should be determined by calibrating representative samples to determine the specific thermocouple emf temperature relation of each wire lot.

NOTE 17—Thermocouples are commercially available in two tolerance grades, as given in Specification E230/E230M. An estimate of the uncertainties contributed by wire selection and testing are included in Table 1. Extension wire Types KX and NX are commercially available in two tolerances, but only the standard tolerances of Specification E230/E230M are stated here. Compensating extension wires for Types R and S are available in several tolerances, but only the standard tolerances of Specification E230/E230M for Types RX and SX are included here.

NOTE 18—The values of thermocouple tolerances given in Table 2 represent initial values, applicable only to new thermocouple material in the as-manufactured condition. The thermocouple emf of a thermocouple during use can change.

NOTE 19—The thermocouple emf stability of special tolerance thermocouples is not necessarily greater than standard tolerance thermocouple. Special tolerance is attained by mating selected pairs of the two thermoelements. Thermocouple emf stability is an inherent characteristic of the thermocouple alloys relative to the environment in which they are used.

8.8.2.1 Because of an expected thermocouple emf shift after initial heating to high temperature and the development of inhomogeneities in the wire, individual Type K or Type N thermocouples should not be calibrated and subsequently used to make critical temperature measurements. Instead representative thermocouples from sections of each lot of wire should

TABLE 1 Thermocouple and Extension Wire Tolerances and Calibration Uncertainties

Thermocouples, °F				
Type	Temperature Range	Tolerance		Typical Calibration Uncertainty[A]
		Standard[B]	Special	
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
Compensating Extension Wire				
RX, SX	32 to 400	9	[D]	2 to 4
Thermocouples, °C				
Type	Temperature Range	Tolerance		Typical Calibration Uncertainty[A]
		Standard[B]	Special	
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
Compensating Extension Wire				
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/E230M).
[C] Worst case, where the temperature of the transition point is 400 °F (differs from the instrument's reference junction by 360 °F (200 °C)).
[D] No special tolerance limits have been established, but it is possible that materials with tolerances closer than the standard limits are available.

TABLE 2 Relative Stability of Thermocouples

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

[4] "*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 http://dx.doi.org/10.1520/STP178-EB.

[5] "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.

be calibrated, and unheated thermocouples made from those lots should be used to make measurements.

8.8.3 *Other Tolerances and Compensations*—Thermocouples with tolerances tighter than special tolerance grade may be prepared by calibrating samples from the lot, and applying corrections (manually or automatically) to the temperature-measuring system to match the thermocouple emf-temperature curve of the lot over the range of test.

NOTE 20—Requesting tighter tolerances than special grade from the thermocouple provider is possible but can incur higher costs and difficulties with availability.

8.9 *Insulation:*

8.9.1 Pre-insulated wire may be used if it is used within the temperature constraints given in Specification E574.

NOTE 21—Any degradation or decomposition of the electrical insulation between the thermocouple wires can introduce errors in the indicated temperature. The basic kinds of materials for this use are fiberglass, fibrous silica, or fibrous ceramic.

8.9.1.1 Impregnants should be avoided, because they can contaminate the thermocouple or test specimen.

8.9.1.2 Silica or insulations containing boron shall not be used with platinum or its alloys at high temperatures.

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

8.9.3 *Reuse of Insulators*—Only clean insulators (new or used) should be used.

NOTE 22—Insulators can become soiled or contaminated by careless or inappropriate storage or handling. Soiled or contaminated insulators can produce conduction.

8.10 *Thermocouple Measuring Junction Fabrication:*

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

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

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

8.10.2.2 Type R or S measuring junctions should be made with an oxygen-gas torch with an excess-oxygen flame, but this process should not be used for Types K or N thermocouples.

8.10.3 *Mechanical Measuring Junctions*—Compression fittings may be used, provided their durability for the intended use is demonstrated. When using compression fittings, the thermoelement ends should be inserted into the fitting from opposite directions so that each end extends sufficiently beyond it to facilitate attaching the thermocouple to the specimen using the thermocouple wires themselves (see 8.12.3).

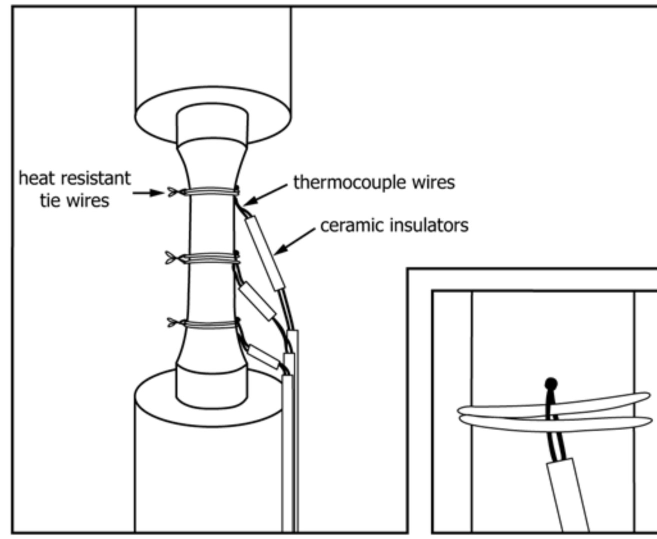

FIG. 1 Installation with Welded Measuring Junctions (Inset: Attachment Detail)

8.11 *Installation of Thermocouples*—Locate the measurement thermocouples as required by the specific test method or indicated by the profile test if no other requirements exist (see 8.2.1 and Note 6). Choose the exact location of the control thermocouples depending upon the specimen configuration and the heating system.

8.12 *Attachment of Thermocouples:*

8.12.1 *Precautions*—Attach the thermocouples to the specimen in such a way that the thermocouple measuring junction will attain thermal equilibrium with the test specimen at the attachment point. The attachment should not affect the specimen in any way. The thermocouple shall be held tightly against the specimen with as good a thermal contact as possible.

NOTE 23— One way to check attachment is to prepare two thermocouples that are cut adjacent from the same lot of wires. Spot weld measuring junction of one thermocouple to the surface, or bury the measuring junction in a tight-fitting hole close to the measuring junction of the attached thermocouple in an expendable sample. Compare the indicated temperatures of the two at operating temperatures.

8.12.2 *Welded Junctions*——The measuring junction shall be held against the specimen by wrapping a length of heat-resistant alloy wire of approximately the same diameter directly across the wires immediately behind the junction and around the specimen, as illustrated in Fig. 1. To minimize contamination of Type R or S thermocouples, employ the technique shown in Fig. 2.

NOTE 24—By wrapping a conductor across the thermoelements, the weld bead becomes shorted. This is not necessarily poor practice; it merely shifts the measuring junction from the weld bead to the points of conduction. Also, the third conductor will not affect the emf, if the path between the positive and negative thermoelements is isothermal. In that case, the resultant thermocouple emf will be unaffected by the third metal.

8.12.3 *Mechanical Measuring Junctions*—Attach crimped-type compression junctions by wrapping the free ends of the thermoelements around the specimen from opposite directions