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THERMAL DIFFUSIVITY, SPECIFIC HEAT, THERMAL CONDUCTIVITY AND THERMAL EXPANSION MEASUREMENTS ON ONE SINGLE DISK SAMPLE

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The Optical Dilatometer (DIL 806) allows for thermal expansion measurement on samples in various shapes. So it can be employed to pair with the Discovery Laser Flash instrument (DLF1600) to determine multiple thermophysical properties such as thermal expansion, thermal diffusivity, specific heat, and thermal conductivity on one single disk sample. An alumina disk sample was measured for these properties from room temperature to 1300^{o}C.

1. INTRODUCTION

Research of new materials requires multiple evaluations of thermophysical properties, such as thermal diffusivity, specific heat, thermal conductivity and thermal expansion. Laser flash thermal diffusivity method, DSC and push rod dilatometry are widely used to measure these parameters. Generally, three samples in different sizes or shapes from the same material are required for the measurements. This causes additional time and material consumption when preparing samples, particularly in the prototype stage that the sample is not available in large sizes. It is highly desirable to simplify sample preparation through the reduction of sample numbers, especially of different shapes and sizes.

The Optical Non-Contact Dilatometer DIL 806 developed at TA Instruments-Waters LLC makes it possible to determine thermal diffusivity, specific heat, thermal conductivity, and thermal expansion on one single disk sample by pairing with a Light Flash thermal diffusivity instrument. In this study, the Discover Discovery Laser Flash instrument DLF1600 is utilized for thermal diffusivity, specific measurement while DIL806 for thermal expansion measurement, then thermal conductivity is calculated with these parameters. The results on an alumina disk sample of 12.7 mm dia. and 3 mm in thickness are displayed.

2. INSTRUMENTS

2.1 Optical Dilatometer DIL 806

A schematic diagram of the optical system of the DIL806 is shown in Fig.1.

The sample is illuminated by the collimated green light beam generated by a Gallium Nitride (GaN) LED. Then the edges of the sample are focused through a telecentric optical system on a high resolution CCD (Charged Couple Device). Therefore, the dimensional changes of the sample are measured by detecting its two edge images on the CCD detector. The resolution of the length change measurement is 50nm.

As shown in Fig.2, both the GaN LED and CCD camera are

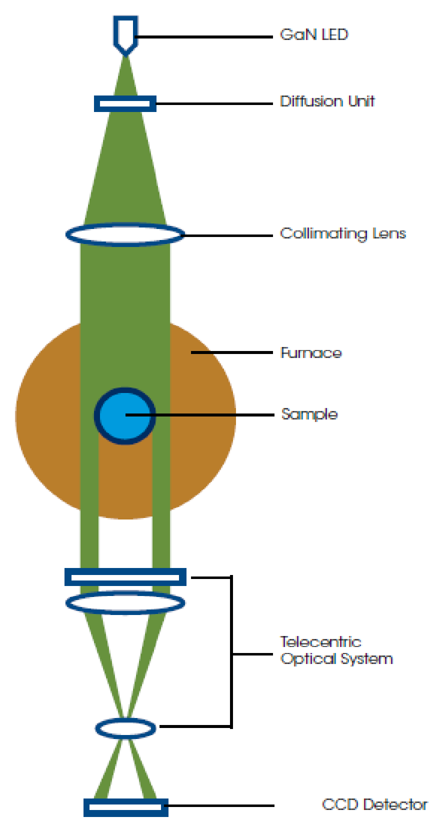

Figure 1 Schematic of the DIL806

well insulated and only the sample is subjected to temperature changes. A plate-shaped structure was designed for the temperature uniformity control and easy-to-loading of samples. The sample is positioned centrally within the wide planar heating element preventing thermal gradients in the lateral direction and making it simple to position the sample. An identical heating element located in the furnace lid is positioned immediately above the sample, thus preventing vertical temperature gradients. The thermocouple is placed inside the sample if a hole is made in it or nearby the opposite side from the light source.

