NATIONAL BUREAU OF STANDARDS REPORT

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THE CURRENT STATUS OF THERMAL CONDUCTIVITY REFERENCE STANDARDS AT THE NATIONAL BUREAU OF STANDARDS

Complementary Report

March 1964

by

H. E. Robinson and D. R. Flynn

to the

Bureau of Ships
Department of the Navy
Washington, D. C.

NBS

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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* NBS Group, Joint Institute for Laboratory Astrophysics at the University of Colorado.

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NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT **NBS REPORT**

1006-20-10461 3 March 1964 8300

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INDEX NO. S-R007 12 01, Task No. 800
(BuShips 1700S-716-62). Code 2781

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FORWARD

This report is an unmodified version of a paper given by the authors at the 1963 Thermal Conductivity Conference sponsored by Oak Ridge National Laboratory and held at Gatlinburg, Tennessee, on 16-18 October 1963. It is being made available as an NBS Report in the belief that the contents may be of general interest.

In this report, thermal conductivity data are presented which have not been formally published. All such data are subject to review, editorially and otherwise, prior to formal publication, and their preliminary status should be recognized. The contents of this report may not be referenced.

THE CURRENT STATUS OF THERMAL CONDUCTIVITY REFERENCE STANDARDS AT THE NATIONAL BUREAU OF STANDARDS

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ABSTRACT

To provide thermal conductivity reference standards to cover the wide range of conductivities exhibited by materials means that a number of different reference materials are needed, each occupying a niche or range of conductivities more or less peculiar to a particular kind of material. Six niches are suggested. Current work at the Heat Transfer Section of the National Bureau of Standards, aiming at development of reference standards in several of these niches, is discussed. Experimental data are given in regard to several materials under consideration.

A summary of information on needs for reference standards, obtained from replies to the questionnaire circulated at Ottawa, is appended.

INTRODUCTION

In the wide spectrum of values of thermal conductivity exhibited by materials, which ranges at room temperatures from under 0.1 to over 5000 mW/cm deg, and which may be of interest at temperatures ranging from the cryogenic to over 2000°C, depending on the material, it is evident that to provide suitable thermal conductivity reference standards, several reference standards are needed, each suitable for its niche. At least six general niches seem discernible, each being more or less the domain of a particular kind of material.

Table 1 gives a breakdown along these lines, with possible reference materials for each niche and kind of material. The materials marked with an asterisk are those which are being used or investigated as reference materials at the National Bureau of Standards.

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* Chief, and Physicist, respectively.

TABLE 1.

Niche	k at 0°C, mW/cm deg	Kind of material	Possible reference material
1	0.2 - 0.5	Thermal insulation	Fibrous glass*
2	1 - 5	Organic solid	Rubber,* plastic
3	5 - 20	Amorphous inorganic solid	Pyrex,* fused silica
4	20 - 100	Polycrystalline solid	Pyroceram*
5	100 - 500	Alloyed metals	Inconel 702,* stainless steel
6	500 - 5000	Pure metal	Iron, platinum, tungsten

The steps which appear to be essential in developing a reference standard include: (a) choosing a material in each niche which appears satisfactory for use over an adequately wide temperature range; (b) estimating whether its probable uniformity and reproducibility of conductivity are ascertainable by means of other properties, or whether its use as a standard requires setting up a uniform stock or making individual measurements on each reference specimen; (c) determining if the stability of conductivity of the material is significantly affected by age or by environmental or temperature experience, and, if so, if adequate stability can be attained by a suitable pretreatment (e.g., a heat treatment). After these steps are satisfactorily taken, the determination of the thermal conductivity with minimum uncertainty follows. Even after successful completion of all of these steps, it may be necessary to recommend precautions on the care of the reference material, such as, "Do not pound with hammers," or "Do not anoint with oil," or "Do not heat in a sulfurous atmosphere."

The current status of the work in our group at the National Bureau of Standards in respect to the asterisked items of the table is presented below. In addition, a summarization of the information from twelve returns of the questionnaire on reference standards circulated at the Ottawa meeting is included as an Appendix.

THERMAL INSULATORS AND ORGANIC SOLIDS

For a number of years, we have furnished, on a fee cost basis, samples of insulating materials, of thickness from 1 in. downward, and 8 in. or more square. The samples consist of two like pieces of a material, the average conductivity of which is determined by a test in a guarded hot-plate apparatus (ASTM C 177). The tests currently cover a limited temperature range (mean temperatures typically are 0, 25, and 50°C); there is now an increasing demand for higher temperatures, up to about 500°C, which currently we are not able to satisfy, partly because of the problem of a suitable material.

