Thermophysical Properties of Pyroceram™ 9606[1]

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ABSTRACT

Pyroceram™ 9606 has been studied extensively over the years by a large number of investigators, mainly to determine whether this material is a suitable candidate for thermal conductivity reference.

This work describes the results of an extensive characterization program aimed to determine thermal conductivity and, in addition to it, thermal diffusivity, specific heat capacity and thermal expansion characteristics over the entire temperature range of use for this material. A comparison between the results of this work and the recently published European round-robin characterization of the same material is presented.

INTRODUCTION

It is a well-known fact that pyroceram™ 9606 is a stable material, used very often as a reference for thermal conductivity measurements, generally for the temperature range from 20 °C to 1000 °C. Little is known, however, or has been published regarding the other thermophysical properties of this material: thermal diffusivity, specific heat capacity and thermal expansion.

This work started by gathering data acquired over a few years time span, obtained on several left-over pieces of pyroceram™. All were purchased from Corning, Inc., USA, but in different years, some of them recently, others going back 10 or 15 years.

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[1]Pyroceram is a trademark of Corning, Inc.

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As the amount of accumulated data increased, so did the authors' interest in exploring more of this material's properties. Thermal diffusivity, specific heat capacity and thermal expansion measurements were performed on several samples from 20 °C to 1000 °C. The results obtained were analyzed to generate thermal conductivity values for the entire temperature range. A comparison with data derived from previous work [1,3] is also provided.

THERMAL DIFFUSIVITY

A total of six samples obtained from five different lots were tested on six different instruments for measuring thermal diffusivity using the laser flash technique. All the instruments were the same model, and had the same technical characteristics, described in earlier publications [4,5]. Three repeated measurements were performed on one sample from each of the first four lots, and on two samples from the fifth lot, from 100 °C to 1000 °C, every 100 °C, in air atmosphere. All the samples were prepared for the tests in the same fashion: they were machined to have the same size (12.7 mm diameter, 3 mm thick, having their faces flat and parallel), and had a thin and uniform layer of gold deposited on both faces, on the top of which a thin and uniform layer of graphite was applied.

The first set of measurements was performed in 1999. Another set followed in 2000, three sets followed in 2001 and one in 2002. The results of the tests, as well as the relative standard deviation of the mean test results are shown in Table 1 for each test temperature.

Table 1. Thermal diffusivity test results

Temp. (°C)	Thermal Diffusivity ($cm^2 s^{-1}$) 1999 Lot 1	2000 Lot 2	2001 Lot 3	2001 Lot 4	2001 Lot 5	2002 Lot 5	Average Values	Relative Standard Deviation of Mean (%)
100	0.01592	0.01608	0.01641	0.01625	0.01623	0.01637	0.01621	0.46
200	0.01322	0.01346	0.01396	0.01329	0.01319	0.01352	0.01344	0.87
300	0.01195	0.01204	0.01181	0.01193	0.01233	0.01212	0.01203	0.61
400	0.01125	0.01139	0.01136	0.01135	0.01106	0.01149	0.01132	0.53
500	0.01066	0.01063	0.01057	0.01036	0.01078	0.01041	0.01057	0.61
600	0.01044	0.01001	0.01031	0.01026	0.00978	0.00994	0.01012	1.01
700	0.00942	0.00955	0.00981	0.00988	0.00975	0.00951	0.00965	0.78
800	0.00911	0.00936	0.00909	0.00946	0.00934	0.00918	0.00926	0.66
900	0.00907	0.00909	0.00917	0.00899	0.00884	0.00876	0.00899	0.71
1000	0.00876	0.00859	0.00858	0.00868	0.00853	0.00967	0.00864	0.39

It is impressive to note that the maximum value of the relative standard deviation of the mean for all test results obtained from all samples at all temperatures is 1.01 %, which is a very small value, considering the fact that six instruments and five lots of material were involved in this comparison study.

A graphical representation of the thermal diffusivity test results is shown in Figure 1.