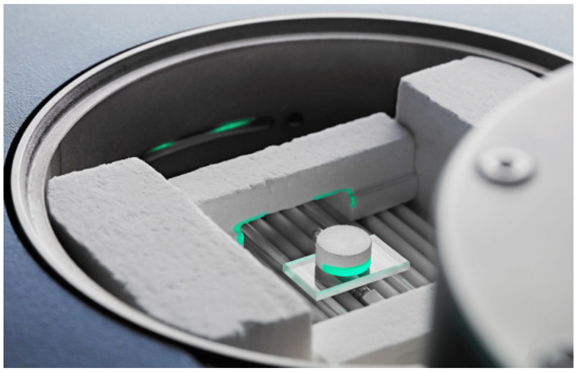

Figure 2 The furnace of the DIL806 in open status

The initial sample length is automatically determined and saved for the subsequent calculation of the linear thermal expansion coefficient once the sample is loaded. Three different models with the temperature range of -150°C to 600°C, room temperature to 1000°C, and room temperature to 1400°C are available with the DIL806. The DIL806 with the temperature range from room temperature to 1400°C is used in the present study.

The verification of the DIL806 was performed on the reference platinum sample with the length of 10 mm [1].

2.2 Discover Laser Flash DLF1600

The instrument used for measuring thermal diffusivity, specific heat is a DLF1600 with an alumina furnace for the sample temperature from ambient to 1600C. The DLF1600 is the current version of FlashLine™ 5000 that was manufactured previously by Anter Corporation. The unit has the capability of testing five or six samples concurrently in one test, side-by-side. The overall performance of the instrument for measuring thermal diffusivity and specific heat were reported previously [2, 3].

3. MEASUREMENTS

The unknown alumina sample of 12.7 mm in dia. and 3 mm in thickness had been sintered at 1600°C so it contains no organic contents. The sample was first measured for thermal expansion with the DIL806 from room temperature to 1300°C. Total three runs were carried out at the same heating rate of 2°C /min.

In the data analysis, the coefficient of thermal expansion (CTE) at the temperature T is defined as:

$$CTE = \Delta L/(Lo\Delta T) \quad (1)$$

Lo is the length of the sample at a reference temperature (25 °C is defined here), ΔL and ΔT is the difference of the length at T and Lo and that of T and 25°C, respectively. Therefore, the term of the CTE used here is the average thermal expansion coefficient defined elsewhere.

The results obtained by fitting the raw data points to quartic polynomial are shown in Fig. 3. The standard deviation of the fit for the individual Run 1, Run 2 and Run is $2x10^{-8}$/K, $1.6x2x10^{-8}$/K, and $1.7x10^{-8}$/K, respectively. The standard deviation of the all raw data points from the overall quartic polynomial fit is $2.6x10^{-8}$/K. The overall fitted data is used as the final CTE results and for calculating density change at different temperature that is used for the calculation of thermal conductivity.