The material most used is one we keep in stock--a dense, semi-rigid resin-bonded fiber-glass insulating board--from which stock are taken the samples to be measured and furnished. The material is very little hygroscopic, has a conductivity of about 0.33 mW/cm deg with a positive temperature coefficient $[(1/k)\ dk/dt = 0.0035$ per deg C$]$ and is slightly compressible so that it should be used at nearly the same thickness as that during our measurements. The uncertainty in our measured conductivities is believed to be about one percent; reproducibility in successive measurements on the same samples is well within 0.5 percent. These reference samples are fairly widely used--our traffic in them is from 30 to 40 samples per year--both to check absolute measurements in other apparatus and by other methods, and to calibrate comparative methods using some form of heat flow meter.

Occasionally requests are received for reference samples of greater conductivity than the fiber-glass material. Generally, these are satisfied by means of specially-purchased samples of gum rubber ($k \sim 1.6$ mW/cm deg) or of silicone rubber ($k \sim 3.5$). Necessary temperature limits on gum rubber (65°C max.) for stability, and the relatively high cost of silicone rubber, tend to restrict their wide use.

At present, it does not appear feasible to supply samples from a stock without individual measurement, if the uncertainty is to be limited to one percent. The variability among different samples, which may vary also in density, is such that the uncertainty would be on the order of three percent if the samples were not individually measured.

AMORPHOUS INORGANIC SOLIDS

We have been asked at times to measure the conductivity of rigid slabs of a material such as Pyrex for use as a reference at around room temperature. The only available equipment for the desired measurement was the guarded hot-plate apparatus, and it soon became evident that measuring the temperature drop through the rigid high conductance specimen was a major problem. Setting fine thermocouples in fine grooves in the surfaces was not allowable in some instances, and in any case this does not obviate the effect of variations of plate-to-specimen contact resistance which arise from imperfect flatness and contact of the specimen and the plates of the apparatus. For some rigid materials, dishing or warping of the specimen as a result of the temperature gradient set up in it may also impair uniform contact with the plates. The conductivity results obtained in the guarded hot-plate apparatus on 1-inch-thick Pyrex samples by the usual temperature-drop measuring techniques were sometimes definitely lower than available literature values, by as much as 20 percent.

A technique was developed which improved matters considerably, and which may be of interest here. A test is made using on each side of the hot plate a composite specimen consisting of the principal specimen sandwiched between two sheets of resilient material. In most applications so far, the resilient material has been soft gum rubber (30 to 45 Durometer floating stock Fed. Spec. MIL R-880A) 1/8-inch thick, which is soft enough to mold to minor irregularities of contacting surfaces, but is practically incompressible as an 8-inch square sheet under the compressive load applied. The thermal resistance of the composite specimens is determined using the temperature drop from the hot to the cold plate, which is measured by permanently-inset thermocouples. A second test is then made at the same mean temperature, and approximately the same plate temperatures, on only the two sheets of resilient material on each side, the principal specimens being removed. The thermal resistance of the sheets is calculated on the basis of the temperature drop indicated by the same plate thermocouples. The thermal resistance of the principal specimen is then calculated as the difference of that of the composite specimen and that of the sheets. The overall thicknesses of the composite and sheet specimens in the apparatus are also observed, and the deduced thicknesses of the specimen and sheets are compared with direct measurements; generally the agreement is excellent.

Use of this technique doubles the work of a measurement, but it has brought results into much closer concordance with literature values. Trials in hot-plate apparatus in three different laboratories, on the same 1-inch Pyrex specimens and rubber sheets, have yielded results in close agreement. The conductivities at 22°C obtained were 11.30, 11.24 and 11.45 mW/cm deg.

All of these measurements were made on the Pyrex slabs as received. Later it was found through refractive index measurements that the material had considerable unrelieved strain. Accordingly the slab specimens were annealed at 570°C and cooled very slowly. Upon testing the annealed specimens in the hot-plate apparatus, using the composite technique, the conductivity at 22°C was found to be 11.14 mW/cm deg as compared to the unannealed value of 11.45. It is felt that the decrease of 3 percent in conductivity is not all due to experimental error difference.

