NASA CR-72710

INTERIM REPORT

AN EXPERIMENTAL INVESTIGATION OF THE THERMAL CONDUCTIVITY AND ELECTRICAL RESISTIVITY OF THREE POROUS 304L STAINLESS STEEL "RIGIMESH" MATERIAL TO 1300°K

by

R. P. Tye
DYNATECH CORPORATION

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA-Lewis Research Center
Contract NAS3-12012
Anthony Fortini, Project Manager

NOTICE

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R. P. Tye
DYNATECH CORPORATION
17 Tudor Street
Cambridge, Massachusetts 02139

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August 27, 1970

CONTRACT NAS 3-12012

NASA Lewis Research Center
Cleveland, Ohio
Anthony Fortini, Project Manager
Chemical Rocket Division

FOREWARD

This document reports on an investigation to determine the thermal conductivity and electrical resistivity of three porous 304 L stainless steel "Rigimesh" materials to 1300°K. This work was performed by the Dynatech Corporation as a subcontract to The Boeing Company as a portion of the "Fundamental Study of Transpiration Cooling," NAS 3-12012, initiated in July 1968. The work was performed under the direction of A. Fortini of the NASA Lewis Research Center and J. L. Dutton of The Boeing Company.

Dr. J. C. Y. Koh of The Boeing Company coordinated the technical aspects of the investigation with the Dynatech Corporation.

AN EXPERIMENTAL INVESTIGATION OF

THE THERMAL CONDUCTIVITY AND

ELECTRICAL RESISTIVITY OF THREE POROUS

304 L STAINLESS STEEL "RIGIMESH" MATERIALS TO 1300K

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R. P. Tye

ABSTRACT

Measurements of the thermal conductivity of a "Rigimesh" stainless steel in three different porosities have been undertaken in vacuum and a nitrogen atmosphere over the temperature range 300 to 1300K by the comparative method with the heat flow perpendicular to the surfaces of the sample.

Electrical resistivity measurements have been undertaken in two mutually perpendicular directions on separate samples of the same material.

The results indicate that although there is a threefold increase in thermal conductivity when the porosity decreases from approximately 40% to 10% and a corresponding decrease in electrical resistivity for the same current and heat flow directions, the value of the derived Lorenz Function remains constant to within 8%.

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1.0 INTRODUCTION

The comparative method of measuring thermal conductivity with samples in the form of slabs 63 mm square and 25 mm thick has been used to evaluate the conductance of three different porosities of a "Rigimesh" woven and sintered stainless steel. Measurements have been undertaken in vacuum and in a nitrogen atmosphere over the temperature range 300 to 1300K to investigate possible gaseous and radiative components of thermal conductivity. Electrical resistivity determinations were made on small rod shaped samples cut from the same pieces as the thermal conductivity samples.

2.0 OBJECT OF INVESTIGATION

Porous metals are of importance in transpirational cooling problems over a wide temperature range. For design purposes basic property data are required for the newer types of materials now being used particularly at temperatures up to 1300K and for different environments.

The total thermal conductivity λ_T of such a material may be represented by

$$\lambda_T = \lambda_E + \lambda_R + \lambda_{C_1} + \lambda_{C_2} \qquad (1)$$

where λ_E is the component due to electronic conduction λ_e, plus lattice conduction λ_g, λ_R is the component due to radiation, λ_{C1} is the component due to gas within the trapped pores including any possible convection and λ_{C2} is the component due to gas within the open pores including any possible convection. In vacuum, this term is zero. For materials of low porosity λ_{C1} will be very small so that the total quantity measured will normally be due to the λ_E and λ_R contributions only.

For solid metals in general

$$\lambda_E = \lambda_e + \lambda_g \qquad (2)$$

$$\lambda_e = \frac{LT}{\rho} \qquad (3)$$

Here λ_e is in the W m^{-1} deg^{-1} unit, ρ in the ohm m unit, T the absolute temperature, K, and L will thus be in the V^2 deg K^{-2} unit. In this particular case L has the Sommerfeld theoretical value of the Lorenz function (2.443×10^{-8} V^2 deg K^{-2}).

Electrical resistivity is a function of electronic conduction only. Thus measurements of ρ and λ_E in vacuum coupled with the assumption that λ_e can be calculated from (3) can lead to the separation of the different components. Measurements will lead not only to true effective properties at elevated temperatures but also to possible examination of the behavior of the different components with temperature.

In most cases λ_g varies as T^{-1}. At temperatures well in excess of the Debye temperature, for a particular system, λ_e is the predominant transport mechanism. However, it has been found empirically from measurements in the range 200 to 800 K on different alloy systems that the Wiedermann-Franz-Lorenz relationship may be modified in the following manner

$$\lambda_E = \frac{L_1 T}{\rho} + b \qquad (4)$$

where L_1 is a number similar to the Sommerfeld value, b is a constant lattice component. This form of relationship has proved very useful in the prediction of the thermal conductivity behavior of new alloys of particular systems from the relatively simple measurement of electrical resistivity. This has proved true for some porous metal systems in addition. It is, therefore, a possibility that some form of empirical relationship may hold for the particular type of porous metal now studied.

The porous stainless steel under investigation is unlike the sintered spherical particle types for which the above analyses are valid. It is basically in the form of a thin woven layer of fibers with successive layers placed on top of each other and the whole sintered together. It is possible, therefore, that for this particular form of the material there will be an additional thermal resistance between the layers and in consequence the separation of the other components of thermal conductivity may be affected by this additional and possibly varying parameter.

3.0 MATERIALS STUDIED

The material supplied for test was described as porous 304 L stainless steel Rigimesh and stated details of the three different porosities are given in Table I. Three samples of each material were supplied and characterized in Reference 1.

