Analysis of Thermal Diffusivity of Specimens with Sample Holder Design in Laser Flash Apparatus

Heemoon Kim, Dae-Gyu park, Sang-Bok Ahn, Woo-Seog Ryu

Korea Atomic Energy Research Institute, 150 Deokjin-dong, Yuseong, Daejeon 305-353, Korea
hkim1211@kaeri.re.kr

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 measure the thermal conductivity, three parameters such as thermal diffusivity, thermal expansion and density must be measured.

LFA(Laser Flash Apparatus) has been introduced for measurement of thermal diffusivity. To measure the thermal diffusivity, specimen holder design must be considered to reduce the contact with specimen. So, 3 tip-point holder, so called STD holder in this paper, is recommended. But, in the case of fuel specimen with high burnup, 3 tip-point holder is not available to load specimen in hot-cell. Because it is difficult to load fuel specimen with manipulator as well as ceramic fuel would be broken under irradiation, so we use piece of fuel pellet. To load specimen on holder with safe and use cracked or broken specimen, holder with large periphery contact area is needed even though it makes radial heat loss.

In this paper, new specimen holder was made for fuel specimen and compared with STD holder. We observed difference of thermal diffusivity with holders.

2. Experimental

2.1 Specimen holders

STD holder was made by LFA manufacturer and we made new holder for hot-cell as shown in Fig.1. STD holder was designed to reduce the contact area with a specimen as much as possible.

But this holder is only for reference specimen because of diameter of 12.5 mm not for fuel specimen(< 10 mm). Holder was made by high pure alumina and its cover ring was SiC but the cover ring of new holder was also alumina.

2.2 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.

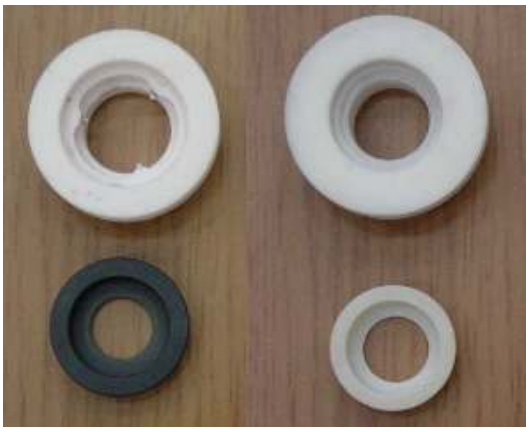

Figure 1. STD holder(left) and new holder(right)

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.

2.3 Measurement of Reference specimens

Three reference specimens were supplied by LFA manufacturer; pyroceram, pure iron and inconel-600 with certificates and measurement data.

Diameter and thickness of the specimens are 12.5 mm and 2.5mm, respectively. All specimens 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. Rate

of the temperature increase was set at 5℃/min. and detecting was carried out 3 times in each temperature. Thermal diffusivities were measured by 100℃ steps. 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 IR detector in advance. The test took 8~9 hours for each specimen. Diffusivity was calculated with model of Cape-Lehman with a pulse correction factor[3].

3. Results

Fig.2, fig.3 and fig.4 show thermal diffusivities of each specimen with temperatures. 'Ref','STD' and 'New' in graphs are reference data, standard and new holders, respectively. Data of iron with STD and new holders revealed good results compared to the reference data. Fig.3 showed that data with new holder were lower than reference data. But data of inconel-600 with new holder showed higher than reference data as shown in fig.4 and maximum error was about 5%.

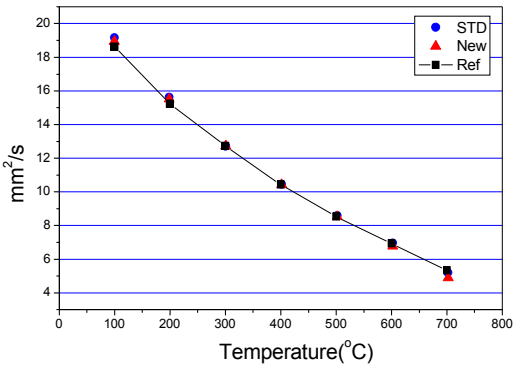

Figure 2. Thermal diffusivity of pure iron

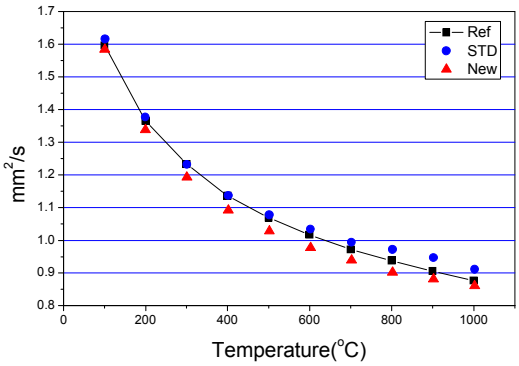

Figure 3. Thermal diffusivity of pyroceram

Eq.(1) is govern equation for LFA system.

$$a = 0.1388\frac{l^2}{t_{0.5}} \qquad (1)$$

Where, 'a' is thermal diffusivity(mm^2/s), 'l' is thickness of specimen(mm) and '$t_{0.5}$' is half time of detector signal(ms). If specimen, temperature and laser condition are same with different holders, only $t_{0.5}$ is important as eq.(1). In the case of inconel-600, $t_{0.5}$ were 148 ms for STD and 134 ms for new one at 1000 °C. So, data difference can be explained by using $t_{0.5}$. On the other hand, however, $t_{0.5}$ were 796 ms for STD and 781 ms for new one at 800 °C in fig.3, data of new holder were lower unlike Inconel-600. It means not only $t_{1/2}$ also heat loss parameter are considered. Moreover, effect of heat loss would be different with sorts of materials.

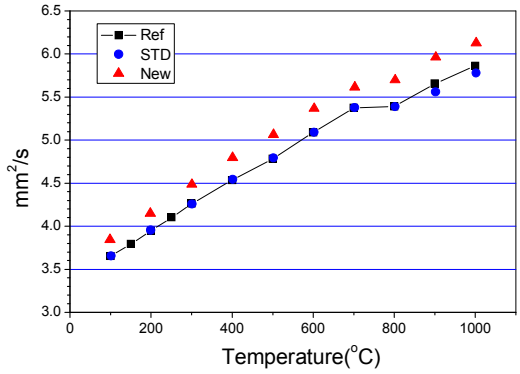

Figure 4. Thermal diffusivity of inconel-600

4. Conclusion

To measure the thermal diffusivity of irradiated fuel, specimen holder must be re-designed to load the specimen with safe in hot-cell. New designed holder showed diffusivity data with difference from STD holder. Data of pure iron test gave good agreement, however, data of inconel-600 and pyroceram tests showed inconsistent behaviors. In this study, heat loss of new holder must be considered but its effect seems to be applied differently with sorts of materials such as ceramics and metal alloy. Additionally, model of Cape-Lehman with a pulse correction factor is needed to be verified in the view point of heat loss.

REFERENCES

[1] J.Nakamura, et al.,'Thermal Diffusivity of High Burnup UO_2 Pellet', IAEA TM on Advances in Pellet Technology for Improved Performance at High Burnup, Japan(1996)
[2] LFA-427 manual, NETZSCH company.(2007)
[3] J.A.Cape, G.W.Lehman, 'Temperature and Finite Pulse-Time Effects in the Flash Method for Measuring Thermal Diffusivity.', Jounal of Applied Physics, vol.34 1909p.(1963)