In the paper on a cut-bar thermal conductivity apparatus to be given later in this conference, D. R. Flynn reports results obtained therein on a piece of Pyrex (stated by the supplier to be from the same sheet as the slab specimens) which had been annealed as indicated above. The results, extrapolated to 22°C, yield a conductivity value of 11.94 mW/cm deg, about 8 percent higher than that obtained on the annealed slab specimens. Further work to resolve the difference, or to pursue the effect of strain or annealing, has not been feasible up to this time, although the matter needs investigation.

The usefulness of Pyrex as a reference material is unfortunately somewhat limited by its relatively low softening temperature, as well as its significantly increasing radiant transmittance above about 350°C. Other possible candidates for this conductivity niche may be fused silica, or a material like Vycor, provided that they can be adequately opacified to reduce radiant transmittance. Some of the people at Corning Glass Works are cooperating with us in exploring the feasibility of this approach.

POLYCRYSTALLINE SOLIDS

The conductivity niche from about 20 to 100 mW/cm deg is one of considerable present interest in connection with semi-conductor materials and refractory solids. At present, it appears that Pyroceram Code 9606 may be a suitable thermal conductivity reference material for this niche, for temperatures up to about 1000°C.

Figure 1 shows the thermal conductivity and resistivity results that have been obtained on a sample of Pyroceram 9606 using two different methods of measurement. Curve A represents data obtained from 200°C to 1000°C using our absolute cut-bar apparatus, with a specimen 1 in. in diameter and 1/2 in. long. These results were reported in the paper by D. R. Flynn at the last conference. It is pertinent to note that the specimen was held at 1000°C for about 275 hours with no perceived change in conductivity at this or lower temperatures as a result of the prolonged high temperature treatment.

Curves B and C were obtained using the low temperature model of our long-bar apparatus for metals (see 1961 Conference, pp. 13-16), with a cylindrical Pyroceram specimen 4.44 cm in diameter and 31 cm long. The powder insulation surrounding the specimen inside the guard-tube was diatomaceous earth for Curve B, and opacified silica aerogel for Curve C. Chromel-alumel 26-gage thermocouples were used, butt-welded and tightly fitted in transverse slits in the cylindrical surface of the specimen. Curve D was obtained in the high temperature model of the metals apparatus, using the same specimen but with 0.38 mm platinum-10% rhodium:platinum thermocouples. The results represented by the smoothed curves B, C and D have been corrected for the estimated longitudinal heat flow in the powder

