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TESTING OF CERAMIC INSULATOR COMPACTION
IN SEALED THERMOCOUPLES

K. R. Con

OAK RIDGE NATIONAL LABORATORY

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TESTING OF CERAMIC INSULATOR COMPACTION
IN SHEATHED THERMOCOUPLES

K. R. Carr

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OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee
operated by
UNION CARBIDE CORPORATION
for the
U.S. ATOMIC ENERGY COMMISSION

Carr

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TESTING OF CERAMIC INSULATOR COMPACTION IN SHEATHED THERMOCOUPLES

K. R. Carr

ABSTRACT

Two improved methods (direct measurement and liquid absorption) were developed for measuring ceramic insulator compaction in sheathed thermocouples. These methods were developed after tests of two methods (dye penetration and helium flow) widely used in the industry showed them to be not suitable. Thermocouple materials from 15 manufacturers were tested to evaluate all four methods.

Although both the direct measurement method and the liquid absorption method, with kerosene as the absorbed liquid, are a satisfactory means of measuring the compaction of magnesium oxide insulation in sheathed thermocouples as small as 1/16 in. OD, the liquid absorption method is superior. Its accuracy is $\sim \pm 2\%$ maximum theoretical density (100% MTD is 3.58 g/cm^3 , the density of a single MgO crystal). The accuracy of the direct measurement method is also $\sim \pm 2\%$ MTD, but the method cannot be applied in every case because sometimes it is difficult to separate the insulator from the sheath and wires.

1. INTRODUCTION

Two improved methods (direct measurement and liquid absorption) were developed to measure the compaction of ceramic insulation in sheathed thermocouples. The methods provide compaction measurements required to determine if there is correlation between compaction and several measures of thermocouple performance, such as durability under thermal cycling (alternately heating to 1200°F and cooling to 800°F), time response (speed at which a thermocouple responds to a step change in temperature), and calibration drift during aging. Prior to this work, discussions with thermocouple manufacturers and a search of the literature had indicated that two compaction test methods (dye penetration and helium flow) were available. After we had evaluated both methods and concluded that neither was suitable for this purpose, we proceeded to develop the two new methods.

The study of thermocouple compaction is part of a broad program, the Fast Flux Test Facility Coolant Thermocouple Development Program, being conducted at ORNL and other AEC national laboratories. One responsibility of ORNL in this program is

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to develop a specification for thermocouples to be used in Liquid Metal Fast Breeder Reactors (LMFBR). The compaction test to be included in the specification must be inexpensive, reliable, accurate, and suitable for use by the thermocouple manufacturers. The overall objective of this work, then, was to determine if the required test method was available and, if not, to develop a suitable method.

The two new methods developed were the result of contributions by several associates in the program at ORNL to develop a specification for thermocouples for LMFBR applications. Their assistance is gratefully acknowledged. Many helpful suggestions were received from M. B. Herskovitz,¹ W. W. Johnston, Jr.,¹ C. A. Mossman,¹ and L. H. Thacker.¹ The statistical analysis was by F. H. Clark¹ and D. G. Gosslee.² Much of the laboratory work was done by K. W. Brownell³ and W. P. Kinsler.¹

2. DESCRIPTION OF TEST METHODS

There are several factors that must be considered in the application of the methods for measurement of insulator compaction, one of which is to measure the void (10 to 30% of the total volume) between the insulator particles in a total volume as small as 0.002 in.³. In this section, the limitations and possible problem areas are defined for the two previously available methods (i.e., dye penetration method and helium flow method) and for the two new methods (i.e., direct measurement method and liquid absorption method). A comparison of these four methods is summarized in Table 1, and detailed test procedures are given in the Appendix, Sect. 6.1.

2.1 Dye Penetration

In the dye penetration method, the rate of travel of a dye solution by capillarity through the insulator is deemed to be a measure of insulator compaction. A test sample ~1/2 in. long is prepared so that both sample ends are open to expose the insulator. The sample is positioned vertically, with the lower end placed in contact with a fluorescent dye solution. The upper end of the sample is observed periodically under ultraviolet light for the appearance of dye. When the dye is first detected or when it completely covers the upper end of the sample (depending on which option the test procedure calls for), the time required for the dye to traverse the sample is recorded. The insulation compaction is read from an experimentally determined graph of penetration time vs compaction.

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Table i. Comparison of Test Methods

