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The results of thermal conductivity measurements on Inconel 718 and 304 stainless steel by the comparative and flash diffusivity techniques are reported for the temperature range 0–700°C. For 304 stainless steel, excellent agreement with published data is found for the specific heat, thermal diffusivity, and thermal conductivity. In the case of Inconel 718, the measurements show that the conductivity depends critically on the sample thermal history and the metallurgical condition of the alloy. Measurements on a solution-treated sample indicated a conductivity function close to that reported previously, while precipitated samples showed a higher conductivity, similar to the conductivity data with Inconel 718 references. These results indicate that Inconel 718 is not a suitable reference for high-accuracy comparative thermal conductivity measurements unless its thermal history and associated conductivity function are known.

**KEY WORDS:** comparative technique; Inconel 718; stainless steel; standards; thermal conductivity; thermal diffusivity.

## **1. INTRODUCTION**

In making comparative thermal conductivity measurements, reliable standards or references are required in various conductivity ranges. Although it is possible to make measurements with substantial differences between the reference and the sample conductivities [1], to obtain the minimum error or variance for the measurement, it is desirable to have a reasonable match between the conductivities [2]. As comparative measurements have grown increasingly more accurate with improvements in equipment and techni-

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que, there has been a need to specify the conductivities of various reference materials to higher accuracies. This paper is devoted to a discussion of two materials, Inconel 718 and 304 stainless steel, which have been used as reference materials in our laboratory for samples with conductivities in the range  $10-30 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ .

Inconel 718 is suggested and supplied as a reference material by Dynatech R/D Co., a manufacturer of comparative thermal conductivity measurement systems [3]. Some preliminary comparative measurements using one of our Dynatech-supplied Inconel 718 references as a sample, together with either Pyroceram 9606 or Armco iron references, suggested that the conductivity of this particular Inconel 718 piece was not in particularly good agreement with the suggested conductivity [2]. This observation led us to study the conductivity of Inconel 718 in more detail and also to extend our study to 304 stainless steel. A recent review of thermal conductivity data for AISI 304 stainless steel indicates that there is a relatively small spread in these data in the temperature range 0–600°C [4]. The maximum deviation between the data and the recommended conductivity function is  $\approx 4\%$  in this temperature range and the average deviation is  $\approx 2\%$  [4].

In this study, we have measured the thermal conductivity of selected samples of Inconel 718 and 304 stainless steel, both directly by the comparative method and indirectly by a combination of flash thermal diffusivity and differential scanning calorimetry. In the case of the comparative measurements, we have used both Armco iron and Pyroceram 9606 references. The Armco iron conductivity-vs-temperature function is felt to be well known because of the large existing data base on this material [5]. In previous measurements reported by us, the flash diffusivity-derived conductivity in the temperature range 0–400°C agreed with the recommended conductivity function within the limits of the estimated error,  $\approx \pm 7\%$  [2, 6].

# 2. EXPERIMENTAL TECHNIQUES

### 2.1. Comparative Thermal Conductivity

The comparative technique employed in our laboratory has been discussed previously [1, 2, 6, 7]. Metal sample and reference diameters were typically in the range 3-5 cm and sample thicknesses were  $\approx 2.5$  cm. Pyrex 7740 and Pyroceram 9606 stack elements had similar diameters but were  $\approx 1.6$  cm thick. Type K thermocouples were inserted in grooves, 0.038 cm deep, milled into the top and bottom faces of the ceramic and glass referen-

ces, while for the metal stack elements, the thermocouples were sheathed in ceramic tubes and inserted in two holes drilled from the cylindrical surface to the axis of the sample, parallel to, and  $\approx 0.32$  cm from, the top and bottom surface. Usually a stack  $\Delta T \approx 50^{\circ}$ C was maintained, although lower stack  $\Delta T$  values were sometimes required with stacks made up entirely of high-conductivity metal elements. After stabilization of all stack temperatures at a measurement temperature, 5–10 sets of thermocouple readings were obtained. Typically the measurement points were  $\approx 50^{\circ}$ C apart.