1. A thermal conductivity sample consisting of a slab approximately 63 mm square and 25.4 mm thick with two small holes approximately 1.3 mm diameter drilled to the center at levels approximately 3 mm from each surface. The surface of this sample was prepared to be as flat and smooth as possible making sure that the total thickness was uniform and the faces were parallel. The heat flow axis was perpendicular to the weave pattern. The samples were marked A1, B1, and C1.

2. One electrical resistivity sample consisting of a rectangular bar approximately 6 mm square and 25 mm long, cut such that current flow would be along the same direction as the heat flow axis in sample 1. These were marked A2, B2, and C2.

3. A similar rectangular bar approximately 6 mm square and 60 mm long, cut such that the current axis was parallel to the weave pattern. These were marked A3, B3, and C3.

On receipt of samples the dimensions were all measured accurately. Following this, electrical resistivity measurements were made at room temperature on the appropriate sample of each material.

For the purposes of this test the ends of the sample were fitted with current electrodes and then laid on knife edges a known distance apart. A steady d.c. current from a 24 V battery was applied to the sample and the potential drop developed across the knife edges was measured with a Leeds and Northrup potentiometer in addition to that developed across a calibrated standard 0.001 Ω resistance in series with the sample. The current was reversed to eliminate thermal voltages and the potential measurements repeated. The electrical resistivity was derived in terms of the ratio of the above voltages, the known dimensions of the sample and the distance between the knife edges. The details of the test samples together with the measured electrical resistivities are given in Table II.

4.0 TEST PROCEDURE

(1) Thermal Conductivity:

For the purposes of the test, fine gauge (0.2 mm diameter) chromel-alumel thermocouples insulated in twin bore silica and alumina 1.3 mm diameter tubing were fixed tightly into the holes in the sample and discharged welded in position so that the bead was in very good contact with the metal. The sample was placed between two similar sized samples of Inconel 702 (a material of known thermal conductivity over the temperature range of the investigation) with the same type of thermocouple instrumentation. To minimize contact resistance a very thin layer of palladium foil was placed between each surface. The composite sample was placed between the plates of an upper heater and heat sink combination. A reproducible load of approximately 10^6 N m^{-2} was applied to the top of the sample stack to assist in reducing contact resistance at the surfaces. A 12 cm diameter alumina guard tube which could be heated and which had thermocouples attached along its length was placed around the system and the whole of the interspaces and surrounds filled with dry sil-o-cel a good heat insulating powder. The complete assembly was covered with a bell jar and the enclosure evacuated to a pressure lower than 10^{-3} torr. Figure I shows a schematic assembly of the Dynatech TCFC-N-20 thermal conductivity apparatus used.

By means of suitable adjustments to the power in the various heaters and of the cold sink temperature a steady temperature distribution was maintained in the system and undue radial heat loss prevented by keeping the guard tube gradient matched approximately to that in the sample stack.

Equilibrium conditions were attained normally in the order of 6 to 8 hours after any large adjustements of power were made to raise or lower the average temperature to the required data point levels. During this period small adjustments were made to insure that the guarding was as good as possible.

At equilibrium conditions the temperature at various points in the system were evaluated from the thermocouple readings obtained with the potentiometer. The heat flow, Q, in the test specimen was derived in terms of that flowing through each Inconel reference material.

From

$$Q = \lambda_r A \frac{dT}{dx} \qquad (5)$$

where A is the area of cross section, dx the distance between the thermocouple positions, dT the temperature difference and λ_r is the thermal conductivity of the reference material at its appropriate mean temperature.

The thermal conductivity of the test sample was evaluated from a knowledge of the mean heat flow in the upper and lower reference materials, the temperature difference across the sample and its known dimensions.

Measurements were made in the above manner at four mean temperatures in the approximate range 400 to 1300K and a repeat measurement taken at a lower temperature in that range after attaining the highest temperature. After this latter measurement the apparatus was backfilled with nitrogen gas to 1 atmosphere and measurements made in this environment at three temperatures in the above range.

Appendix A contains the following details of the evaluation of thermal conductivity in this investigation:

1. A curve of the thermal conductivity versus temperature of the Inconel 702 material used as the reference material (References 2 and 3).

2. A copy of the complete set of data and the subsequent evaluation of thermal conductivity for a random data point.

3. A table listing details of the environmental conditions, the mean temperatures, and the calculated thermal conductivity showing the spread in the mean deviation of thermal conductivity.

(2) Electrical Resistivity

From the initial room temperature measurements it appears that the 9.3% porosity materials was reasonably homogeneous with only the order of 5% anisotropy in the electrical resistivity. However, for the other two materials there was a considerable difference in the values obtained for the two directions. This would indicate a large anisotropic effect. Presumably this is due to the fact that the material is formed by pressing and sintering successive layers of the woven material to form the final body. In this case it is quite likely that for the higher porosities, additional resistances will be formed at the interfaces of the successive layers. These will affect both electrical and thermal flow in the direction perpendicular to the weave pattern while not restricting that in the direction parallel to the weave pattern. Measurements of electrical resistivity were made on both samples 2 and 3 of each of these materials.

For the purpose of this test, two thermocouples were spot welded into small grooves cut into one edge of the sample. Current electrodes were welded to each end and the sample was assembled at the center of a furnace which could be heated uniformly. Measurements were made as described previously in Section 3.0, except that now the potential difference across the sample was measured using "like" arms of the two thermocouples, at successive increments of temperature in a nitrogen environment at 1 atmosphere over the temperature range covered in the thermal conductivity measurements. Repeat measurements were taken on cooling to see if any changes had been produced by the heat treatment.

5.0 RESULTS

The experimental results of thermal conductivity and electrical resitivity obtained for the three particular samples tested are shown in Figures 2 and 3 (a) - (c) respectively.

Brief mention should be made of the fact that in the sample preparations some of the pores in the surface layers of all samples were filled with metal due to the smoothing processes required to obtain uniform thickness. The effect of having this very small skin layer of higher density is felt to be insignificant. The properties being measured are the bulk properties of substantial samples of material and the skin layer contribution to the total is negligible. There is one advantage in that this skin provides a large surface area of contact between the sample and the reference materials and thus helps to improve uniform linear heat flow within the sample.