Item of Comparison	Method			
	Dye Penetrant	Helium Flow	Direct Measurement	Liquid Absorption
Sample length, in.	0.5	1	1	1
Destruction of sample tested	Yes	No	Yes	Yes
Test measurements	Length of sample. Rate of dye travel through sample by capillarity.	Length of sample. Rate of helium flow through insulator.	Weight and dimensions of sample sheath, wires, and insulator.	Weight of sample. Sheath ID and wires OD. Weight and specific gravity of liquid absorbed by capillarity.
Test accuracy	$\sim \pm 10\%$ MTD	$\sim \pm 10\%$ MTD	$\pm 2\%$ MTD	$\pm 2\%$ MTD
Principal disadvantages	Must derive standard graph of penetration time vs compaction. Test results likely to be a function of particle size and arrangement. Test accuracy is poor.	Must derive standard graph of helium flow rate vs compaction. Test results likely to be a function of particle size and arrangement. Test accuracy is poor.	Must remove insulator from sample, which is difficult in some cases for 1/16 in. OD and smaller material.	Must initially verify complete filling of void space by capillary liquid.
Principal advantages	After standard graph has been derived, several hundred samples can be tested at one time.	None.	Insulator compaction is measured directly rather than inferred. Test yields accurate measurement of compaction.	After void filling is verified, insulator compaction is measured directly rather than inferred. Test yields accurate measurement of compaction.
Effect of nonuniformly compacted insulator on test results	Data will vary unpredictably.	Data will vary unpredictably.	Test yields an average compaction value regardless of nonuniformity.	Test yields an average compaction value regardless of nonuniformity.
Analyst time required per sample	Depends on number of samples and how often samples are checked for dye penetration.	1 hr/sample in addition to time required to derive standard graph.	1 hr.	1 hr.
Origin of test	Unknown, but test is widely known in the industry.	Unknown, but test is widely known in the industry.	Developed at ORNL.	Developed at ORNL.

One way by which such a graph of dye penetration time as a function of insulator compaction can be prepared is by use of a material balance.⁴ Before assembly of a thermocouple insulator, wires, and sheath, the amount of each component is measured. After assembly and reduction of the sheath diameter by swaging or drawing, the amount of each component in the finished thermocouple is known from the material balance, since the starting amount of each component must equal the finished amount. The insulator compaction for a given thermocouple can be determined from the finished thermocouple dimensions and the amount of each component in that thermocouple. To prepare a graph requires fabrication of several thermocouples with compaction values over the entire compaction range. Usually, thermocouple manufacturers do not furnish such information to a thermocouple purchaser who needs the information to measure the insulator compaction of finished thermocouples.

Several factors must be considered in applying the dye penetration method. Since the compaction is inferred from the penetration time, the time must be a function only of the insulator compaction. The precision of the test is dependent on the care taken in preparing the sample ends to avoid any glaze or obstruction that might retard the capillary flow. The attractive forces that control the capillarity can be kept uniform from test to test by rigid specification and standardization of the dye solution, since the thermocouple materials are also rigidly specified. A factor which might prove detrimental to the method, however, is variation of the size of insulator particles and their arrangement in the finished thermocouple. Since the thermocouple manufacturers obtain their insulator material from several suppliers and since the insulators are compacted in the assembled thermocouples by two different methods (swaging and drawing), the dye penetration time might not be a function only of insulator density. For example, a longitudinal void in the insulator could be present and drastically alter the penetration time.

An important consideration is the accuracy of the graph of penetration time as a function of compaction, since errors in the graph will be reflected in the test results.

2.2 Helium Flow

The helium flow method correlates insulator compaction with the rate of longitudinal flow of helium through a thermocouple sample. Helium at ~100 psi flows through a 1-in.-long sample. The gas passed through the sample is collected, and the time necessary to accumulate a prescribed amount of helium is taken as a measure of compaction. A graph of helium flow rate vs compaction shows the insulator compaction value corresponding to the helium flow rate through the test sample. The graph is derived by use of the material balance method.

⁴E. L. Babbe, Increasing Thermocouple Reliability for In-Pile Experiments, NAA-SR-10511 (April 20, 1965).

This method has several possible sources of error, such as (1) deformed or obstructed sample ends which could hamper the flow of helium, (2) moisture introduced into the insulator which could impede the flow of helium, and (3) variations in the helium pressure. These can be overcome by proper laboratory procedures. The most important consideration is that the helium flow rate must be a function not only of the volume of the voids but also of their distribution. An infinite number of flow configurations is possible for the same degree of insulator compaction. Since thermocouple manufacturers obtain their insulator material from different suppliers and since two different methods (swaging and drawing) are used during fabrication to reduce the diameter of the thermocouple, the insulator particle size and arrangement are expected to vary among thermocouples having the same compaction value but obtained from different manufacturers. Another source of error is an inaccurate graph of helium flow rate vs compaction.

2.3 Direct Measurement

The direct measurement method requires mechanical separation of the thermocouple components and measurement of the insulator weight and bulk volume. The bulk volume of the insulator can also be determined before separation of the thermocouple components. A direct calculation of insulator weight per unit volume yields the insulator compaction value. Thermocouple samples ~1 in. long are tested.

The direct measurement test offers several advantages. The uncertainty associated with correlating an observed characteristic of the thermocouple material, such as dye penetration time, with insulator compaction is not a factor in the direct measurement method. The accuracy and precision of this method are dependent only on how well the physical dimensions and weight of the thermocouple components can be determined. Once error limits are established, this method can be applied with confidence, since a change in a manufacturer's fabrication process or source of materials should not be reflected in the insulator compaction test results.

An important factor in the direct procedure is the technique of separating the thermocouple components. Even though the dimensions and weights of the components can be accurately measured after separation, these measurements will be of little use if appreciable weight loss occurs during separation of the components.