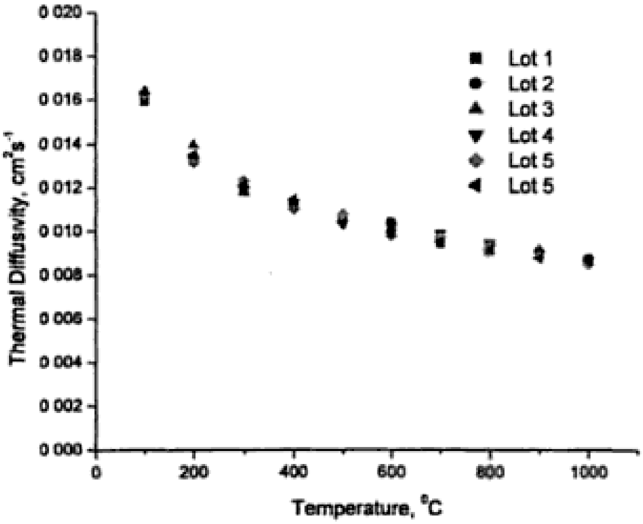

Figure 1. Thermal diffusivity versus temperature (six samples from five lots)

Descriptions of the type of instrument used and a detailed analysis of the instruments' performances were published elsewhere [4,5]. The expanded uncertainty associated with the thermal diffusivity measurements was proven to be 2.10 % for 95 % confidence level [5].

SPECIFIC HEAT CAPACITY

Additional measurements were performed on three of the samples used for thermal diffusivity testing, in order to derive their specific heat capacity values at 100 °C intervals, from 100 °C to 1000 °C. This was performed using the same laser flash thermal diffusivity equipment employed in the first phase of the study. Taking advantage of the multi-sample testing capability of these instruments, for each measurement, a reference material was tested concurrently with each of the pyroceram™ samples (subsequent laser shots were applied at each temperature to the reference material and then to the sample, immediately one after another). Since the time between two measurements was only a few minutes, it was legitimate to consider the environmental conditions in the furnace and the energy profile of the laser, as being the same when the reference material and the samples were tested.

Table 2 shows the results of the specific heat capacity measurements performed, as well as the standard deviation of the mean obtained for each test temperature.

Table 2. Specific heat capacity test results

Temperature (°C)	Specific Heat Capacity (J kg^{-1} K^{-1}) 2001 Lot 3	2001 Lot 4	2002 Lot 5	Average Values	Standard Deviation of Mean (%)
100	902.91	863.71	882.71	882.93	2.42
200	994.38	968.49	1000.76	987.88	2.37
300	1056.28	1098.42	1079.43	1078.04	2.07
400	1072.01	1061.04	1088.45	1073.83	1.81
500	1124.11	1125.01	1132.41	1127.18	0.51
600	1177.19	1181.82	1183.54	1180.85	0.27
700	1182.86	1191.81	1194.97	1189.88	0.52
800	1196.08	1218.74	1196.48	1203.77	1.41
900	1208.62	1193.24	1244.22	1215.36	3.00
1000	1278.98	1211.45	1255.83	1248.75	3.20

A visual representation of the specific heat capacity data versus temperature is shown in Figure 2.

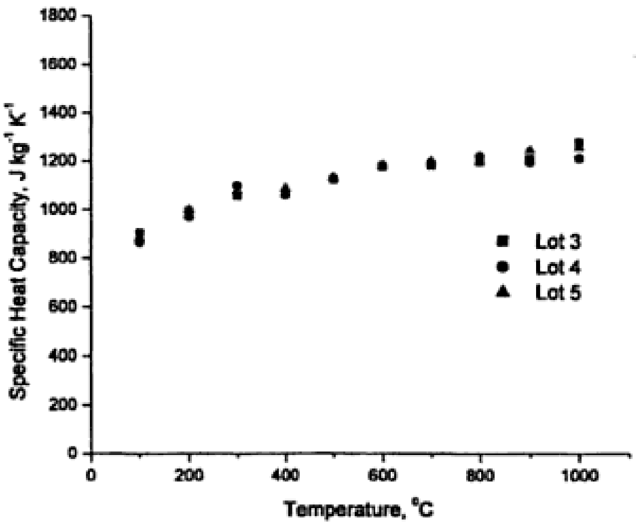

Figure 2. Specific heat capacity versus temperature (three samples from three lots)

All values obtained for the standard deviation of the mean, at all temperatures, were smaller than 3.2 %. Since the three samples tested were obtained from three different lots, it was concluded that the specific heat capacity results obtained are very good and the data is representative for the material.