# 2.2. Flash Diffusivity

The thermal diffusivity was measured using the laser flash technique originally described in Ref. 8. In this method, a disk-shaped sample, 1.27 cm in diameter, was heated with a short laser pulse and the subsequent temperature rise of the opposing sample surface was monitored with an infrared detector system as the data were stored in a transient recorder. Data analysis was performed using a method which allows simultaneous correction for finite laser pulse width and heat loss from the sample surfaces [9]. Although no absolute references exist for this measurement technique, measurements of AXM-5Q POCO graphite were performed and compared with the results of a round-robin study for this material [10]. Our measurements agreed with the round-robin data within the  $\pm 5\%$  uncertainty limits obtained from that study. The results of measurements on Pyroceram 9606 discussed above also support the claimed accuracy of the technique.

# 2.3. Specific Heat

The specific heat was measured using a Perkin–Elmer DSC2 differential scanning calorimeter. This instrument was interfaced with an HP1000 laboratory computer which performed all instrument control, data acquisition, and data analysis. The specific heat was measured in a continuous temperature scan using NBS sapphire references within measured accuracy limits of  $\pm 2\%$ . The general measurement technique is reviewed in a recent article by the NBS [11]. Temperature calibrations were performed using NBS melt standards at the scan rate of the sample measurement to minimize the effect of possible temperature lags. In addition, slow scan rates were used (20°C/min for 304SS and 10°C/min for Inconel) to minimize temperature lag. Repeat measurements, performed for both materials, agreed within  $\pm 1\%$ . As a check for possible sample size and mass dependence, two samples of the 304SS were measured (64 and 340 mg) over different temperature ranges. The data agreed within +2% in the temperature overlap range.

## **3. RESULTS**

## 3.1. Results for 304 Stainless Steel

The stainless-steel samples were cut from a piece of stock material. An additional piece of the same stock was analyzed quantitatively for elemental composition. The analysis results shown in Table I are consistent with the specifications for AISI 304 stainless steel given by Bogaard [4]. The flash diffusivity sample had a diameter of 12.7 mm and a thickness of 1.62 mm. Two different flash diffusivity runs were made with this sample and the results for the diffusivity,  $\alpha$ , are shown in Fig. 1. Also shown in Fig. 1 is a recommended thermal diffusivity function for the 304 series stainless steel, with an error bar on the reference curve point at 225°C representing the suggested  $\pm 10\%$  accuracy of the reference curve [12]. The thermal expansion of a sample of 304 stainless steel was also measured in order to correct the thickness used to calculate the conductivity. The error bars on the experimental diffusivity points represent the +5%accuracy which we feel characterizes our flash diffusivity measurements for materials with diffusivities in the range shown. Except at low temperatures, it is evident that the temperature variation of the data indicates a slope,  $d\alpha/dT$ , somewhat smaller in magnitude than that of the suggested reference diffusivity. However, all of our data fall within the  $\pm 10\%$  reference curve accuracy, so this conclusion is somewhat tentative. Some of the data used to derive the reference curve has a slope similar to that characterizing our data [12]. The measured specific heat data for both 304 stainless steel and

by the X-Ray Fluorescense Techniqu			
Element	Weight (%)		
Fe	70.1		
Cr	18.7		
Ni	8.3		
Mn	1.6		
Si	0.6		
Others	≤0.2		

Table I. Composition of the 304 a. 1 a



Fig. 1. Thermal diffusivity of 304 stainless steel as measured by the flash diffusivity technique. The error bars represent the  $\pm 5\%$  error associated with the measurement. Two different runs were made with the same sample. The error bar on the CINDAS curve represents the  $\pm 10\%$  suggested accuracy of that curve [12]. Note that the  $\alpha$  axis spans the range  $0.03 \le \alpha \le 0.06$  cm<sup>2</sup> · s<sup>-1</sup>.