2.4 Liquid Absorption

The liquid absorption method requires measurement of the volumes of the insulator and the void. The insulator volume is calculated from the sheath ID, wire OD, and sheath length. The void volume is determined from the weight of liquid absorbed by the ceramic insulator owing to its capillarity. The specific gravity of the liquid must be known to relate the weight of absorbed liquid to the void volume.

There are several sources of error in this method. The most important consideration and one not easily determined, is the extent to which the liquid fills the insulator interstitial spaces. The probable error of measurement of the insulator volume and the specific gravity and weight of liquid absorbed by the sample can be determined easily. Any condition such as glaze at the sample ends which would retard the capillary action and restrict the liquid absorption should be avoided to minimize such errors.

This method is useful in setting an upper limit for the insulator compaction of a test sample within the error limits of the method. An upper limit for the actual insulator compaction value can be established, because, if the liquid does not penetrate fully into the interstitial space, the derived insulator compaction value will be higher than the actual insulator compaction value. If the maximum probable error from all sources other than incomplete penetration of the liquid is $\pm 2\%$ and the test yields a compaction value of 80% of maximum theoretical density (MTD), the insulator compaction is indicated to be no greater than 82% MTD.

3. EXPERIMENTAL PROCEDURE

To evaluate the four methods described in Sect. 2, the insulator compaction was determined for 60 thermocouples purchased from 15 manufacturers. These were type K thermocouples sheathed with 304 stainless steel and insulated with MgO. The sheath was 1/16-in. OD on 30 units and 1/8-in. OD on the other 30.

The insulator compaction of each of the 60 assemblies was measured using both the direct measurement method and the liquid absorption method (with both kerosene and water as the absorbed liquids). Also, the compaction of 10 thermocouples was measured using the liquid absorption method, with water containing a wetting agent as the absorption liquid. Since the dye penetration and helium flow methods are only inferential, not every assembly was tested by these methods; twelve 1/16-in. OD and twelve 1/8-in. OD thermocouples were tested by each of these two methods. The procedures are outlined in the Appendix, Sect. 6.1. All samples were taken within a 10-in. length from the original thermocouple to allow a comparison of results from the various tests on similar thermocouple material.

To provide additional information on the repeatability and accuracy of the liquid (kerosene) absorption and direct measurement methods, 20 samples were taken from each of two additional thermocouples, one with low-compaction and one with high-compaction material. The samples were taken from adjacent positions along each assembly length and numbered sequentially. The odd-numbered samples were tested by the direct measurement method, and the even-numbered were tested by the liquid absorption method. These 40 samples were tested along with 11 samples from other thermocouples. The samples were coded so that the operator taking the data was unaware of which samples were taken from the same thermocouples.

4. DISCUSSION OF RESULTS

The data were analyzed to evaluate the four methods and to determine the compaction characteristics of the thermocouples tested. The results show that the two new methods developed (direct measurement and liquid absorption) are more accurate and reliable than the dye penetration and helium flow methods. Since the thermocouples tested were the products of 15 manufacturers, the data were analyzed to determine their distribution and to correlate insulator compaction with the manufacturer's sheath reduction method (swage or draw) and sheath finished size.

4.1 Direct Measurement and Liquid Absorption Methods

The results of the direct measurement and liquid absorption methods on 60 thermocouples are listed in Tables 2 and 3, and plotted in Figs. 1-4. The direct measurement results are based on a 100% MTD value of 3.58 g/cm^3 , the density of a single MgO crystal.

When kerosene was the absorbed liquid for the liquid absorption method, the data agreed more closely with data from the direct measurement method than when water was the absorbed liquid. In general, higher values of insulator compaction were obtained with water as the absorbed liquid than with kerosene (Figs. 1-4). A possible explanation is that water did not penetrate the insulator interstitial spaces as completely as did kerosene. The addition of a wetting agent (Triton X-100) to pure water appears to have increased the penetration of the water (Tables 2 and 3). A result of incomplete penetration is that the experimental value of compaction will be higher than the true value.

Compaction data were obtained from twenty 1-in.-long samples taken from each of two thermocouples. The values for the direct measurement method ranged from 75.3 to 76.1% MTD for the low-compaction thermocouple and for the kerosene absorption method ranged from 75.1 to 76.6% MTD (Fig. 5). For the high-compaction thermocouple, values for the direct measurement and kerosene absorption methods were 88.6 to 89.8% MTD and 89.0 to 90.5% MTD, respectively (Fig. 6). These results indicate that the compaction in both thermocouples was uniform in the 20-in.-long test sections and that the analysis of errors given in the Appendix, Sect. 6.2, was confirmed experimentally. A statistical correlation of these data is given in the Appendix, Sect. 6.3.