THERMAL CONDUCTIVITY

For homogeneous, isotropic, single-phase solid materials, thermal conductivity can be calculated from the results of thermal diffusivity, specific heat capacity and density measurements, following the equation:

$$\lambda = \alpha \cdot C_p \cdot \rho \quad (1)$$

where λ is the material's thermal conductivity, α is the thermal diffusivity, C_p is the specific heat capacity and ρ is the density, each measured at the same temperature.

Pyroceram™ 9606 is one of the materials for which it is possible to follow this path for calculating the thermal conductivity. The results obtained in the current study for thermal diffusivity, specific heat capacity and density were further used to derive the thermal conductivity at each test temperature. Table 4 is showing a comparison between the thermal conductivity values obtained in this fashion in the current study, and well known, benchmark data, obtained via direct measurements of thermal conductivity, published in 1966 [1].

Table 4. Comparison between thermal conductivity values: current study versus a previous study [1]

Temperature (°C)	Thermal Conductivity ($W\,m^{-1}\,K^{-1}$) This Study	Previous Study [1]	Relative Difference (%)
100	3.65	3.71	1.59
200	3.38	3.50	3.40
300	3.30	3.34	1.25
400	3.09	3.22	4.13
500	3.02	3.12	3.13
600	3.03	3.05	0.68
700	2.91	2.98	2.46
800	2.82	2.92	3.58
900	2.76	2.88	4.32
1000	2.72	2.85	4.68

Taking into consideration the fact that the current study involved performing measurements on samples from different lots, the conclusion of this comparison was that the results are very close and the material is exhibiting a very consistent behavior, independent of processing variations.

A polynomial curve was further fitted to the thermal conductivity values derived in this study. The temperature dependency of thermal conductivity was found to be best described by equation 2.

two studies are very close to each other and both describe very well the temperature dependency of thermal conductivity of pyroceram™ 9606 for the entire temperature range.

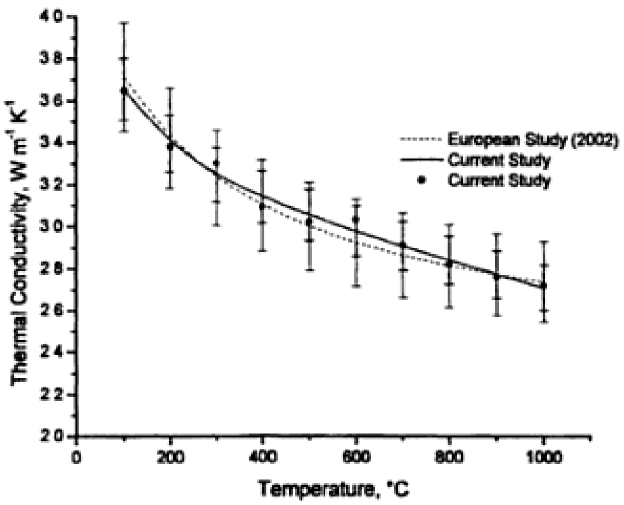

Figure 3. Thermal conductivity: current study versus the 2002 European study

CONCLUSIONS

Samples from five lots of pyroceram™ 9606 were used for thermal diffusivity, specific heat capacity and thermal expansion measurements, with the purpose of exploring the material's thermophysical properties, other than just thermal conductivity. Once again, pyroceram™ 9606 proved to be a good, reliable material, very suitable for thermal conductivity measurements from 20 °C to 1000 °C. In addition to this, the material proved to be very good as a thermal diffusivity reference as well, and showed stable specific heat capacity characteristics.

The results generated by the current study were compared with previously published data on this material. The data also matched very well the results generated by a recently completed European program, and were provided with a smaller uncertainty than those.

REFERENCES

1. Powel, R. W. *et al*, Thermal Conductivity of Selected Materials, National Standard Reference Data Systems, Washington DC, 1966.
2. Salmon, D. R., R. P. Tye, "Pyroceram 9606, a certified ceramic reference material for high temperature thermal transport properties: Part 1 Material Selection and Characterization", presented at the 16th European Conference on Thermophysical Properties, London, UK, 2002.
3. Salmon, D. R., R. Brandt, R. P. Tye, "Pyroceram 9606, a certified ceramic reference material for high temperature thermal transport properties: Part 2 Certification Measurements", presented at the 16th European Conference on Thermophysical Properties, London, UK, 2002.