Inconel 718 are shown in Fig. 2. Also shown for stainless steel are recommended values from the compilation by Touloukian and Ho [12], with the error bar indicating the  $\pm 5\%$  uncertainty which they associate with the recommended values. Our DSC data fall within this  $\pm 5\%$  uncertainty band.

Comparative measurements on 304 stainless-steel samples were made with four different sets of references: Armco iron, Pyroceram 9606, Pyrex 7740, and Inconel 718. All of the references except the Pyrex were supplied by Dynatech R/D Co. The Pyrex references were cut from stock supplied by the Corning Glass Co. The results for the thermal conductivity, k, are shown in Fig. 3, together with the conductivity data derived from the flash diffusivity, specific heat, and density data. Also shown is a recommended conductivity function (filled circles) given by Bogaard [4], with the error bar on the second data point of the suggested reference curve representing the  $\pm 4\%$  estimated accuracy of this curve [4]. The  $\pm 5.4\%$  error bars for the flash diffusivity-derived conductivity represent the RMS error for independent errors of  $\pm 5\%$  in the diffusivity,  $\pm 2\%$  in the specific heat, and  $\pm 0.5\%$  in the density. An error analysis for the comparative system indicates that the comparative measurements should be accurate to



Fig. 2. Specific heat of 304 stainless steel and Inconel 718 determined from differential scanning calorimetry data. The CINDAS recommended 304SS function is from Ref. 12 and the Brooks Inconel 718 data are from Ref. 14. Note that the  $c_p$  axis spans the range  $0.4 \le c_p \le 0.6 \text{ kJ} \cdot \text{kg}^{-1} \cdot ^{\circ}\text{C}^{-1}$ .



Fig. 3. The 304 stainless-steel conductivity as measured by the comparative technique and by flash diffusivity/differential scanning calorimetry. The recommended curve is from Ref. 4.

 $\approx \pm 7\%$  if the reference conductivity used in the data analysis is known to  $\pm 5\%$  [2]. The curves shown for the comparative results are least-squares fits to the primary data as discussed in Ref. 2. The RMS deviation of the data from the fitted curve is generally in the  $\pm 0.5\%$  range, and hence the data scatter does not make a significant contribution to the overall uncertainty associated with a comparative measurement. As a result, we have omitted plotting the data points for clarity.

An examination of the results in Fig. 3 shows that all of the experimental results are in good agreement among themselves and also in good agreement with Bogaard's recommended conductivity function except for the comparative data obtained with Inconel 718 references. The observed discrepancies of these data ( $\approx 12\%$ ) stimulated further examination of the conductivity of this material, as discussed below. For the data obtained with Pyrex 7740 references, a Pyrex conductivity slightly different from the Dynatech recommended function was used as discussed in Ref. 2. The diffusivity-derived conductivity function appears to have a somewhat different shape than either the comparative or the recommended conductivity functions, but within the error limits of all of these functions it is not possible to say whether this difference is real or not. The variation in the conductivities derived from diffusivity and comparative measurements is qualitatively similar to the variation shown in Fig. 1 between the measured and the recommended thermal diffusivities.

## 3.2. Inconel 718 Results

In order to resolve the discrepancy in the comparative data on 304 stainless steel obtained with Inconel 718 references we undertook a series of additional measurements using Inconel both as a sample and as a reference in comparative measurements and as a sample in flash diffusivity measurements. In initial measurements with a fused silica sample, we obtained comparative results in excellent agreement with the TPRC recommended conductivity function with both Armco iron and Pyroceram 9606 references. With Inconel 718 references, the fused silica-measured conductivity in the range 0-400°C was high by a fractional amount, consistent with the corresponding 304 stainless steel-vs-Inconel 718 data. Measurements were then made with Inconel 718 and Armco iron, using each first as a sample and then as a reference. The results of a determination of the ratio of the Armco iron to the Inconel 718 conductivities are shown in Fig. 4. Also shown in Fig. 4 is the ratio of the Dynatech recommended conductivities [7]. The determination of the conductivity ratio function from comparative data is discussed in Ref. 6. This function is determined solely from the measured thermocouple readings and stack