The close agreement among the data from the two methods confirms that these two methods are accurate and reliable for measurement of insulator compaction in finished thermocouples. An analysis of errors (Appendix, Sect. 6.2) predicts the limits of error when these two methods are applied to 1/16- and 1/8-in. OD material. The analysis of the kerosene absorption method is based on the assumption that the insulator void is completely filled with kerosene. On this basis, the predicted maximum disagreement

Table 2. Insulator Compaction for 1/16-in. OD Thermocouples Determined by Direct Measurement and Liquid Absorption Methods

Thermocouple Number ^a	Insulator Compaction (% MTD) Determined by		
	Direct Measurement ^b	Kerosene Absorption	Water Absorption ^c
A001	74.1	73.3	79.1 (76.1)
A003	73.3	74.4	77.3
B001	79.0	78.0	79.0
B003	73.5	73.5	76.4
C001	73.3	72.4	77.6
C003	72.9	72.2	77.4
D001	72.7	73.1	77.2
D003	71.5	72.7	76.6 (75.7)
E001	d	90.3	95.1
E003	d	90.2	96.4
F001	d	89.8	92.0
F003	d	89.2	90.7
G001	d	72.8	77.9
G003	79.0	74.6	77.9
H001	74.9	75.8	79.0
H003	76.2	74.5	85.3 (85.1)
I001	74.7	75.9	77.4
I003	74.4	74.1	77.5
J001	72.8	73.7	80.2 (78.9)
J003	73.6	73.0	80.0 (80.4)
K001	d	91.0	92.7
K003	d	91.8	93.8
L001	73.7	71.8	73.5
L003	74.5	74.7	76.2
M001	d	70.9	73.5
M003	d	71.5	80.4
N001	74.6	74.2	78.5
N003	74.4	73.1	79.6 (75.6)
P001	d	89.8	93.3
P003	d	90.2	94.3

^aAll thermocouples having the same first letter were from the same manufacturer.

^bThe direct measurement calculations were done on the basis that 100% MTD is 3.58 g/cm³, the density of a single MgO crystal.

^cValues in parentheses were obtained with a wetting agent (Triton X-100) in the water, 1 part per 1000 parts of water.

^dUnable to separate MgO from sheath and wires without appreciable metal loss.

Table 3. Insulator Compaction for 1/8-in. OD Thermocouples Determined by Direct Measurement and Liquid Absorption Methods

Thermocouple Number ^a	Insulator Compaction (% MTD) Determined by		
	Direct Measurement ^b	Kerosene Absorption	Water Absorption ^c
A005	75.9	76.4	77.5
A012	74.9	76.6	75.0
B005	74.6	74.9	77.9
B007	73.6	75.1	75.7
C005	76.9	77.2	79.4
C007	75.6	76.6	80.4
D005	74.8	77.7	79.1
D007	74.3	78.0	79.6
E005	86.6	88.1	90.0
E007	88.8	88.1	90.5
F005	73.8	75.4	77.6
F007	72.3	76.4	76.4
G005	77.2	74.0	76.6
G007	78.3	73.4	76.0
H005	74.5	76.6	78.4
H007	75.8	76.5	78.1
I005	72.6	71.8	75.2
I007	72.8	73.5	76.6
J005	75.4	73.6	76.4
J007	70.3	71.8	76.2 (75.2)
K005	94.4 ^d	90.9	92.5 ^c
K007	92.4 ^d	90.5	92.2
L005	74.5	74.7	77.3
L007	74.4	74.3	77.2
M005	72.7	71.9	78.0 (73.7)
M007	71.6	72.5	79.9 (74.5)
N005	72.3	72.4	80.0 (72.1)
N007	72.7	72.4	74.6
P005	86.6	88.6	89.5
P007	86.3	87.1	87.9

^aAll thermocouples having the same first letter were from the same manufacturer.

^bThe direct measurement calculations were done on the basis that 100% MTD is 3.58 g/cm³, the density of a single MgO crystal.

^cValues in parentheses were obtained with a wetting agent (Triton X-100) in the water, 1 part per 1000 parts of water.

^dThe insulator was removed by drilling, because the air-abrasive tool would not remove the insulator.

Fig. 1. Comparison of Kerosene Absorption and Direct Measurement Test Values for Insulator Compaction of 1/16 in. OD Thermocouples.

Fig. 2. Comparison of Kerosene Absorption and Direct Measurement Test Values for Insulator Compaction of 1/8 in. $\odot$ Thermocouples.

Fig. 3. Comparison of Water Absorption and Direct Measurement Test Values for Insulator Compaction of 1/16 in. OD Thermocouple.

Fig. 4. Comparison of Water Absorption and Direct Measurement Test Values for Insulator Compaction of 1/8 in. OD Thermocouples.

Fig. 5. Results of Tests Made on a Low Compaction Thermocouple to Confirm the Direct and Kerosene Methods of Measuring Insulator Compaction.

Fig. 6. Results of Tests Made on a High Compaction Thermocouple to Confirm the Direct and Kerosene Methods of Measuring Insulator Compaction.

in data taken by the two methods on identical material is 10.7% MTD for 1/16-in. OD thermocouples and 4.7% MTD for 1/8-in. OD thermocouples. The data from 1/16-in. OD thermocouples are well within the 10.7% MTD limit in every case. The data from 1/8-in. OD thermocouples are generally well within the 4.7% MTD limit, but two samples had a disagreement slightly greater than the limit, 4.9% and 5.2% MTD. Based on the experimental results and an analysis of the errors, therefore, the direct measurement and kerosene absorption methods are expected to consistently provide compaction measurements with an accuracy of $\pm 2\%$ MTD.