For the case of the thermal conductivity, particularly for the 20.3 and 38.5% porosity samples, the values obtained in vacuum were in all cases slightly lower than those obtained in nitrogen. In all cases the vacuum level of the tests was below 2×10^{-2} torr and in the majority of cases below 10^{-3} torr. It is possible that the presence of a gas in the pores would tend to give higher values but for the present porosities they should be of the order of a maximum of 1 to 2% when one takes into account the very low thermal conductivity of nitrogen gas even at elevated temperatures.

In measurements of thermal conductivity by the comparative method, errors occur due to non-uniformity of heat flow due mainly to uncertainties of resistances at the contacting surfaces caused by non-uniformity of sample and warpage on heating. Heat losses or gains from the side of the sample stack will occur whenever the temperature of the surroundings do not exactly match those at corresponding levels in the sample stack. To overcome this a cylindrical guard tube surrounds the stack and the top and bottom temperatures are matched. However, these are always interfacial resistances at contacting surfaces which will create step functions in the complete gradient along the stack. When measurements are carried out in vacuum, the interstitial gas at the surfaces is removed and the magnitude of the step functions increase thus making guarding more difficult and the likelihood of larger uncertainties in extraneous heat flows.

Experimentally, the percent deviation from the mean thermal conductivity as measured by using the top and bottom reference materials was generally less than 5% when measurements were undertaken in nitrogen. For the case of measurements in vacuum this difference was usually of the order of 10% and in two extreme cases was 11 and-15% respectively. Thus the uncertainty of the value obtained in vacuum is greater than that obtained with the nitrogen present. If the uncertainty limits are applied to each data point individually then they overlap in all cases. Smooth curves have been drawn through the total number of data points for each material and as a result only one of the individual data points is more than 5% from the mean curves.

For the case of the electrical resistivity smooth curves have been drawn through the experimental points and all values are seen to be within 1% of the appropriate curve. In all of the measurements no significant changes were noted due to the heat treatment which the samples endured during the investigation.

Table III contains values of the measured total thermal conductivity, λ_T, and electrical resistivity, ρ , obtained at regular temperature intervals from Figures 2 and 3(a) - (c). Also included are values of the Lorenz Function $\frac{\lambda_T \rho}{T}$ using the appropriate samples of each, identified in Table II, where the heat and current flows were for the same orientation. For the three samples measured, although the individual values λ_T and ρ differ significantly, the values of the Lorenz Function at different respective temperatures differ by less than 5%. This would indicate that for other samples of this type of material the thermal conductivity could be calculated to better than 5% if electrical resistivity measurements were combined with the mean values of the Lorenz Function obtained in this investigation. The fact that the Lorenz Function is approximately constant also indicates that under the test conditions of these studies the radiation component λ_R is negligibly small.

6.0 REFERENCES

1. R. E. Regan, "Characterization of Porous Metal Matrices for Transpiration Cooled Structures," NASA CR-72699 (1970).

2. H. E. Robinson and D. R. Flynn, "The Current Status of Thermal Conductivity Reference Standards at the National Bureau of Standards," The Third Conference on Thermal Conductivity, Vol. I (1963).

3. M. J. Laubitz, "Inconel 702 - Thermal Conductivity Standard," The Third Conference on Thermal Conductivity, Vol. I, (1963).

TABLE I

DETAILS SUPPLIED OF THE 304L STAINLESS STEEL RIGIMESH MATERIALS USED IN THE PRESENT MEASUREMENTS

POROSITY (%)	WARP WIRE DIAMETER (In.)	DUTCH WEAVE (OR WOOF OR FILL) WIRE DIAMETER (In.)	THICKNESS PER LAYER (In.)	NUMBER OF WARP/IN.	NUMBER OF WEAVE/IN.
9.3	.014	.0094	1.02×10^{-2}	22.3	109*
20.3	.014	.0094	1.36×10^{-2}	22.7	110
38.5	.014	.0094	2.11×10^{-2}	23.4	108

* The nominal number is 110. Due to non-uniformity of material there may be small variations from location to location.

TABLE II

DETAILS OF SAMPLES TESTED

DESIGNATION	STATED POROSITY, %	SIZE, mm	ELECTRICAL RESISTIVITY, ohm m at 22°C
A 1		63.9 x 63.8 x 20.6 +	-
2	9.3	6.36 x 6.26 x 20.11	92.6×10^{-8}
3		6.22 x 6.30 x 59.46	87.2×10^{-8}
B 1		64.4 x 64.6 x 26.19 x	-
2	20.3	6.55 x 6.26 x 26.32	147.5×10^{-8}
3		6.70 x 6.19 x 58.71	120×10^{-8}
C 1		62.75 x 62.59 x 24.46 *	-
2	38.5	6.64 x 6.45 x 25.52	300×10^{-8}
3		6.73 x 6.60 x 59.07	205×10^{-8}

+ Distance between Thermocouple Holes 14.4 mm

x Distance between Thermocouple Holes 20.1 mm

* Distance between Thermocouple Holes 18.2 mm

TABLE III

TOTAL THERMAL CONDUCTIVITY, λ_T, W m^{-1} deg^{-1}, 10^8 x ELECTRICAL RESISTIVITY, ρ, ohm m AND DERIVED 10^8 x LORENZ FUNCTION, L, V^2 deg K^{-2}, FOR POROUS 304L "RIGIMESH" STAINLESS STEEL