Fig. 4. The Armco iron-to-Inconel 718 conductivity-ratio function as measured with a Dynatech-supplied Inconel sample with Armco references (S = Inconel) and an Armco sample with Inconel references (S = Armco). The plotted points represent basic data, while the curves are derived from least-squares fits to these data. The recommended curve was calculated from the Dynatech suggested reference curves for these materials [7].

element thicknesses, and thus it does not depend on any assumptions about the conductivity function for the reference elements. The two experimental curves differ from each other by about 5%, and they both differ from the recommended curve by approximately 12%. The Dynatech recommended conductivity function for Inconel 718 [7] appears to be derived from data obtained by Tye et al. in a Dynatech axial rod system [13], as the data in Ref. 13 and the Dynatech reference curve [7] are in close agreement.

In order to determine whether the discrepancies discussed above were caused by compositional effects in the Inconel 718 specimens, we fabricated new specimens from stock supplied by the Sandia Glass Shop. The results of a compositional analysis of both the Dynatech and the Sandia Inconel 718 are given in Table II. An examination of this table shows no large differences between the two materials and indicates that both are in reasonably good agreement with the compositional analysis of the Inconel

	Weight (%)			
Element	Dynatech	SNL Glass Shop 53.5 17.7		
Ni	52.0			
Cr	18.7			
Fe	18.9	18.5		
Nb	5.4	5.1		
Мо	3.0	2.9		
Ti	1.0	1.0		
Al	0.5	0.6		
Others	≤0.1	≤0.1		

Table	II.	Co	mposit	ion o	f Inconel	718	Determined
	by	the	X-ray	Fluo	rescence	Tech	inique

718 samples used by Tye et al. [13]. The specific heat and thermal diffusivity of samples cut from this new stock are shown in Figs. 2 and 5, respectively. The dashed curve and points in Fig. 2 represent adiabatic calorimetry data reported by Brooks et al., with the error bar on that curve representing the reported  $\pm 1.7\%$  spread in the data [14]. Our specific heat data agree with those of Brooks et al. within the uncertainty



Fig. 5. Thermal diffusivity of Inconel 718 measured by flash diffusivity with samples of two different thicknesses.

associated with each data set,  $\pm 1.7\%$  for Brooks et al. [14] and  $\pm 2\%$  for our data. For the flash diffusivity measurements, two different runs were made with samples of thicknesses 0.199 and 0.103 cm to check the reproducibility of the data. As can be seen in Fig. 5, no sample thickness dependence was measured within the error limits of the technique.

The results of all the conductivity measurements are shown in Fig. 6, together with two suggested Inconel conductivity functions. The Dynatech recommended curve appears to be a smooth curve derived from the data of Tye et al. 13, and the McElroy et al. curve is from data reported by McElroy et al. [15].

Examination of Fig. 6 shows that all of our data fall appreciably below the Dynatech reference curve and that there appears to be some variation in the results for the Dynatech- and the Sandia-supplied materials, especially at low temperatures. We suspected that the specimens' thermal history was playing a role in the observed conductivity variations, and this led us to conduct a study of the dependence of Inconel 718 thermal conductivity on heat treatment parameters.

Inconel 718 ages through a complex, thermally induced process of precipitate nucleation and growth. Optimum mechanical properties such as



Fig. 6. Thermal conductivity of Inconel 718 measured by the comparative and flash diffusivity techniques. Measurements were made on samples supplied by Dynatech (Dyn) and the Sandia Laboratories Glass Shop (GS). Two different runs were made on GS Inconel with Pyroceram 9606 references to check for repeatability. The Dynatech recommended curve is from Ref. 7 and the McElroy et al. recommended curve is from Ref. 15.

strength, creep rupture, and hardness are achieved through controlled heat treatments. In contrast, 304 stainless steel is not an age-hardenable alloy. The metallurgical condition of as-received Inconel is frequently unknown, as in this study. As shown in Fig. 6, the measured thermal conductivities of both Dynatech and Sandia Glass Shop Inconel not only disagree with the Dynatech calibration curve but differ from each other. The source of the discrepancies was sought in the metallurgical condition of the alloy.