The direct measurement method cannot be applied in some instances because of the difficulty in separating the insulator from the sheath and wires (Tables 2 and 3). The kerosene absorption method can be applied in every case, is as accurate as the direct measurement method, and does not have any characteristics which would prevent its inclusion in a workable thermocouple specification. The kerosene absorption method, therefore, is the superior of the two developed compaction measurement methods.

4.2 Dye Penetration Method

The dye penetration method seems to be an unreliable method of measuring insulator compaction. Note for example, in Fig. 7 samples H003 and K001 with compaction values near the opposite extremes of the compaction range have comparable dye penetration times. Other similar examples are evident in both Figs. 7 and 8. The best straight line fit to the data in these two figures will deviate from at least one data point by $\sim 9\%$ MTD, which is close to half the entire compaction range. Since the material balance procedure that has been followed by some thermocouple users to obtain a standard graph of dye penetration time vs compaction contributes additional error, the accuracy of results obtained with the dye penetration method according to the previous standard practice is probably no better than $\pm 10\%$.

A specified minimum dye penetration time for a given sample length has been used sometimes by thermocouple purchasers as an acceptance criterion, who reasoned that if the dye penetration time of a sample exceeds a specified minimum time, insulator compaction exceeds a specified minimum value. However, a procedure based on this criterion would require rejection of material having acceptable insulator compaction along with material not acceptable.

For example, if a purchase specification for 1/16-in. OD material required a minimum compaction of 90% MTD and if the acceptance test specified the dye penetration time of 500 min., then in Fig. 7 two (K001 and K003) of four thermocouples having acceptable compaction would be rejected along with the low-compaction thermocouples.

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Fig. 7. Dye Penetration Time vs Insulator Compaction of 1/16 in. OD Thermocouples.

ORNL-DWG 69-11103

Fig. 8. Dye Penetration Time vs Insulator Compaction of 1/8 in. OD Thermocouples.

ORNL-DWG 69-11102

Fig. 10. Helium Flow vs Insulator Compaction of 1/8 in. OD Thermocouples.

ORNL-DWG 69-11105

Fig. 11. Distribution of 1/16 in. OD Thermocouple Insulator Compaction Measured by the Kerosene Absorption Method.

ORNL-DWG 69-11107

Fig. 12. Distribution of 1/8 in. OD Thermocouple Insulator Compaction Measured by the Kerosene Absorption Method.

ORNL-DWG 69-11106

Fig. 13. Distribution of 1/16 and 1/8 in. OD Thermocouple Insulator Compaction Measured by the Kerosene Absorption Method.

Table 4. Compaction vs Sheath Reduction Method and Sheath Diameter

Manu- facturer Code	Sheath Reduction Method ^a	Insulator Compaction Determined by Kerosene Absorption Method					
		Low Compaction (70.0-78.0%)			High Compaction (86.1-92.0%)		
		1/16-in. OD	1/8-in. OD	Difference ^b	1/16-in. OD	1/8-in. OD	Difference ^b
A	D	73.3	76.4	+3.1	--	--	--
A	D	74.4	76.6	+2.2	--	--	--
B	D	78.0	74.9	-3.1	--	--	--
B	D	73.5	75.1	+1.6	--	--	--
C	D	72.4	77.2	+4.8	--	--	--
C	D	72.2	76.6	+4.4	--	--	--
D	S and D	73.1	77.7	+4.6	--	--	--
D	S and D	72.7	78.0	+5.3	--	--	--
E	S	--	--	--	90.3	88.1	-2.2
E	S	--	--	--	90.2	88.1	-2.1
F	S	--	75.4	--	89.8	--	-14.4
F	S	--	76.4	--	89.2	--	-12.8
G	D	72.8	74.0	+1.2	--	--	--
G	D	74.6	73.4	-1.2	--	--	--
H	S	75.8	76.6	+0.8	--	--	--
H	S	74.5	76.5	+1.0	--	--	--
I	D	75.9	71.8	-4.1	--	--	--
I	D	74.1	73.5	-0.6	--	--	--
J	D	73.7	73.5	-0.2	--	--	--
J	D	73.0	71.8	-1.2	--	--	--
K	S	--	--	--	91.0	90.9	-0.1
K	S	--	--	--	91.8	90.5	-1.3
L	S and D	71.8	74.7	+2.9	--	--	--
L	S and D	74.7	74.3	-0.6	--	--	--
M	S and D	70.9	71.9	+1.0	--	--	--
M	S and D	71.5	72.8	+1.3	--	--	--
N	D	74.2	72.4	-1.8	--	--	--
N	D	73.1	72.4	-0.7	--	--	--
P	S	--	--	--	89.8	88.6	-1.2
P	S	--	--	--	90.2	87.1	-3.1

^aD, drawing; S, swaging.^b% MTD for 1/8-in. OD material minus % MTD for 1/16-in. OD material.