Temperature, C	K	9.3% Porosity				20.3% Porosity				38.5% Porosity			
		λ_{A1}	ρ_{A2}	ρ_{A3}	$L_{A1\,A2}$	λ_{B1}	ρ_{B2}	ρ_{B3}	$L_{B1\,B2}$	λ_{C1}	ρ_{C2}	ρ_{C3}	$L_{C1\,C2}$
100	373	13.7	100	94	3.68	8.6	159	130	3.67	4.3	327	221	3.76
300	573	16.2	117	110.2	3.31	10.1	187	152.5	3.30	5.1	384	255	3.41
500	773	18.9	130	122.8	3.18	11.6	208.5	170	3.13	5.8	424	287	3.18
700	973	21.5	140.5	132.7	3.11	13.0	225	184	3.02	6.55	451	307	3.03
900	1173	24.2	148.5	140.2	3.06	14.4	237.5	194.5	2.92	7.3	478	328	2.97
1000	1273	25.4	152	144	3.03	15.2	243	199.5	2.90	7.6	492	338	2.93

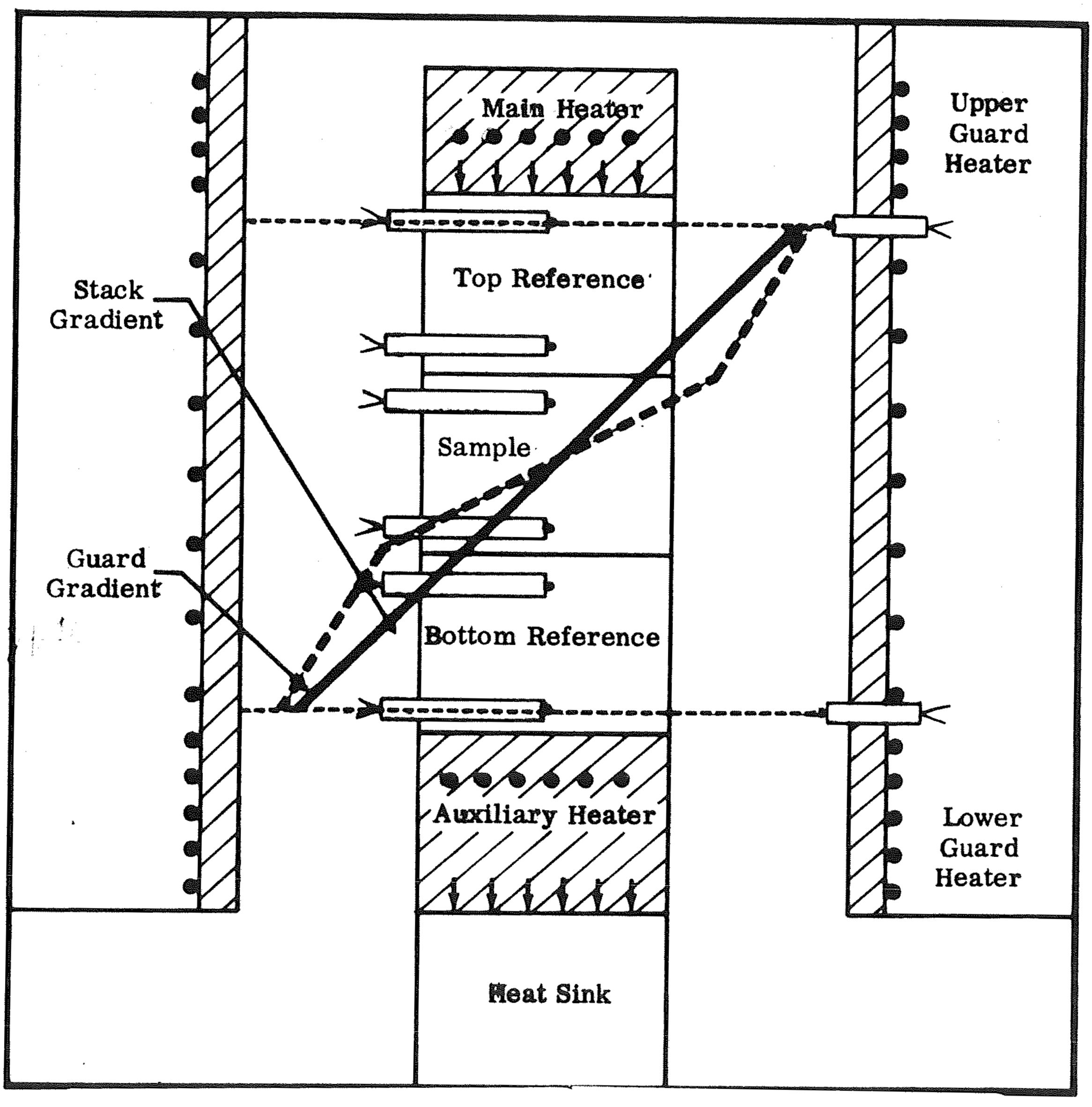


Figure 1 Schematic Assembly of Dynatech TCFCM Comparative Thermal Conductivity Apparatus