The Inconel from the Sandia Glass Shop was solution-treated for 1 h at 1065°C in a static argon atmosphere and quenched in water. The heating should have dissolved any precipitates that might have been present, and the quench should have retained the alloying elements in solution. Measurements were then conducted in air in the thermal comparator, using Pyroceram 9606 references. The thermal conductivity of the alloy in the solution-treated condition at 25°C (Fig. 7) coincides with the value of McElroy et al. [15] (Fig. 6) at the same temperature to better than 1%. The Inconel was then brought to 620°C in 1.5 h and aged *in situ* for 22.5 h while the conductivity was monitored continuously. During the last 17.5 h of this period, the conductivity was stable within  $\approx \pm 0.7\%$ . The aging treatment at 620°C should have resulted in the formation of some



Fig. 7. Thermal conductivity of Inconel 718 determined by comparative measurements with Pyroceram 9606 references. The heating part of the cycle (squares) begins with the alloy in the solution-treated condition. The cooling part (circles) represents the alloy in an aged condition. A second-order equation was fitted to the k-vs-T cooling data (see text). The solid line is Dynatech's calibration curve [7]. A value for the solution-treated alloy found from flash diffusivity is also shown.

precipitates. The sample was then raised to  $720^{\circ}$ C in 1 h and aged further for 21 h to cause further precipitation. It was then cooled to room temperature in steps, the conductivity being measured at each step. A secondorder equation was fitted to the cooling data:

$$k = 11.45 + 1.156 \times 10^{-2}T + 7.72 \times 10^{-6}T^2 \tag{1}$$

in which k is in  $W \cdot m^{-1} \cdot K^{-1}$  and T is in °C.

The repeatability of measurement was tested at 620 and 720°C. At each temperature approximately 60 data points were taken after temperature stability was assured. The mean coefficient of variation (COV), or relative standard deviation, at these temperatures was 0.8%. The pre- and post-aging treatment values of the conductivity at 25°C were 10.59 and 11.74 W  $\cdot$  m<sup>-1</sup>  $\cdot$  K<sup>-1</sup>, respectively. If a COV = 0.8% is applied to each of these values, then the difference between them,  $1.15 \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ , may be considered accurate to 11%. Since the sample, the references, and all thermocouple wiring remained physically undisturbed throughout the cycle, the comparison of the values at 25°C is that much more valid. It seems clear that the metallurgical condition of Inconel 718 affects its thermal conductivity to a significant extent. The Dynatech calibration curve was generated, in part, from data on eight samples of Inconel 718, all of which were subjected to heat treatments only within the normal operating range of the comparator. History and, hence, metallurgical condition were not known [13]. A more detailed study of how thermal history affects the thermal, electrical, and mechanical properties of Inconel 718 is under way [16].

## 4. CONCLUSIONS

The measurements on 304 stainless steel confirm Bogaard's suggested conductivity function in the temperature range 0-550°C. The comparative data were obtained with references spanning a wide conductivity range, and with the exception of the data obtained with Inconel 718 references, the maximum data spread was about 5%. All of these data agree well with Bogaard's suggested function. The flash diffusivity-derived conductivity deviates somewhat more but still agrees with Bogaard's function within the joint uncertainties associated with the Bogaard curve and our data. Thus, it would appear that 304 stainless steel is well suited for use as a reference material in comparative measurements. A potential problem is possible corrosion of the stainless-steel reference pieces in certain environments.

The data for Inconel 718 indicate that the thermal conductivity depends on the sample thermal history, yielding variations of as much as 10%. Thus, the use of Inconel 718 for precise comparative measurements is

not recommended. In any case, it is worthwhile to check the conductivity of Inconel 718 references against 304 stainless steel, Armco iron, or Pyroceram 9606 before the Inconel is used for precise work. The recommended Dynatech conductivity function appears to describe Inconel 718 in a particular metallurgical condition.

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