All four thermocouples produced by three manufacturers showed a compaction greater than 78% MTD; these manufacturers ordinarily use the swage method of sheath reduction (manufacturers E, K, and P). The 1/16-in. OD thermocouples from manufacturer F, who also usually uses swaging, showed high compaction results, but his 1/8-in. OD thermocouples showed low compaction results. All four thermocouples from manufacturer H, who also uses swaging, showed low compaction results. Three manufacturers with material in the low compaction range (manufacturers D, L, and M) use both the swage and draw methods.

Although these data indicate that drawn material with high insulator compaction is not being supplied by the industry, it does not indicate that high compaction material cannot be produced by drawing. However, there might be limitations in the drawing process that could eliminate drawing as a means of achieving high compaction. Subsequent testing is planned in the LMFBR thermocouple specification program to determine the effect of insulator compaction on thermocouple performance. The optimum insulator compaction, which might be a low value, may then be specified.

4.4.3 Manufacturers' Compaction as a Function of Sheath Diameter

Table 4 shows the difference between the insulator compaction values for the two sheath sizes supplied by each manufacturer. These values, tabulated as the compaction value for the 1/8-in. OD thermocouple minus the value for the 1/16-in. OD thermocouple, range from +5.3% to -4.1%, except in one case the values are -11.4% and -12.8% for manufacturer F.

5. CONCLUSIONS

The mathematical analysis of probable errors and the experimental results both indicate that the direct measurement and kerosene absorption methods are satisfactory means of measuring magnesium oxide insulator compaction in sheathed thermocouples as small as 1/16-in. OD. The agreement of the experimental results from the developed methods shows that kerosene will fill virtually all of the insulator interstitial space by capillarity. The possibility of incomplete filling was the only probable source of error in the absorption method that could not be estimated prior to the experimental work. Since these two methods require two very different procedures, the excellent experimental agreement and the confirming theoretical analysis of errors have proved the newly developed methods. The kerosene absorption method, which is superior to the direct measurement method, has an accuracy of $\sim \pm 2\%$ MTD and is suitable for inclusion in a workable thermocouple specification.

The dye penetration and helium flow methods seem to be unreliable; each method yielded comparable results for thermocouple material having widely different values of insulator compaction.

The compaction values of all the material tested ranged from 70 to 92% MTD. The compaction of ~78% of the thermocouples ranged from 70 to 78% MTD, and of the remaining 22% ranged from 86.1 to 92.0% MTD. No thermocouples had compaction in the range from 78.1 to 86.0% MTD. All thermocouples with compaction greater than 78% MTD were products of manufacturers who normally reduce the sheath diameter by swaging.

6. APPENDIX

6.1 Test Procedures

6.1.1 Dye Penetration Method

The procedure for the dye penetration method is as follows:

1. Cut the samples at least 9/16 in. long either by hand filing or power-driven abrasive such as an aluminum oxide wheel. If a power-driven abrasive is used, the rate of cutting must not cause excessive heat that would result in a glaze at the finished sample ends.
2. Dress the sample ends manually with a mill-smooth file and finish manually with No. 3/0 emery polishing paper. The finished sample ends should be perpendicular to the sample length, free of any burrs or glaze, and 0.500 ± 0.010 in. long.
3. Position each sample vertically with the lower end inserted about 1/8 in. into Zyglo-Pentrex fluorescent dye penetrant, type ZL-22, a product of Magnaflux Corp. Record the time at this point.
4. At 10- to 15-min. intervals, view the upper end of each sample under ultraviolet light. When the upper end is covered with dye, record the time and terminate the test on that sample. Record the dye penetration time as the elapsed time between the two recorded times.

6.1.2 Helium Flow Method

The procedure for the helium flow method is as follows:

1. Prepare the samples as described in steps 1 and 2 of Sect. 6.1.1 except that the finished sample lengths should be 1.000 ± 0.010 in.
2. Clamp a flexible tubing from a helium supply at 100 psi pressure to one end of the sample.

3. Connect the other end of the sample to a burette by a short length of rigid tubing. Determine proper size of burette for a given sample by making a trial run. The full-scale graduated length of the burette is chosen so that slight errors do not become large percentage errors in reading the burette scale or in measuring time.
4. Position the burette vertically and place the low-pressure end downward in water so that the water level in the burette is above the zero (uppermost) graduation.
5. To begin a test, allow the 100-psi helium to abruptly start flowing through the sample. The water level in the burette will decrease as the helium flows through the sample. Start measuring time when the water level reaches the zero graduation.
6. Reposition the outer container of water with respect to the burette so that the starting and ending pressures inside the gas-filled region of the burette will be approximately equal. This precaution will ensure that no error is introduced in the helium volume measurement due to tubing expansion. The ending pressure will be several inches of water higher than the starting pressure if this step is not followed.
7. Stop measuring time when the water level inside the burette reaches the full-scale (lowest) graduation on the burette. The elapsed time and the full-scale volume of the burette indicate the helium rate of flow through the sample.