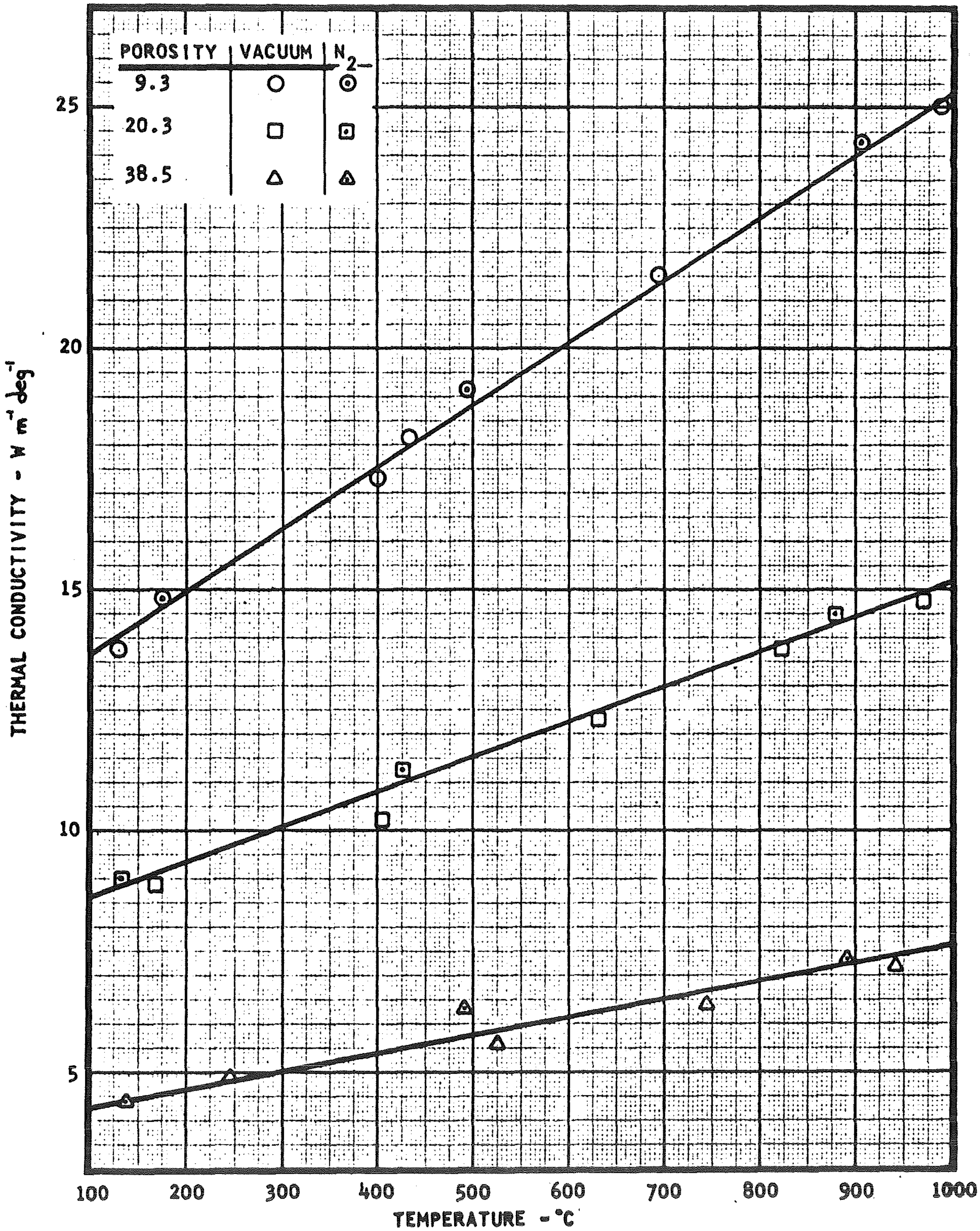


FIGURE 2. THERMAL CONDUCTIVITY OF 304L STAINLESS STEEL RIGIMESH

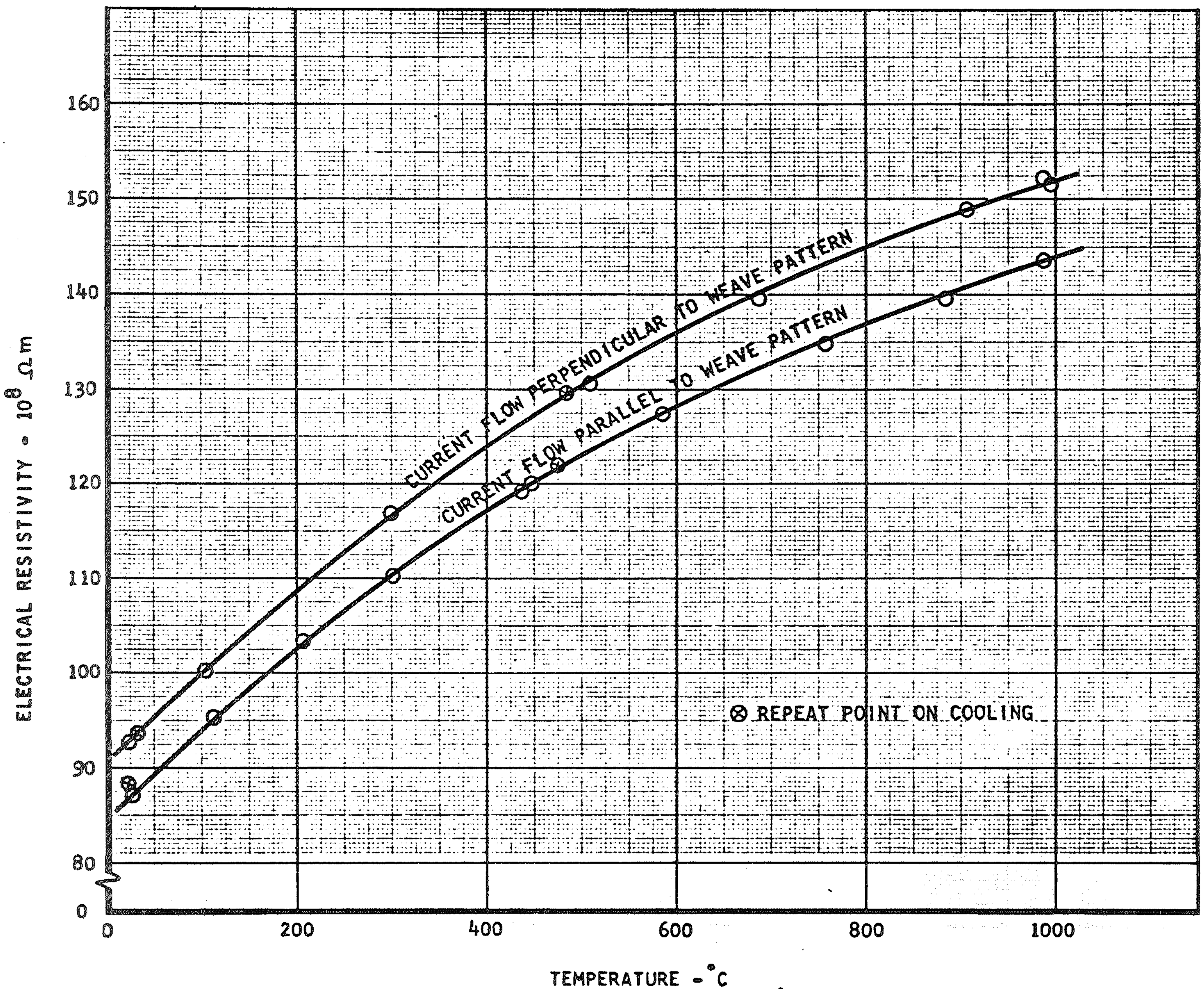


FIGURE 3a. ELECTRICAL RESISTIVITY