Heat Loss Effects of Laser Flash Method for Low Thermal Diffusivity Materials

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1. Introduction

The flash method for measuring thermal diffusivity was developed by Parker[1] in 1961. A small sample of cylindrical shape is subjected to a short energy pulse on the front face and the temperature-vs-time history of the rear face is recorded. From this history the thermal diffusivity of the sample can be determined directly. In the laser flash diffusivity technique, there are three critical factors to affect the measured data such as finite pulse time effects, heat loss effects and non-uniform heating effects. The finite pulse time effects are appeared, when the pulse-time(τ) is not infinitesimal but becomes comparable to $t_c \equiv a^2 / \pi^2 t_{1/2}$. It is apparent that the rise in temperature of the rear face will be retarded[2][3]. The Parker model for measuring thermal diffusivity is based on adiabatic and one dimensional assumption. But real experimental circumstance is not fulfilled ideal condition, so that there are always heat loss effects due to heat conduction loss through specimen holder, thermal radiation from specimen's surface and convective gas flow ambient specimen. Heat loss effects are generally appeared in the case for low thermal diffusivity materials and thick sample to examine, because of long time duration to reach maximum rear surface temperature. Such a heat loss effects should be corrected using appropriate manners. Now a day, well known methods are Clark and Taylor rising curve method [4] and Cowan cooling curve method[5]. Cooling curve and rising curve corrections are affected by conduction losses to the holders in addition to the radiation losses from the surfaces. Thus, the errors in the correction procedures are affected by different phenomena and a comparison of thermal diffusivity values corrected by the two procedures is useful in determining the presence or absence of these phenomena. Though two methods of heat loss corrections are based on the use ratio techniques, We don't know which method is more useful than other for a given experimental situation. Then, the purpose of investigation is evaluation and verification of heat loss correction ability experimentally for the future use of determination thermal diffusivity of irradiated UO2 materials.

2. Experimental

The thermal diffusivity analysis equipment in experiment is LFA-427 Laser Flash supplied by NETZSCH. To facilitate heat loss effect, we have to select low thermal diffusivity materials and very thick specimens. And we need to select suitable thickness specimen for reference measured value. The available

material is industrial Inconel-6xx series material, because of easy handling in experiment and machining in precise dimension. Two test specimens are machined to get disk type form with diameter 8.95 mm and thickness 2.0 and 4.0 mm respectively. In addition, in order to enhance thermal conduction loss through specimen holder we enter disk type support plate, which is made of sapphire being optically transparent for laser, between test specimen and specimen holder. The sapphire specimen support plate is fabricated to have form of flat plate to contact tightly to specimen. The experiments have performed under normal test condition not adopting sapphire support plate and with sapphire support plate for each thickness specimen. All the test runs are performed varying temperature from room temperature to 1000 °C and repeat three times each specimen and each testing temperature condition.

3. Results

3.1.Reference value of thermal diffusivity

In order to determine the reference thermal diffusivity of Inconel-6xx material, we use 2 mm thick specimen not adopting sapphire support plate. According to ASTM 1461-01, optimum specimen thickness should be chosen so that the time to reach the maximum temperature falls within the 40 to 200 ms range. The half rise time of specimen is 140.87 ms at room temperature and 84.6 ms at 1000 $^{\circ}$ C. Fig 1. Reveal measured thermal diffusivity value for each calculation model.



Fig 1. Thermal Diffusivity for Various Model(1)

The figure shows that Parker and Cape-Lehman model are almost coincided, but the others model are under estimated. Cape-Lehman model is based on finite pulse time, thermal radiation from specimen's surface and convective gas flow ambient specimen so that this model is generally adopted thermal diffusivity calculating model. But this model don't take account for heat conduction loss through specimen holder. As a conclusion, there are no heat loss by thermal radiation and convection. As a result, we accept Cape-Lehman and Parker value is reference thermal diffusivity for inconel-6xx materials.

3.2 Effects of specimen thickness

To investigate effects of specimen thickness, test was done using 4 mm thickness specimen. Measured half rise time is 571 ms at RT and 339.7 ms at 1000 $^{\circ}$ C. According to ASTM standard, this are not permitted. Fig 2. show measured thermal diffusivity value of 4mm sample thickness for each calculation model. The results are about 9 % smaller value than the thin specimen. But the tendency is consistence with 2mm thickness specimen.



Fig 2. Thermal Diffusivity for Various Model(2)

3.3 Effects of sapphire support plate

In order to enhance thermal conduction loss through specimen holder, sapphire specimen support plate is located between specimen and specimen holder. Fig 3. reveal the effect the sapphire support plate. Generally well known fact is if there is heat loss effects, then rear surface thermo-gram is distorted and as a result the thermo-gram is shifted to front. Resultant is over estimated.



Fig 3. Diffusivity change between standard and sapphire support plate condition for Parker model

3.4 Correction capabilities of Clark- Tailor model

Fig 4 and Fig 5 are plots of the corrected diffusivity value using Clark-Tailor model for specimen thickness 2 mm and 4 mm. Corrected value are accordance with

standard test condition. But measured value are estimated less than other calculation model, e.g., Cape-Lehman, Cowan.





4. Conclusion

Inappropriately selected sample thickness will not only cause unnecessary frustration for the experimenter, but also can be a major source of error in the measurement. Clark-Tailor model is superior to other heat loss correction model, if only heat loss of thermal conduction through specimen holder exist. Since the thermal diffusivity is proportional to the square of the thickness, it may be desirable to use different thicknesses in different temperature ranges.

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