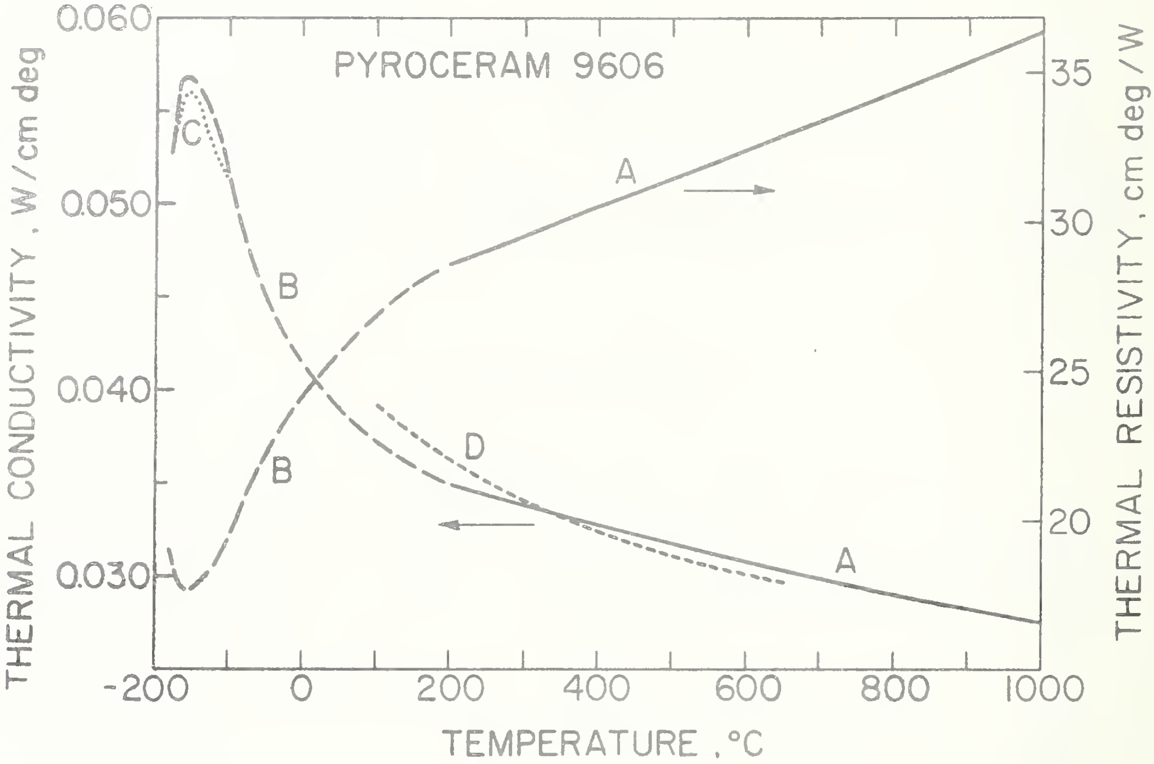

FIGURE 1. THERMAL CONDUCTIVITY AND THERMAL RESISTIVITY OF A SAMPLE OF PYROCERAM CODE 9606. CURVE A: DATA FROM ABSOLUTE CUT-BAR APPARATUS, SHORT CYLINDER; CURVES B, C, AND D: DATA FROM LONG-BAR METALS APPARATUS, WITH DIATOMACEOUS EARTH INSULATION (B); OPACIFIED SILICA AEROGEL (C); AND POWDERED ALUMINA (D)

insulation surrounding the specimen, on the assumption that the relative contributions from the specimen and guard are proportional to their cylindrical surface areas (i.e., to their radii). The correction increased with temperature, ranging from -0.4 to -1.8 percent for Curve B, from -0.2 to -0.3 percent for Curve C, and from -2.3 to -4.8 percent for Curve D.

Curve B is in good agreement with Curve A at their juncture at 200°C; there is a maximum difference of about 3 percent between Curves B and C in their common temperature range from -100°C to -180°C. Curve D, obtained in the high temperature metals apparatus with alumina powder insulation, departs rather widely (from +5 to -2 percent) from Curves A and B. It is believed that the error in Curve D is considerably larger than that of the other curves, because of the greater ratio of the conductivity of the powder insulation to that of the specimen, for alumina.

Conductivity values taken from the smooth curves A and B of Figure 1 are given in Table 2. It is worth noting that the thermal resistivity of the sample increases linearly with temperatures above 200°C.

TABLE 2. THERMAL CONDUCTIVITY OF A SAMPLE OF PYROCERAM CODE 9606

T °C	k W/cm deg	T	k
-180	0.0527	200	0.0350
-158 approx.	.0568 (peak)	400	.0327
-100	.0514	600	.0308
0	.0413	800	.0290
100	.0372	1000	.0275

In view of the apparent promise of Pyroceram 9606 as a conductivity reference material, as indicated by the information given here obtained on the single sample available to us, it was decided to purchase a stock of about two cubic feet--large enough to enable all necessary measurements and tests, and yet to leave enough material for distribution as conductivity reference samples for the proximate future _if_ the material proves satisfactorily uniform and stable for such use. The stock was received a few months ago, in the form of seven 18-in. diameter disks about 2 1/4 in. thick, all poured from one melt, and all given the same ceramming heat treatment. Currently, seventeen core-drill samples have been taken from the seven disks for density determinations to provide a first, and statistical, estimate of uniformity. If this appears satisfactory, the stability of the conductivity of the material in the face of temperature exposure will be investigated by successive measurements at temperatures near that at which the peak of conductivity occurs (approximately -158°C), in the comparative apparatus mentioned earlier in our

Progress Report, to be made on one or more samples subjected to various heat treatments. The same apparatus can also be used to investigate uniformity of thermal conductivity, as compared to uniformity of density. If the material passes well enough through these gantlets, the final step will be to determine its conductivity as accurately as possible.

ALLOYED METALS

A reference material in the niche or conductivity range 100 to 500 mW/cm deg seems especially desirable in view of the current importance of heat resistant alloys and stainless steels, which are relatively low in conductivity for metals.