OF 304L STAINLESS STEEL RIGIMESH (POROSITY 9.3%)

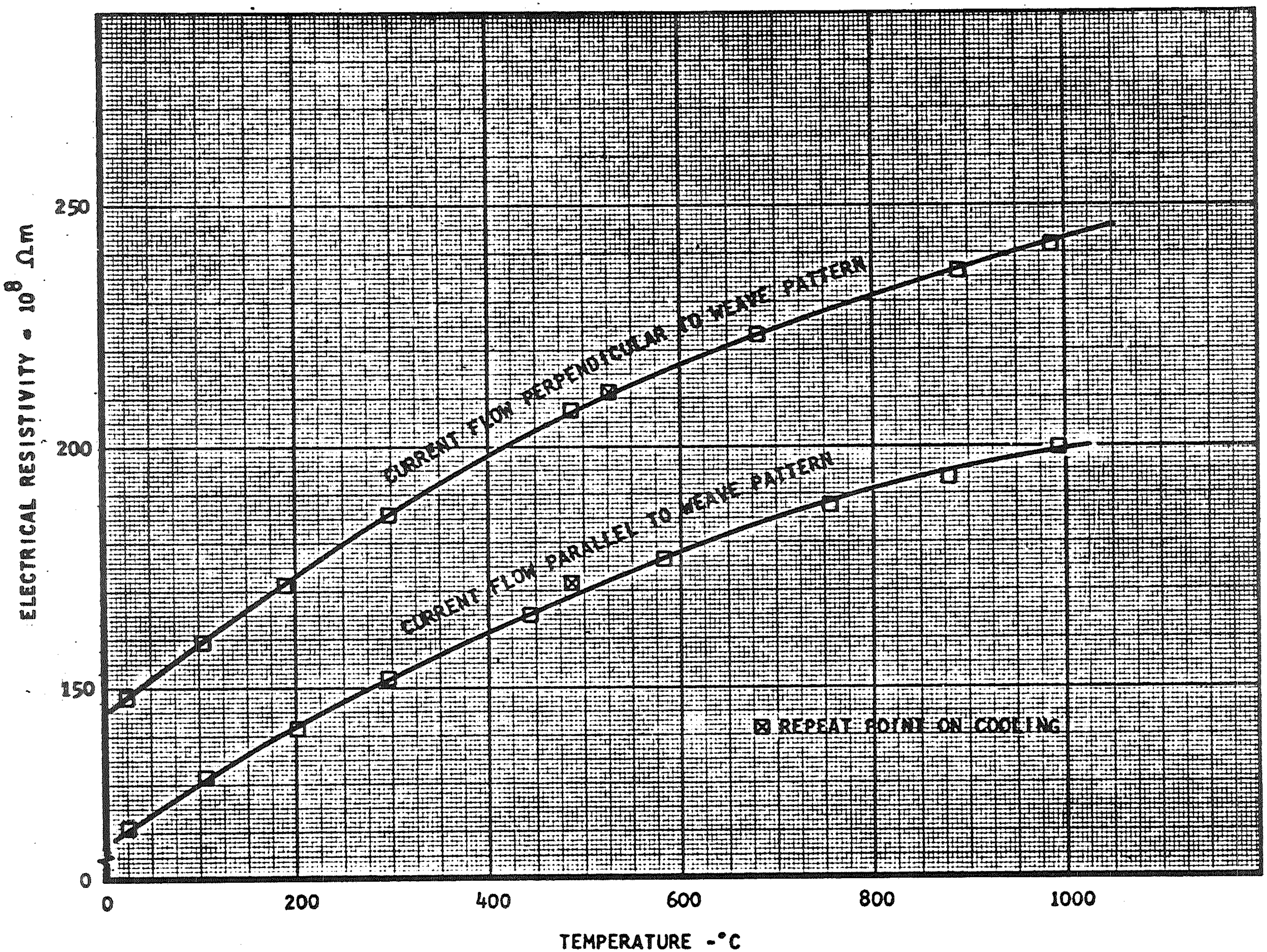


FIGURE 3b. ELECTRICAL RESISTIVITY OF 304L STAINLESS STEEL RIGIMESH (POROSITY 20.3%)

