Volumetric Dilatometer Using Buoyancy under High Pressure Gas

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An innovated volumetric dilatometer was developed using buoyancy under high pressure gas. The advantage of this volumetric dilatometer is independence of the number and shape of samples, which is the characteristic highly demanded among industrial fields. An aluminum block as sample with the volumetric dilatometer was placed in the HIP chamber using argon gas as pressure medium to examine the voltage output change with respect to the pressure and temperature change of the HIP chamber. The voltage output change corresponding to sample weight change caused by argon gas density change was monitored by this volumetric dilatometer.

[volumetric dilatometer, van der Waals' equation of state, AC potential method, HIP, buoyancy]

1. Introduction

Since a HIP treatment chamber is surrounded by a pressure vessel, densifying or sintering behavior which influences the mechanical properties of samples can not be observed directly without sensors inside the chamber.

As a sensor, a dilatometer is used for the monitoring the behaviors with respect to temperature and pressure of the chamber. A conventional dilatometer consists of two graphite rods to examine linear thermal expansivity of one sample. This method has a few disadvantages. The pressure of the graphite rods may act as an uni-axial hot press. It may prevent samples from free thermal expansion and be difficult for measuring the densification of encapsulated samples correctly. In the industrial applications, this method is capable for only one sample, and not suitable for complicated shape samples [1].

A volumetric dilatometer was developed using buoyancy under high pressure gas by Ishizaki et al. [2]. The advantage of this volumetric dilatometer, on the other hand, is that the volumetric dilatometer is independent of the number of samples, therefore all samples are evaluated at once as an average, which is a greatly demanded characteristic from industrial HIP applications. This method is also applicable for complex shaped samples and can detect capsule fracture during capsule-HIPing, so that excess HIP treatment is suspended at that moment. This is useful for industrial applications.

2. Concept

Argon is commonly used for HIPing process, and has large density under high pressure in typical HIPing conditions (refer to Fig.1). If samples are in high pressure gas and change their volume through densification or thermal expansion, for instance, the buoyancy that the samples gain changes with respect to the volume change. This concept can be explained by the following

Archimedes' equation:

$$\Delta W = g \cdot \rho(T, P) \cdot \Delta V \quad (1)$$

where ΔW is weight change of the samples, g gravitational acceleration constant, $\rho(T, P)$ gas density as a function of its temperature and pressure and ΔV volume change of the samples. In the conditions under 200MPa and at 1500K where alumina becomes densified, for example, the density of argon is about 500 kg/m³, which is a half of water under ambient conditions and large enough to provide ΔW for the sample in accordance with ΔV . This ΔV can be calculated by using Equation (1), if a certain sensor to detect ΔW is installed inside a HIP chamber.

Fig.1. Relationship between pressure and density on argon at different temperatures, calculated based upon van der Waals' equation on argon gas [3].

Figure 1 shows the relationship between density and pressure of argon at various temperatures and is plotted by using van der Waals' equation for 1 mole as:

$$\left(p + \frac{a}{V_m^2}\right)(V_m - b) = RT \quad (2)$$

where p is pressure, V_m molar volume, R gas constant, T temperature, a the parameter for van der Waals force and b the parameter named excluded volume characteristically dependent on gases. The two parameters, a and b , of the gases which can be used as pressure medium for HIPing process are given in Table 1. If xenon or krypton is employed as pressure medium for HIP treatment, the sensitivity of the dilatometric measurement is improved.

Table 1. Parameters, a and b , of van der Waals' equation for argon, krypton and xenon [3]. Since krypton and xenon have higher density than argon, it is better to employ them for higher precision measurements. Note that the three gases are all recyclable.

gases	$a / \text{N} \cdot \text{mol}^{-2}$	$b \times 10^{-3} / \text{m}^3 \cdot \text{mol}^{-1}$
Ar	0.136	0.0322
Kr	0.232	0.0396
Xe	0.417	0.0513

3. Weight Measurement System

The dilatometer has a balance bar which suspends a sample holder from its one end and is connected to a cantilever as the pickup electrode by a connecting rod at the other end. The cantilever is situated between two micrometer heads as outer electrodes in a certain distance (refer to Fig.2).