Because a moderate stock of Inconel 702 all from one billet was in our possession, which had the desirable qualities of excellent oxidation resistance to over 1200°C, and an appropriate conductivity for this niche, it appeared worthwhile to investigate its possible usefulness as a thermal conductivity reference material. Experimental data on the conductivity of this nickel-chromium alloy were reported to you at the Ottawa conference; since then a few additional measurements have been made, at our laboratory, and at least at one other which is scheduled to report at this conference.

The chief question now appears to be that of the stability of thermal conductivity of the alloy in the face of its temperature history and thermal experience, and whether it may be possible to make it adequately stable by a suitable heat treatment. The alloy was obtained in a "solution-annealed" condition (i.e., heated to 1080°C and air-quenched to room temperature). It is age-hardenable, however, by precipitation of a gamma-prime phase, which occurs at temperatures between about 900° and 650°C. The coarseness of the precipitate, and its amount, depend on the thermal history.

At the Ottawa conference, we presented our available results (Table 3 and Figure 3, pp. 331-332), concluding that from 800° to 1200°C common values of conductivity were satisfactory for both the previously solution-annealed and the previously age-hardened alloy, but that at and below 700°C the age-hardened alloy was from one to three percent higher in conductivity than the solution-annealed alloy. Further evidence in this connection has been obtained in some measurements just completed, made in our long-bar apparatus for metals (low temperature model) from -157°C to 540°C.

A bar specimen of the alloy 2.54 cm in diameter and 37 cm long was machined for testing, and solution-annealed by heating to 1080°C, followed by rapid air-cooling in a fan blast. After its conductivity had been measured as indicated below, the same specimen was age-hardened by heating it to 1080°C, and slowly cooling it with two constant-temperature steps as shown in Figure 2. The age-hardened specimen, with new thermocouples, was then measured, undergoing substantially the same thermal history in the second series of tests as in the first.