6.1.3 Liquid Absorption Method

The procedure for the liquid absorption method is as follows:

1. Cut samples ~1-in. as described in steps 1 and 2 of Sect. 6.1.1.
2. Measure the average inside diameter of the sheath and the average outside diameter of the wire with a microscope having a reticle with maximum graduations of 0.001 in., scale length of at least 0.100 in., and minimum magnification of 60X. A Cole-Parmer Instrument Co., catalog No. 3890 microscope or equivalent will be suitable.
3. Weigh the sample with an analytical balance to an accuracy of ± 0.1 mg.
4. Measure the sample length with a micrometer to an accuracy of ± 0.001 in.
5. Position the sample vertically and insert the bottom end at least 1/8 in. below the surface of the liquid to be absorbed. Allow the sample to absorb liquid by capillary action for at least 24 hr. Kerosene meeting Federal Specification VV-K-211d is the recommended liquid, although other kerosene may be acceptable. The specific gravity of the liquid must be known to within 1%.

6. Remove the sample from the liquid and wipe dry the outside surface of the sample, including the ends. Within 2 min after removing the sample from the liquid, weigh the sample to an accuracy of ± 0.1 mg.
7. Determine the % MTD of the MgO insulator (for thermocouples having two conductors):

$$\% \text{ MTD} = 100 \left[1 - \frac{0.000061W}{SL(0.785A^2 - 1.57B^2)} \right] , \quad (1)$$

where

- A = sheath average inside diameter, in.,
 B = wire average outside diameter, in.,
 L = length of sample, in.,
 S = specific gravity of the absorbed liquid,
 W = weight of wet sample - dry sample, mg.

6.1.4 Direct Measurement Test

The procedure for the direct measurement method is as follows:

1. Cut samples ~1 in. as described in steps 1 and 2 of Sect. 6.1.1.
2. Measure the outside diameter of the sheath and the length of the sample with a micrometer to an accuracy of ± 0.001 in. or better.
3. Weigh the prepared sample with an analytical balance to an accuracy of ± 0.1 mg or better.
4. Remove the insulator from the sample with an air abrasive tool (air-driven abrasive particles). A suitable air abrasive tool is the S. S. White, industrial abrasive unit, model C, used with S. S. White, "Airbrasive" powder No. 2.
5. Weigh the wires together and the sheath to an accuracy of ± 0.1 mg or better.
6. Determine the % MTD of the MgO insulator:

$$\% \text{ MTD} = \frac{100(A - B)}{46,060C^2D - (E/F + G/H)3,580} , \quad (2)$$

where

- A = weight of entire sample, mg,
- B = weight of sheath and wires, mg,
- C = outside diameter of sheath, in.,
- D = length of sample, in.,
- E = weight of sheath, mg,
- F = density of sheath, mg/cm³,
- G = weight of wires, mg,
- H = average density of wires, mg/cm³.

6.2 Mathematical Derivations and Analysis of Errors

6.2.1 Liquid Absorption Method

The basic equation to be applied is

$$\% \text{ MTD} = \frac{100(\text{space occupied by the insulator} - \text{interstitial space in the insulator})}{\text{space occupied by insulator}} \quad (3)$$

If all the interstitial space is filled with a liquid by capillary movement, the equation is

$$\% \text{ MTD} = \frac{100(\text{space occupied by insulator} - \text{volume of liquid absorbed})}{\text{space occupied by insulator}} \quad (4)$$

$$= 100 \left(1 - \frac{\text{volume of liquid absorbed}}{\text{space occupied by insulator}} \right) \quad (5)$$

The volume of liquid absorbed is calculated by dividing the weight (W) of liquid absorbed by the specific gravity (S) of the liquid.

The space occupied by the insulator for a sample having two conductors is

$$\left[\frac{\pi (\text{sheath ID})^2}{4} - 2 \frac{\pi (\text{wire OD})^2}{4} \right] \left[\text{sample length} \right] .$$

Let

A = inside diameter of sheath,

B = outside diameter of wire,

L = length of sample,

and substitute these symbols in Eq. (5):

$$\% \text{ MTD} = 100 \left[1 - \frac{W/S}{L(0.785A^2 - 1.57B^2)} \right] \quad (6)$$

$$= 100 \left[1 - \frac{W}{SL(0.785A^2 - 1.57B^2)} \right] \quad (7)$$

If A, B, and L are in inches and W is in milligrams, Eq. (7) is

$$\% \text{ MTD} = 100 \left[1 - \frac{0.000061W}{SL(0.785A^2 - 1.57B^2)} \right] \quad (8)$$

The maximum limits of error for the method can be estimated as follows:

1. Decide the limits of error for the values determined for A, B, S, L, and W in Eq. (8).
2. To estimate a lower limit for the compaction of a given sample, insert into Eq. (8) the maximum values of W and B and the minimum values of S, L, and A within the limits decided in step 1.
3. To estimate an upper limit for the compaction of a given sample, insert into Eq. (8) the minimum values of W and B and the maximum values of S, L, and A within the limits decided in step 1.

The estimated maximum limits of error for typical low- and high-compaction thermocouples when tested in accordance with the procedure of Sect. 6.1.3 are listed in Table 5 and the procedure given above. The assumed specific gravity of the absorbed liquid was 0.805. The measurements will usually be more accurate than shown in the table, since the tabulation lists the worst cases which can occur.