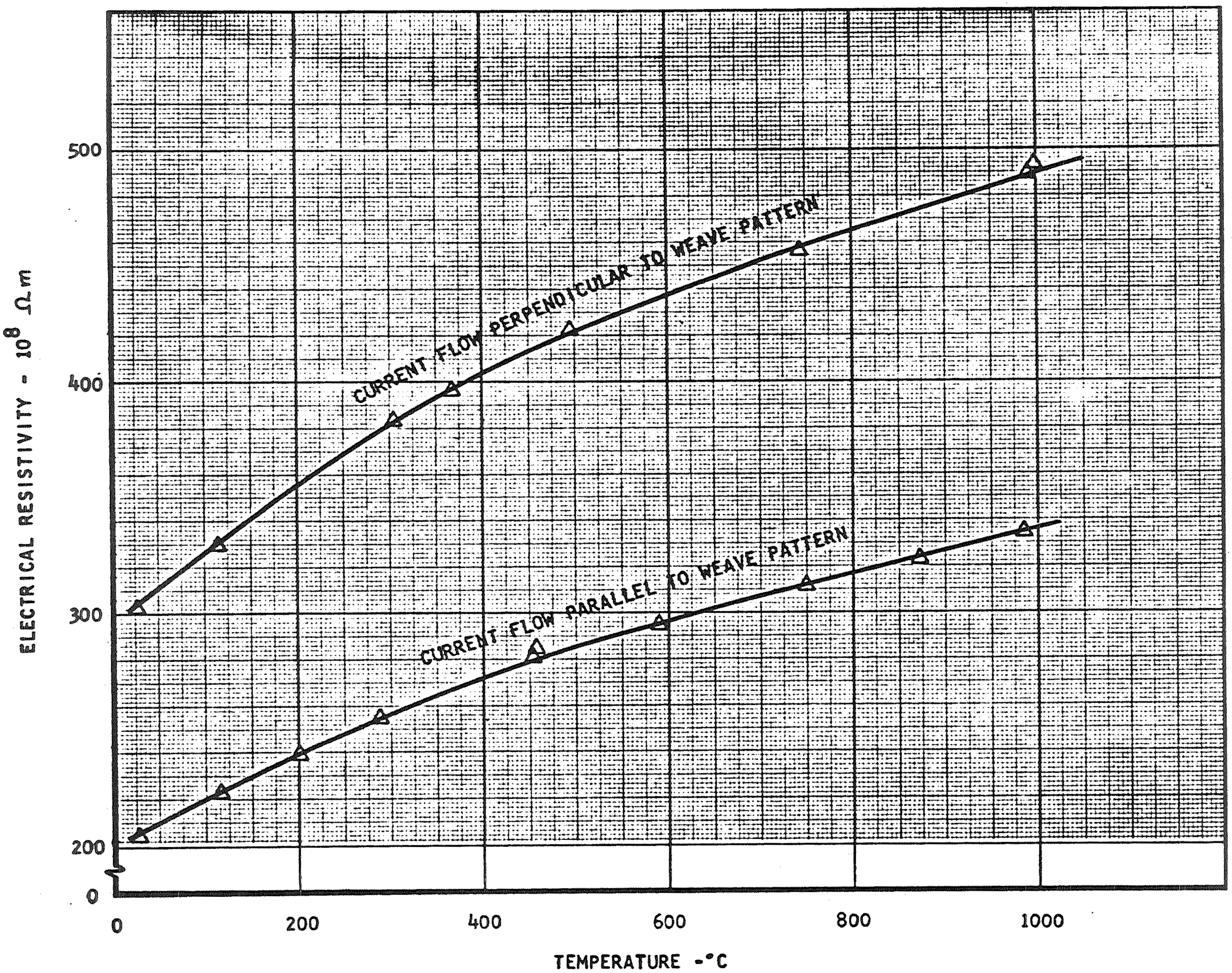
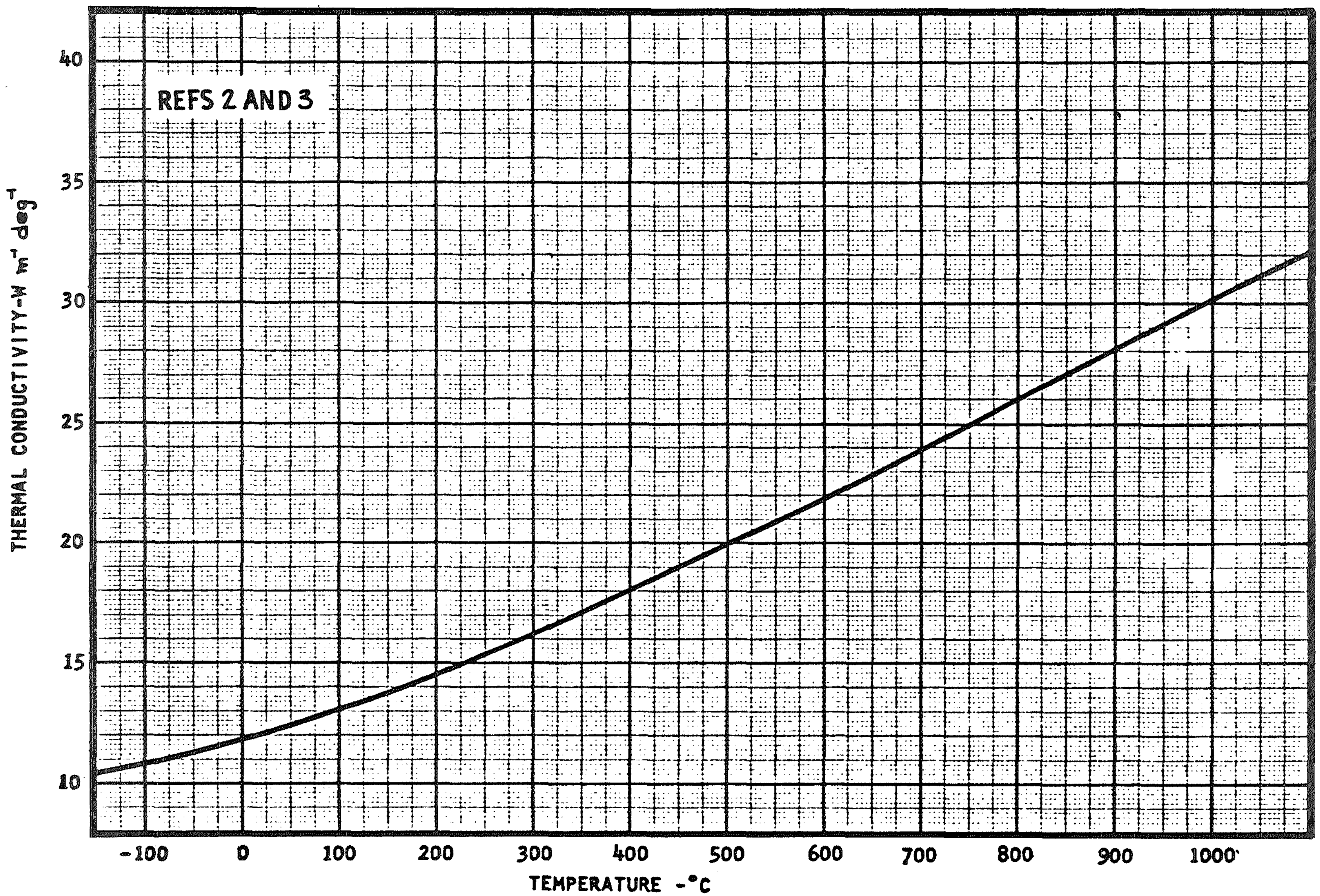