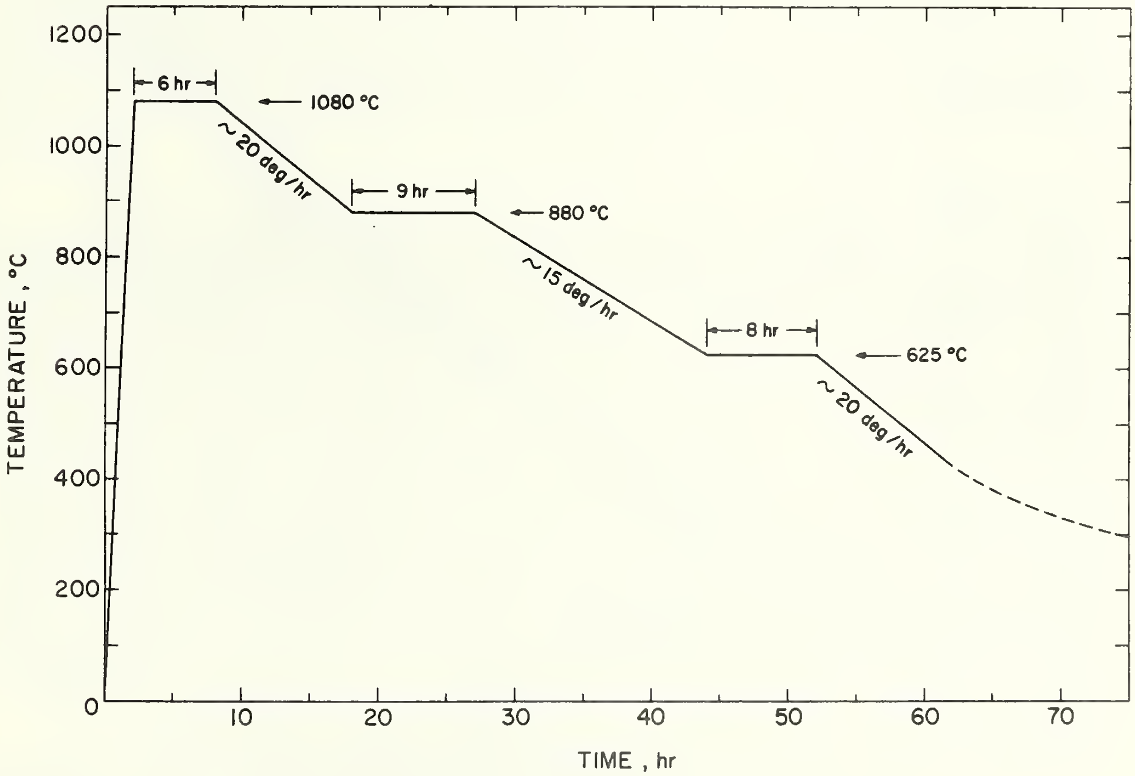

FIGURE 2. AGE-HARDENING TREATMENT OF INCONEL 702 SPECIMEN

The tests were conducted using diatomaceous earth powder insulation, and chromel-alumel thermocouples, and using as coolants both liquid nitrogen and 40°C water. The temperatures of the specimen, at the same test stages, were substantially duplicated in the two series of tests; the times elapsed in each stage were roughly the same.

The results are shown in Figure 3, by the smoothed curves A (for the age-hardened specimen) and C (for the solution-annealed specimen).

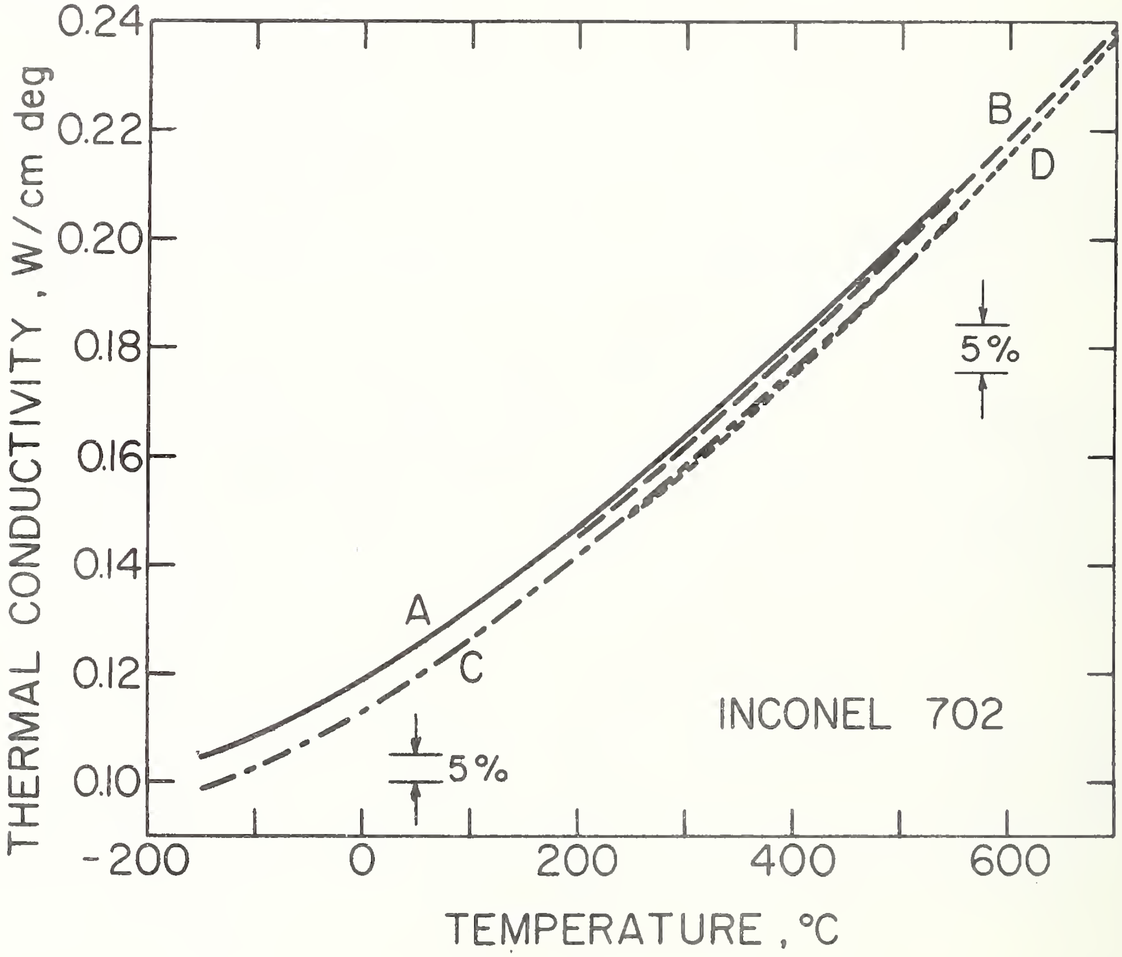

FIGURE 3. THE THERMAL CONDUCTIVITY OF INCONEL 702 ALLOY, SOLUTION-ANNEALED (CURVES C AND D) AND AGE-HARDENED (CURVES A AND B), AS DETERMINED BY TWO DIFFERENT METHODS: LONG-BAR APPARATUS FOR METALS (CURVES A AND C), AND ABSOLUTE CUT-BAR APPARATUS (CURVES B AND D)