The three electrodes mentioned above constitute the weight change detector based upon AC potential method (refer to Fig.3). This method is simple as well as precise, and its principle is expressed as:

$$\Delta V_p = \frac{\Delta y}{L} V_a \quad (3)$$

where ΔV_p is AC potential change of the pickup electrode, Δy position change of the pickup electrode from the grounded outer electrode surface, L distance between the inner surfaces of the outer electrodes and V_a applied alternative voltage to the outer electrodes [4]. The sample weight change, ΔW , is given from dilatometric measurement and the density of a gas at a certain temperature and pressure is provided from Equation (2), thus the sample volume change, ΔV , can be calculated using Equation (1).

One advantage of the micrometer head is that just adjusting thimble of the micrometer heads easily creates

initial distances between the electrodes. Another advantage is that the micrometer is made of the materials with low thermal expansivity, therefore the effect of thermal expansion is minimized. Adjusting L can widen measurement limit, however sensitivity decreases.

Fig.2. Schematic illustration of volumetric dilatometer. By employing the micrometer heads as outer electrodes, it is easy to create the initial clearances between electrodes.

Fig.3. Schematic illustration of the principle of the AC potential method. V_a stands for alternative voltage input (in this study alternative rectangular voltage input $\pm 2.5\text{V}$), V_p voltage output from the pickup electrode. Lower outer electrode (LOE) is the grounded outer electrode upon which AC potential measurement is based.

4. Experimental procedure

To examine the voltage output change with respect to the pressure and temperature change of the HIP chamber, the following experiment was carried out:

The dilatometer with an aluminum block ($41 \times 31 \times 25\text{mm}$, 84.7g) as sample was placed in the HIP chamber. Using argon gas as pressure medium, the HIP chamber was

pressurized to 75MPa, and then heated and held at 600°C for 0.5 hours, which is about 60°C lower than the melting point of aluminum in atmospheric pressure, as the pressure was maintained about 75MPa by means of degassing.

The electrodes were short-circuited each other, and then initial clearance between the electrodes were created in 20 μm .

5. Results

Figure 4 (a) shows the data from the period of pressuring the HIP chamber and the increase of voltage output as pressure of the HIP chamber increases.

Figure 4 (b) shows the data from the period of heating the HIP chamber and the decrease of voltage output as temperature of the HIP chamber increases.

Fig.4. Relationship between HIP chamber pressure and voltage output as only pressurizing (a). And relationship between HIP chamber temperature and voltage output as only heating where the pressure of the HIP chamber was maintained about 75MPa by degassing (b).

6. Discussion

In the HIP chamber, density of argon gas increases as

pressurized, and then the sample, which gains buoyancy, loses its weight. The cantilever of the dilatometer then deforms downward approaching the lower outer electrode and voltage output increases according to the weight loss. Figure 4 (a) shows this phenomenon. The dilatometer can detect the weight change of the sample caused by density change of argon gas as the pressure of the HIP chamber changes.

The voltage output changed dramatically after the pressure of the HIP chamber reached 52MPa and was saturated in 3.1V. This was because the pickup electrode and lower outer electrode are short-circuited, therefore the measurement could not be completed beyond this pressure.

In the HIP chamber where the pressure is maintained about 75MPa by degassing, on the other hand, density of argon gas decreases as heated, and then the sample, which loses buoyancy, gains its weight. The cantilever of the dilatometer then deforms upward and voltage output decreases according to the weight gain. Figure 4 (b) shows this phenomenon. The dilatometer can detect the weight change of the sample caused by density change of argon gas as the temperature of the HIP chamber changes.

The voltage output started scattering after the temperature of the HIP chamber reached 450°C and was saturated in 3.1V when the temperature reached 580°C, therefore the measurement can not be completed beyond this temperature.

This dilatometer to measure weight change of samples is capable for density change of argon gas caused by both pressure and temperature changes of the HIP chamber.

7. Conclusion

An experiment to examine voltage output change with respect to the pressure and temperature change of the HIP chamber was carried out. The voltage output change corresponding to sample weight change caused by density change of argon gas with respect to the temperature and pressure change of the HIP chamber was observed by this volumetric dilatometer.

References

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