Table 5. Estimated Limits of Error for the Kerosene Absorption Method

Sheath OD (in.)	Actual Compaction (% MTD)	Estimated Limits of Error	
		(-% MTD)	(+% MTD)
1/16	73.5	3.0	2.5
1/16	92.3	1.4	1.1
1/8	72.3	1.5	0.3
1/8	90.6	0.6	0.6

6.2.2 Direct Measurement Method

The basic equation for determining the percentage of maximum theoretical density of the insulator is

$$\% \text{ MTD} = \frac{100(\text{actual insulator density})}{\text{maximum theoretical insulator density}} \quad (9)$$

$$= \frac{100 \left(\frac{\text{actual insulator weight}}{\text{actual insulator volume}} \right)}{\text{maximum theoretical insulator density}} \quad (10)$$

Let

A = total weight of sample, mg,

B = weight of sheath and wires, mg,

C = outside diameter of sheath, in.,

D = length of sample, in.,

E = weight of sheath, mg,

F = density of sheath, mg/cm³,

G = weight of wires, mg,

H = density (averaged if applicable) of wires, mg/cm³,

J = maximum theoretical density of insulator, i.e., the density of a single crystal (mg/cm³)

Then the actual insulator weight of the insulator (mg) is $A - B$. The actual volume of the insulator (in.^3) is the volume of the initial sample minus the volume of the sheath and wires:

$$0.785C^2D - (E/F + G/H)(1 \text{ in.}^3/16.39 \text{ cm}^3) .$$

Therefore

$$\% \text{ MTD} = \frac{100 \left[\frac{A - B}{0.785C^2D - (E/F + G/H)(1/16.39)} \right]}{J} , \quad (11)$$

$$\begin{aligned} &= \frac{100(A - B)}{[0.785C^2D(\text{in.}^3) - (E/F + G/H)(1/16.39)(\text{in.}^3)] (J/\text{cm}^3)(16.39 \text{ cm}^3/\text{in.}^3)} , \\ &= \frac{100(A - B)}{[12.866C^2D - (E/F + G/H)J]} . \end{aligned} \quad (12)$$

For MgO insulation, $J = 3,580 \text{ mg/cm}^3$:

$$\% \text{ MTD} = \frac{100(A - B)}{46,060C^2D - (E/F + G/H)3,580} . \quad (13)$$

The maximum possible limits of error in the test may be estimated by this procedure:

1. Decide the limits of error for the values determined for A , C , D , E , F , G , and H in Eq. (11).
2. To estimate a lower limit for compaction of a given sample, insert into Eq. (12) the maximum values of C , D , F , H , and J and the minimum values of A , E , and G within the limits decided in step 1.
3. To estimate an upper limit for compaction of a given sample, insert into Eq. (12) the minimum values of C , D , F , H , and J and the maximum values of A , E , and G within the limits decided in step 1.

The estimated maximum limits of error for typical low- and high-compaction thermocouples when tested in accordance with the procedure of Sect. 6.1.4 are listed in Table 6. The direct measurement test will usually be more accurate than indicated

in the tabulation, since the worst cases which can occur are shown. The estimated errors include those due to a weight loss of 1.5 mg from the sheath and 0.5 mg from the wires which occurred when the insulation was removed from the sheath.

Table 6. Estimated Limits of Error for the Direct Measurement Method

Sheath OD (in.)	Actual Compaction (% MTD)	Estimated Limits of Error	
		(-% MTD)	(+% MTD)
1/16	73.5	2.7	7.7
1/8	70.2	1.7	3.0
1/8	85.4	2.2	3.3
1/8	92.3	3.0	4.1

6.3 Statistical Analysis

Statistical correlation of the developed methods has been accomplished with the method known as reverse application of the line of linear regression. In general, the method is valid if the variance of the independent variable is relatively small, the variance of the dependent variable has a Gaussian distribution, and the samples are randomly selected. There are four steps in the statistical procedure:

1. The data are plotted with values of the dependent variable on the y axis and of the independent variable on the x axis.
2. A regression line is determined. This is a straight line fitted to the data which, when used for estimating x values from given y values, will minimize errors of estimation.
3. The correlation coefficient r is calculated. This coefficient indicates the usefulness of the regression line for estimating x values from given y values. The range of r is from -1 to $+1$, with estimation accuracy increasing as r approaches either of the limiting values. An $r = 0$ indicates that the regression line has no value for estimating purposes; i.e., the x and y variables are uncorrelated.
4. The degree of correlation of the data is tested by making use of the calculated value of r and the t distribution to generate curves about the regression line that define confidence regions. For any given y value, the curves will show within stated confidence limits a range of values for x.

This procedure was followed in plotting the data shown in Figs. 5 and 6. The variance of data for the direct measurement method was assumed to be negligible, and these data were plotted as the independent variable (Fig. 14).⁶ The calculated correlation coefficient was 0.997 at the 99% confidence level. As illustrated in Fig. 14, a kerosene absorption test value of 80% MTD indicates that the direct measurement test value for the same material is 79.5% MTD, with limits of 78 and 81% MTD at the 99% confidence level.

⁶Data obtained by the kerosene absorption method were plotted as the dependent variable.

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Fig. 14. Statistical Correlation of Developed Test Procedures.