Curves B and D, respectively, show for comparison the results previously reported by D. R. Flynn for the age-hardened and the solution-annealed alloy, as obtained by measurement of a 1-in. diameter by 3-in. long specimen in the absolute cut-bar apparatus.

The conductivity obtained for the age-hardened alloy is consistently greater than that obtained for the solution-annealed alloy, by about 6 percent at -150°C and about 3 percent at 540°C. The difference is in good agreement with the difference reported earlier by Flynn.

The difference between the age-hardened and the solution-annealed alloy would not militate against its use as a reference material if either one or both of the conditions was stable under later thermal experience. To investigate this point, the specimen, in each condition, was held steadily at the temperatures existing during the measurements at the highest temperature (i.e., that yielding a mean temperature of 540°C for the hottest of the six measuring spans of the specimen) for 90 hours (solution-annealed) and 127 hours (age-hardened), at which times observations were made to re-determine the conductivity at that sustained test condition. Following this, the specimen in each case was lowered in temperature to the test temperature conditions yielding a mean temperature of 290°C for the hottest span, at which condition the conductivity was again determined.

The results of these tests are given in Figure 4, which shows the percentage change of conductivity of each of the six 3.51-cm measuring spans of the specimen as a result of the thermal experience of the span. A slight decrease was obtained for both the solution-annealed and the age-hardened specimen, in the spans held above 300°C, as a result of sustained holding of the highest-temperature test condition. However, on lowering the specimen to the cooler test condition shown at the bottom of Figure 4, a marked decrease of conductivity, up to 4 percent, was obtained for the three hotter spans of both specimens. The apparent reduction of more than one percent in the conductivity of the three cooler spans of the age-hardened specimen, not evident for the solution-annealed specimen, may be spurious. It is difficult to account for a change which, if real, must have occurred at temperatures below 325°C, and even below 165°C. These results became available to us less than a week ago, and there has not been time to scrutinize all data for accidental errors.

During these measurements we were able also to make measurements of the average electrical resistivity and thermoelectric power (against chromel) of the several spans of the specimens at their various mean temperatures as the tests progressed. We have not had time to analyse these results fully, or to prepare them for presentation here. The electrical resistivity changes appear consistent with the thermal conductivity changes, though opposite in magnitude. In connection with the lower plot of Figure 4, the resistivity changes for all spans of the age-hardened specimen were substantially less than the corresponding changes for the solution-annealed specimen, suggesting that the indicated thermal

