Transactions of the Korean Nuclear Society Autumn Meeting
PyeongChang, Korea, October 25-26, 2007

Performance Test of a Laser Flash Apparatus for a Thermal Diffusivity measurement in a Hot-Cell

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1. Introduction

Many experiments for a thermal conductivity of a nuclear fuel were carried out to observe a change of the thermal diffusivity with the burnup[1]. To evaluate the safety of a fuel rod under a reactor operation, a conductivity change of a high burnup fuel is an important factor in a temperature analysis.

New LFA(Laser Flash Apparatus) was set up in IMEF(Irradiated Materials Examination Facility) for a cold test before a installation in a glove-box. It came from the Netzsche company in Germany. Firstly, it was remodeled for a hot-cell operation, but its installation was changed to a glove-box which is better for a handling. Before we set up the LFA in a glove box, it was tested with several reference samples to obtain a reliability. For the measurement of a diffusivity of a sample, most of all, the design of sample holder is very important because of a thermal loss by a contact.

In this test, we observed a difference with the holder type and sample size.

2. Experimental

2.1 Apparatus

LFA was temporary set up in the front area of the M5 cell for a cold test. It consists of a main system(heater, IR detector, sample loader and laser cable device), a controller, a laser generation, a chiller and a computer. For a gamma shielding, some parts of the electronics were surrounded by a lead panel. Lead block was placed in front of the IR(infra red) detector as well. Sample holder was designed to reduce the contact area with a sample as much as possible.

Heating element of the furnace is a graphite wire, so high pure argon gas must flow continuously under a heating to prevent a oxidation. Furnace is available up to 2,000℃. Its inside material(carrier tube, thermal couple and protective tube) is alumina, so we can control its temperature upto 1,600℃. The measurement accuracy of a thermocouple was 0.1℃. IR detector must be cooled with liquid nitrogen for 30 minutes at least before a measurement. Duration of a liquid nitrogen container is 8 hours.

Laser generation capacity is 40 J of a pulse energy and the source material is Nd:YAG.

All the devices can be controlled by a computer system and a diffusivity detection with the temperatures is automatically carried out by a program setting.

2.2 Measurement of Reference samples

The 3 samples were supplied by LFA manufacturer; Pyroceram, Pure iron and Inconel-600 with certificates and measurement data. We verified our system with these samples at the same temperatures.

Diameter and thickness of the samples are 12.5 mm and 2.5mm, respectively. Three sample holders were supplied; one is 12.5 mm in diameter, second is 9 mm and last is 6 mm. Every holder was used for the samples(12.5 mm of dia.) to observe the effect of the contact area even though two holders were not available.

All samples were coated with carbon to prevent a laser reflection. Detection temperature range is 100℃ ~ 1,000℃ for pyroceram and inconel-600 and 100℃ ~ 700℃ for pure iron by 100℃ steps. Rate of the temperature increase was set at 5℃/min. and detecting was carried out 3 times in each temperature. Pure argon gas was blown into the furnace and sample chamber at 0.7 ml/min. and 0.15 ml/min., respectively. Surface of the furnace was cooled by a chiller and liquid nitrogen was filled in advance. The test took 8~9 hours for each sample. Diffusivity was calculated with method of Cape-Lehman with a pulse correction factor[3].

3. Results

Fig.1, fig2 and fig3 show thermal diffusivities with temperatures. STD is data with using 12.5 mm holder.

Data of the STD holder revealed good results compared to the reference certificates. Maximum error was below 4% in the test of pyroceram. But data of the 6mm and 9mm holders showed deviations. They need to be calibrated.

We observed a difference of the diffusivity in a sample with thickness. It means the measurement range of the LFA system must be evaluated by the thickness of a sample. Another Inconel-600 was used to make

samples(dia = 9mm, t =1,2,4,6 mm). These samples were loaded into the holder of 9 mm.

