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Thermophysical Property Measurement of $ZrSi_2$ at high temperature using Electrostatic Levitation

Hakjun Lee [a], Changhwan Yu [b], Geun Woo Lee [b], Ho Jin Ryu [a*]
[a] *Department of Nuclear & Quantum Engineering, Korea Advanced Institute of Science & Technology, 291 Daehakro, Yuseonggu, Daejeon 34141*
[b] *Korea Research Institute of Standards & Science, 267 Gajeongro, Yuseonggu, Daejeon 34113*
[*] *Corresponding author: hojinryu@kaist.ac.kr*

1. Introduction

Developing new materials that can be used in advanced types of reactors or fusion reactor, the accurate measurement of material thermomechanical properties in high temperature is extremely important. Existing measuring methods for thermomechanical properties of various materials had severe problem that the measurement was affected by the sample-container interface, especially in high temperature. [1,2] To deal with this issue, levitation methods including ElectroMagnetic Levitation (EML), Aerodynamic Levitation, and ElectroStatic Levitation(ESL), which enables the levitation of sample via electric force between electrodes and charged sample.

$ZrSi_2$ is a one of the most promising material to be coated on Zircaloy cladding. $ZrSi_2$ layer is powerful preventer against the oxidation of the cladding, when it is coated. However, it was always difficult to measure the thermophysical properties of hot $ZrSi_2$ with container, even though it should be utilized in high temperature. In this set of experiments, ESL device was used to measure the thermophysical properties of high temperature $ZrSi_2$ on partial liquid state and high temperature solid state.

2. Experiment

In this section, the procedures including preparation of $ZrSi_2$ sample and experiment operation is explained.

2.1 Sample Preparation

Sample which is used in ESL device have two conditions. First condition is proper mass and second condition is round shape. If the sample is too heavy or too light or it has irregular shape, the location control system of ESL cannot maintain the stable position of the sample.

Bulk chunk of Zr (Alfa Aesar, 3% Hf) and Si (Alfa Aesar, 99.99%) are cut to be in appropriate mass ratio of $ZrSi_2$. That bulk sample is once melted in arc melter (24 torr Ar atmosphere, 10~15kV), and then it is divided into 30~38mgs pieces. Each pieces are melted again in arc melter of same condition, which ensures proper mass and the spherical shape with high homogeneity of the sample.

2.2 Levitation

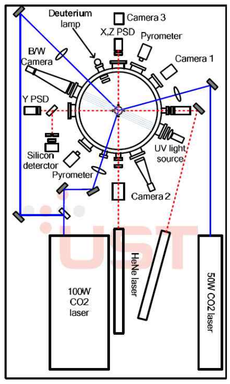

Fig. 1. ESL System Simplified

In order to maintain the stable position of levitated sample, two PSD (Position-Sensitive Detector) will determine x, y, z coordinate of the sample by catching its shadow made by imaging UV.

Fig. 2. Levitated $ZrSi_2$

Position data is sent to the desktop in real time, and then by LabVIEW™'s PID Algorithm according to the following equation, the voltage in the electrode is determined.

$$V(t) = K_c\left(e + \frac{1}{T_i}\int_o^t e\,dt + T_d\frac{de}{dt}\right) \tag{1}$$

K_c, T_i, T_d are P gain / I gain / D gain, which are empirically determined or real-timely adjusted by the operator, and e is the error between real position and aiming point. [3, 4]

During the levitation, the temperature of the sample is measured by two pyrometers (1.55, 1.60 μm) in real time. Simultaneously, contour data of the given sample is imaged so that volume can be measured. However, the contour data is reliable only when the sample is similar enough to sphere.

3. Results

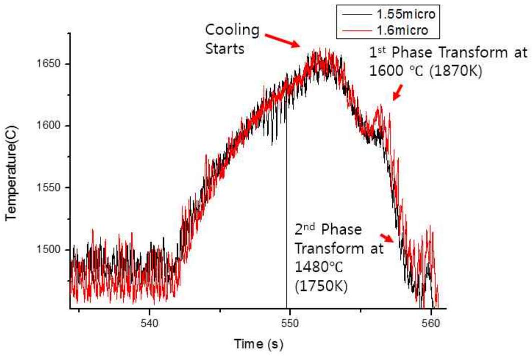

Fig. 3. Time-Temperature Curve of molten $ZrSi_2$

It is obvious that the left region which the temperature is almost constant, represents the melting point (in this case, phase transform point). After the heating laser (CO_2, total 150W) shutdown and natural cooling took place, the temperature did not simply fall down quickly. It showed a bit of 'hesitation' to fall, and the point is phase transform points. In that temperature region, the temperature values are averaged to determine the phase transform point in Fig 3.

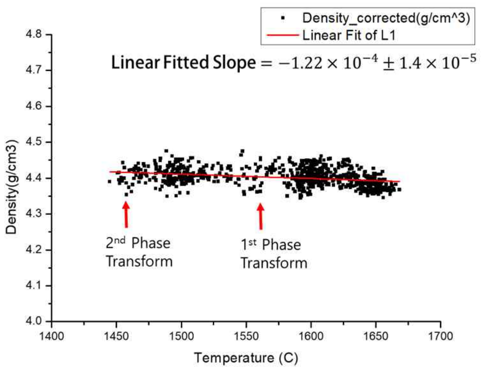

Fig. 4. Temperature – Density Curve of cooling $ZrSi_2$

From calculated volume data, the temperature-density curve is acquired. Each points are pointed every 6ms (milliseconds), so the temperature region which points are concentrated means that phase transform occurred in that region of temperature. After linear-fitting those points and getting the slope, volumetric thermal expansion coefficient is determined by following equation.

$$\frac{1}{V(T)}\frac{\partial V}{\partial T} = -\frac{1}{\rho(T)}\frac{\partial \rho}{\partial T} \qquad (2)$$

Table I: Acquired Thermophysical Property of $ZrSi_2$

	Theoretical Data	My Data
Phase Transform Point	1890K(1620C) 1730K(1460C)	1870K (1600C) ±6K 1750K (1480C) ±13K
Vol. Ther. Expansion Coefficient at MP (per K)	-	2.77 E-05 (about 62% of Zr)
Density At MP(g/cm3)	No Data In Room Temp, 4.80	4.40

Theoretical Data [5]

4. Conclusion

Despite the difficulties on precise measurement of thermomechanical properties of hot metals, ESL experiment suggested a reliable new method to determine the thermomechanical properties like density, phase transform points including melting point, and thermal expansion coefficient, etc. As a result, some of the properties of $ZrSi_2$ is successfully measured using ESL.

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