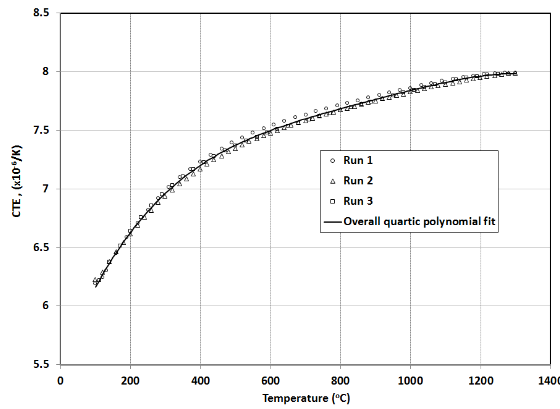

Figure 3 Measured CTE for three runs on the DIL806

After the thermal expansion experiments, the sample was prepared for thermal diffusivity and specific heat measurement by the DLF1600. It was coated with Platinum on both end surfaces along with other two pure alumina reference samples in the same diameter of 12.7 mm and the thickness of 3.2 mm. The thickness of the Pt thin film coated on each sample surface is about 1μm. All three samples then were coated with dry graphite spray to have identical surfaces. One alumina reference sample worked as the specific heat reference and was loaded in the positon No. 1 of the carousel of the DLF1600, and the unknown alumina sample was loaded in the position No. 2 of the carousel. Another pure alumina reference samples was loaded in the positon No. 3 which was for verifying the specific heat measurement. The testing was programed as three laser shots on each sample at each temperature segment. The minimum laser energy was set to avoid high laser energy wearing the Pt and graphite thin films on the sample surfaces.

Fig. 4 shows the thermal diffusivity and specific heat results of the unknown alumina sample from room temperature to 1300°C. Above 700°C, the radiative heat transfer mode in the alumina samples causes the baseline shift of the thermogram which is compensated by the FlashLine™ software.

The standard deviations of thermal diffusivity value at all temperature segments are less than 2%, except at 1200C which is 2.4%. The average standard deviation for all temperature segments is 1.1%. The standard deviation of specific heat value at all temperature is less than 4%, except at 1100°C, which is 4.6%. The average standard deviation of specific heat values for all temperature is 1.9%.

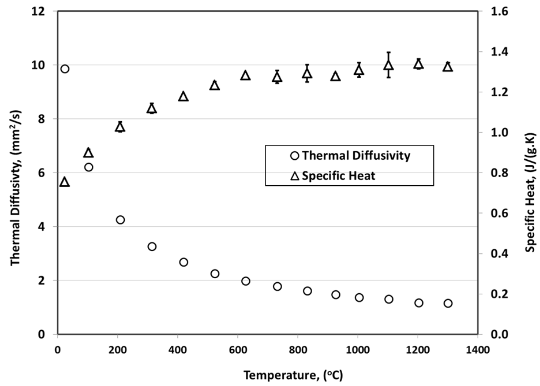

Figure 4 Thermal diffusivity and specific heat results of the unknown alumina sample, the error bars are the standard deviations of the values by three laser shots.

The thermal conductivity was calculated according to the equation below.

$$\lambda = \alpha c \rho \qquad (2)$$

Where λ is thermal conductivity, c specific heat, and ρ density, respectively. The density ρ values at different temperature were corrected with the CTE value generated by the DIL806.

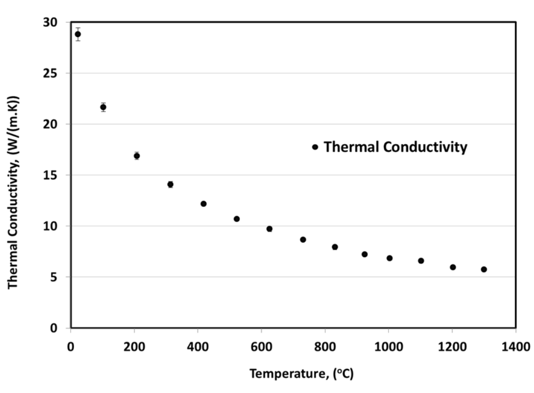

Figure 5 Calculated thermal conductivity of the unknown alumina sample, the error bars are the standard deviations of the values by three laser shots.

Fig. 5 shows the thermal conductivity results of the sample. The standard deviations of all the thermal conductivity values at all temperature segments are less than 3%, except at 1100°C, which is 3.3%. The average standard deviation of thermal conductivity values for all temperature segments is 2%.

5. CONCLUSION

The combination of an Optical Dilatometer and a Laser Flash instrument provides an effective approach for determination of thermal expansion, thermal diffusivity, specific heat, and thermal conductivity on one single disk sample. This should be helpful in the material characterization if the material is not available in quantity. An example of testing an alumina sample up to 1300°C was given.

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