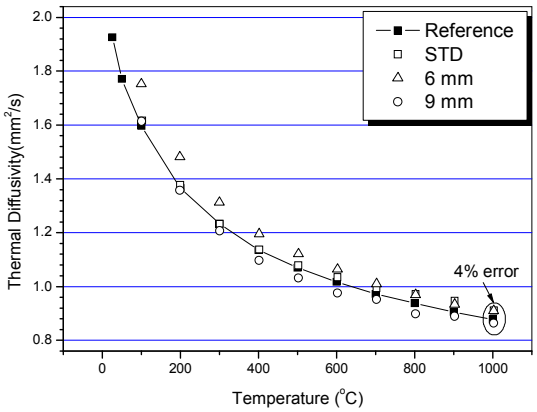

Figure 1. Thermal diffusivity of Pyroceram

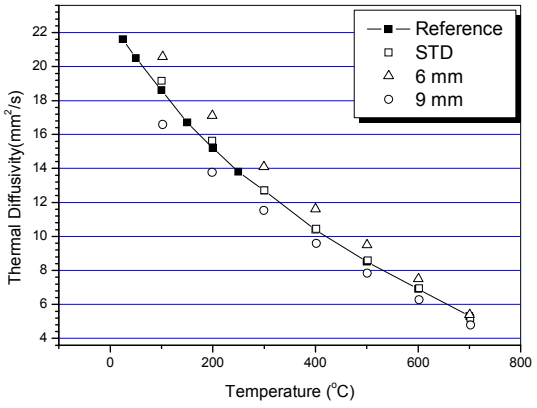

Figure 2. Thermal diffusivity of Iron

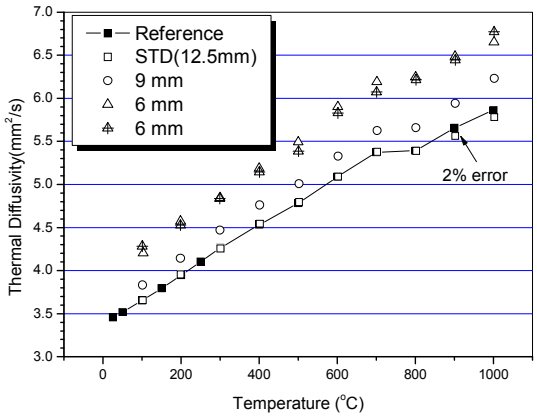

Figure 3. Thermal diffusivity of Inconel-600

Fig.4 shows the thermal diffusivity of Inconel-600 with the thickness. It seems that the diffusivity is higher as the thickness becomes higher. But samples of a 4 mm and 6mm thickness were not placed in the holder correctly due to a holder depth(3.5 mm).

It seems that the thermal loss increases at the side surface of a sample as the thickness increases. There are equations that can be applied to a thermal diffusivity, which neglect a thermal loss at the side surface of a sample. It caused an higher error with a higher thickness. On the other hand, a smaller thickness would cause a dimensional measurement error. So, 2~3 mm of a thickness is better in this test.

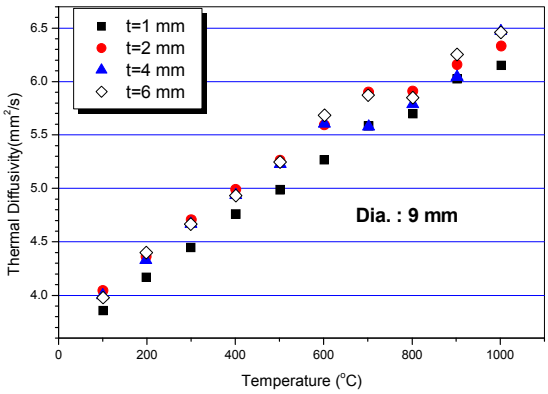

Figure 4. Thermal diffusivity of Inconel-600 with thickness

4. Conclusion

LFA system for the measurement of a thermal diffusivity was set up temporarily. To verify a measurement of the system, three samples were measured with several temperatures. The results revealed a good agreement. We made samples of Inconel-600 with a different thickness, which revealed differences. Especially, thermal diffusivity of both Inconel-600 made by us and supplied from Netzsch company were different. As the thickness became higher, the thermal diffusivity increased due to thermal loss occurring at the side surface of the sample. So, 2~3 mm of a thickness was available to measure the diffusivity in this test.

REFERENCES

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