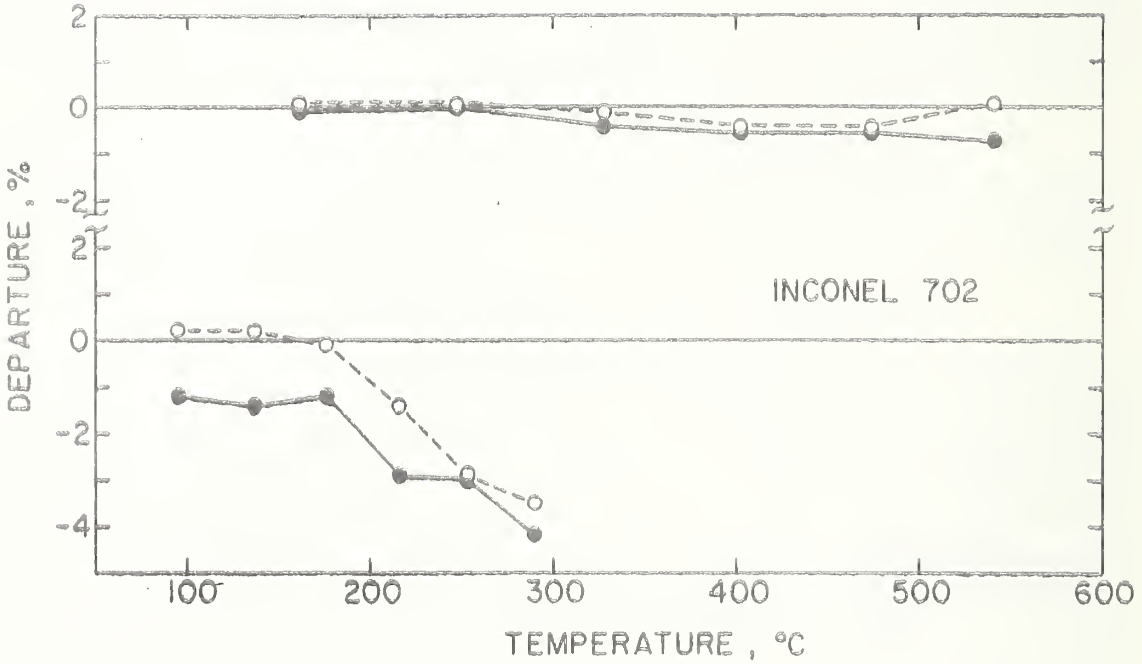

FIGURE 4. THERMAL CONDUCTIVITY CHANGES OF INCONEL 702 ON HOLDING STEADILY AT THE TEMPERATURES SHOWN IN THE TOP PLOT FOR FROM 90 TO 127 HOURS. THE LOWER PLOT SHOWS THE CHANGES FOUND IN A FOLLOWING TEST AT LOWER TEMPERATURES, ------ SOLUTION-ANNEALED AND ——— AGE-HARDENED

conductivity changes for the age-hardened material may be in error, and ought to be less than those of the solution-annealed alloy.

The change of the conductivity of the hotter spans indicated by the lower temperature test is great enough to be very seriously considered as unfavorable to use of Inconel 702 as a reference material. For the present, we are reluctant to dismiss Inconel 702 at once from further consideration, although it may be necessary in the end. A considerable amount of the last data obtained needs further scrutiny and perhaps additional measurements are in order. We hope also to receive at this conference information developed by others which may illuminate the path to a conclusion. A practical consideration which concerns us is that if a metallic alloy suitable as a reference for this particular conductivity niche is to be found, it is more than likely to contain as a constituent one of the transition elements, which may be the cause of the instability that appears so far to be indicated. In such case, a suggestion for an alternative material may not be easy.

PURE METALS

We have not yet undertaken any serious work on development of a pure metal reference standard, partly because R. W. Powell's work, and evaluation of data, on Armco iron have made the latter an available standard for conductivities from 500 to 1000 mW/cm deg. Aside from the measurements undertaken in connection with the B.M.I. round-robin on Armco iron, reported at the last conference (Ottawa, pp. 325-329), and some later electrical resistivity measurements, we have not worked further with Armco iron.

At the last conference we expressed a desire to make some absolute determinations of the thermal conductivity of NBS freezing point standard lead, but we have not been able to undertake them thus far. Also, we have considered from time to time the practicability of using pure platinum as a thermal conductivity reference standard material, especially for high temperatures, but the matter has not as yet progressed to the stage of undertaking it.

SUMMARY OF (11) RETURNED QUESTIONNAIRES
Number of Replies in Various Categories

Category	Test Materials	Desired Reference Standards
Thermal Conductivity, mW/cm deg		
0.1 - 1	2	2
1 - 20	7	7
20 - 70	7	7
70 - 500	5	6
500 - 5000	3	3
	24	25
Thermal Diffusivity, cm^2/sec		
< 0.01	1	0
0.01 - 2	2	2
	3	2
Temperatures, °C		
− 270 to 0	4	3
0 to 1200	10	9
1200 to 2200	6	6
2200 to 3000	2	1
	22	19
Materials		
Ceramics	7	9
Metals	6	12
Semi-Conductors	4	0
Powders	1	1
	18	22
Limit of Test Uncertainty, %		
< 1	1	
1 - 3	4	
3 - 5	6	
5 - 15	2	
	13	
Use of Reference Standard		
To check apparatus		9
To calibrate apparatus		6
To train operator		1
		16

Types of Apparatus	
Absolute--along bar	0
--radial	4
--flat hot plate	1
Comparative--meter	4
--Powell ball	1
Diffusivity	4
	14

Atmospheres	
Air	0
Vacuum	10
Special	6
Chemical problems	5


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