FIGURE 3c. ELECTRICAL RESISTIVITY OF 304L STAINLESS STEEL RIGIMESH (POROSITY 38.5%)



APPENDIX A(1) THERMAL CONDUCTIVITY OF INCONEL 702

DYNATECH CORPORATION — DATA REDUCTION SHEET

Thermal Conductivity by Comparative Method

Temperature Point: ~ 900 C

Project: BOE-3

Sample Designation: 20% Porosity

Bottom Heat Meter Material: } INCONEL 702
Top Heat Meter Material: } 3.1 UPPER, 3.2 LOWER

Thickness, X_s : ( ) - ( ) = (2.01)cm

Thickness, X_{BHM}: ( ) - ( ) = (2.54)cm

Thickness, X_{THM}: ( ) - ( ) = (2.54)cm

Thermocouple Material: CH/AL

Avg. of (how many) readings: 3 Data of (date): 6 AUG
15.00 — 16.30 hrs

T/C No	Readings, Mv.	°C
1	28.773	691.3
2	32.387	777.9
3	34.163	821.1
4	38.810	936.25
5	39.898	963.95
6	42.552	1031.8

Average Temperatures

BHM	Sample	THM
+ 2) 1469.2	+ 2) 1757.3	+ 2) 1995.7
734.6	878.7	997.9

Temperature Drops

BHM: 86.6

Sample: 115.15

THM: 67.85

k Heat Meters, watts / cm deg C

BHM: 24.7 @ 734.6 °C

THM: 30.2 @ 997.9 °C

$$k_s = k_{HM}\left[\frac{\Delta T_{HM}}{\Delta T_s}\right]\left[\frac{X_s}{X_{HM}}\right]$$

$$k_s\Big|_{BHM} = 24.7 \times \frac{86.6}{115.15} \times \frac{2.01}{2.54} = 14.7$$

$$k_s\Big|_{THM} = 30.2 \times \frac{67.85}{115.15} \times \frac{2.01}{2.54} = 14.08$$

Energy Spread

$$200\,\frac{k_s\big|_{BHM} - k_s\big|_{THM}}{k_s\big|_{BHM} + k_s\big|_{THM}} = 4.5\ \%$$

$\bar{k}_s$ = 14.4 watts / cm deg C @ 878.7 °C

 By: HS Date: 7 AUG '69

APPENDIX A (3)

DETAILS OF EXPERIMENTAL CONDITIONS FOR EACH DATA POINT

Sample	Environment	TEMPERATURE, C						λ Measured	Conductivity
		Mean Sample	ΔT	Mean Top Reference	ΔT	Mean Bottom Reference	ΔT	Wm^{-1} deg^{-1}	Difference *% T - B
A1	3×10^{-3} torr	131	17.0	192	29.5	104	30.0	13.7	7.0
	4×10^{-4} torr	402.5	22.1	552	33.1	248	43.1	17.3	4.8
	5×10^{-4} torr	695.1	48.4	786	73.1	599	81.6	21.5	5.4
	8×10^{-4} torr	990.5	35.0	1108	47.1	899	55.6	25.0	-3.6
	2×10^{-4} torr	433	40.0	520	64.4	360	71.9	18.1	4.4
	N_2	175	17.8	211	31.5	137	34.2	14.8	-1.1
	N_2	497	42.2	610	62.7	380	81.5	19.0	-4.6
	N_2	908.5	50.0	991	70.6	805	82.5	24.3	-2.0
B1	2×10^{-3} torr	169	15.7	189	13.0	138	12.4	8.9	9.7
	4×10^{-3} torr	628	49.9	735	33.2	520	36.1	12.3	11.2
	3×10^{-3} torr	825	87.8	936	52.8	755	60.3	13.7	0.8
	5×10^{-3} torr	970	102	1116	56.9	854	72.9	14.8	-7.3
	6×10^{-4} torr	404	68.1	516	45.8	323	50.9	10.3	8.4
	N_2	133	19.8	160	16.3	98	17.4	9.1	0.1
	N_2	431	74.3	513	52.2	339	62.9	11.3	-1.4
	N_2	878	115.1	998	67.9	735	86.6	14.4	-4.6
C1	2×10^{-3} torr	245	20.2	265	8.2	197	10.3	4.9	-15.2
	5×10^{-4} torr	526	62.0	624	22.7	375	26.4	5.6	9.1
	3×10^{-4} torr	943	118.8	1045	37.8	815	45.4	7.2	-2.2
	2×10^{-2} torr	740.5	85.8	851	29.2	636	32.7	6.4	7.2
	N_2	142	29.8	182	13.5	101	14.0	4.5	5.1
	N_2	495	122.5	613	49.0	360	61.1	6.3	2.9
	N_2	895	189.8	1067	63.5	679	82.8	7.4	2.4

$$\text{* Conductivity Difference T - B} \quad \frac{(\lambda_T) \text{ evaluated based on top reference} - (\lambda_B) \text{ evaluated based on bottom reference}}{\lambda T + \lambda B} \times 200$$

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