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Viscosity Measurements of Molten Sn–Ag–Cu Alloy Using an Oscillating Crucible Viscometer

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In this study, the viscosities of molten pure Sn, a Sn–Pb eutectic alloy, and Sn–Ag–Cu alloys, whose properties such as liquidus temperature were similar, were measured using an oscillating crucible viscometer over a temperature range of 483–668 K. For the Sn–Ag–Cu alloys, measurements were conducted on Sn–0.8Cu, Sn–1Ag–0.7Cu, and Sn–4Ag–0.5Cu alloys (mass%) with various Ag contents. The viscosities of molten pure Sn obtained using a viscometer ranged from 1.2 to 1.6 mPa·s, which were in good agreement with those reported in the literature. Additionally, the viscosity of the molten Sn–Pb alloy closely matched the values reported by T. Ejima, Y. Sato, T. Yamamura, A. Hayashi, and T. Yamazaki, "Viscosity of molten Sn–Pb alloys," *J. Jpn. Inst. Met.*, vol. 54, pp. 1005–1010, 1990, whereas that of the molten Sn–Ag–Cu alloys was similar to that reported by A. Adachi, Y. Ogino, and M. Shiraishi, "Viscosity of molten Sn–Ag–Cu alloys," *J. Jpn. Inst. Met.*, vol. 36, pp. 927–933, 1972. The molten Sn–Ag–Cu alloys also exhibited a temperature dependence similar to that of the pure molten Sn and Sn–Pb alloys. However, no significant changes in the viscosities were observed with varying Ag content. A temperature-dependent viscosity equation was developed for each alloy on the basis of the measured viscosity data.

1. Introduction

In response to the European Union's Restriction of Hazardous Substances (RoHS) directive, Japan advanced the transition from Sn–37Pb solder (hereafter, all compositions are expressed in mass%) to Pb-free solder.(1,2) To support a smooth transition, mass-production technologies must be established for Pb-free solder alloys with properties similar to those of Sn–37Pb alloys within a short period.

Pb-free solder alloys with several compositions, such as Sn–Ag–Cu, Sn–Cu, Sn–Zn, Sn–Ag–Bi, and Sn–Ag–In, have been developed.(3) Recently, Sn–Ag–Cu alloys, which are based on Sn and contain Ag and Cu, have become mainstream Pb-free solders owing to their similarities to Sn–Pb solders in terms of liquidus temperature and reactivity with substrates.

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However, the amount of experimentally measured viscosity data necessary to simulate the production processes of these alloys is currently limited, and the reported values vary across different sources. Although viscosity measurements of solder alloys have been reported by Adachi *et al.*(4) and Tan *et al.*,(5) these studies were conducted under conditions that differ from practical compositions and thus do not provide sufficient data for process simulation. Hashimoto *et al.*(6) measured the viscosity of practical Sn–Ag–Cu alloys using a coaxial double-cylinder rotational viscometer; however, the data obtained lacked sufficient precision, making it difficult to use them for accurate simulations.

In this study, we measured the viscosities of pure molten Sn, a Sn–Pb eutectic alloy, and Sn–Ag–Cu alloys, which share similar properties such as liquidus temperature, using an oscillating crucible (OC) viscometer over a temperature range of 483–668 K. The key development of this study is the simplification of the detection system and analytical procedure for the OC viscometer, leveraging recent improvements in sensor performance and data processing capabilities. We developed a measurement technique that directly determines the oscillation period and logarithmic decrement, eliminating the need for the complex approximations and threshold settings required in conventional methods. This approach enables a concise and intuitive analysis based on the least squares method. Furthermore, using the measured viscosity data, a temperature-dependent viscosity equation was established for each alloy.

2. Experimental Procedure

2.1 Pure Sn alloy samples

Viscosity measurements were conducted using 5N and 7N purity Sn produced by JX Nippon Mining & Metals Co., Ltd., and Sn–37Pb, Sn–0.8Cu, Sn–1.0Ag–0.7Cu, and Sn–4.0Ag–0.5Cu alloys manufactured by Senju Metal Industry Co., Ltd. The chemical compositions of the alloys are listed in Table 1.

Table 1
Compositions of Sn–37Pb, Sn–0.8Cu, Sn–1.0Ag–0.7Cu, and Sn–4.0Ag–0.5Cu alloys.

Component (mass%)	Sn–37Pb	Sn–0.8Cu	Sn–1.0Ag–0.7Cu	Sn–4.0Ag–0.5Cu	Sn–0.8Cu
Sn	62.85	Bal.	Bal.	Bal.	Bal.
Pb	Bal.	0.00076	0.0088	0.0045	0.00076
Ag	0.021	0.001	0.93	3.97	0.001
Cu	0.0007	0.76	0.69	0.49	0.76
Sb	0.02	0.0077	0.021	0.0065	0.0077
Bi	0.0038	0.0021	0.0034	0.0023	0.0021
Zn	0.00	0.00	0.00	0.00	0.00
Fe	0.0008	0.0006	0.0015	0.0006	0.0006
Al	0.00	0.00	0.00	0.00	0.00
As	0.0029	0.00	0.0009	0.0017	0.00
Cd	0.00	0.00	0.00	0.00	0.00
In	0.0011	0.0035	0.0043	0.0007	0.0035

2.2 Viscosity measurement by the oscillating crucible method

The OC method was used in this study. Because the principles of the measurement and the experimental apparatus were previously described in detail,(7) only the important points are described below. An alumina crucible containing a molten alloy sample was suspended and electromagnetically subjected to rotational oscillation. The oscillation was dampened by the internal friction of the molten alloy. Viscosity was determined by factors such as the period of oscillation and logarithmic decrement. The crucible was connected to a mirror block and an inertial disk made of aluminum, and the entire system was suspended using a thin wire made of a platinum-13% rhodium alloy. The mirror was irradiated with a laser light. The reflected light was detected using photodetectors, and the logarithmic decrease was determined. The temperature was measured using a chromel-alumel thermocouple. Low-viscosity helium was introduced into the apparatus. A zirconium sponge was placed immediately under the crucible as an oxygen absorber to remove oxygen from the atmosphere. A crucible made of 99.5 mass% alumina was used in this experiment.

To determine the viscosity by the OC method, the rotation angle θ of the crucible was evaluated as

$$\theta = A\exp\left(-Bt\right)\cdot\sin\left(\omega t + a\right) + C, \tag{1}$$

where A, C, and a denote the initial amplitude (rad), the deviation of the center of oscillation from the center of the photodetectors (rad), and the initial phase (rad), respectively. The period T(s) and logarithmic decrement δ are defined as

$$T = 2\pi/\omega, \tag{2}$$

$$\delta = BT, \tag{3}$$

where ω denotes the angular frequency (rad·s^{-1}) and B is a constant related to decay (s^{-1}). The viscosity η is determined by a method based on Roscoe's equation [Eq. (4)](8) as

$$\eta = \left(\frac{I\delta}{\pi R^3 HZ}\right)^2 \frac{1}{\pi\rho\tau}, \tag{4}$$

where τ, I, R, H, and ρ denote the oscillation period (s), the moment of inertia of the suspension system (kg·m^2), the inner radius of the crucible (m), the height of the sample (m), and the density of the sample (kg·m^{-3}), respectively. Furthermore,

$$Z = \left(1 + \frac{R}{4H}\right)a_0 - \left(\frac{3}{2} + \frac{4R}{\pi H}\right)\frac{1}{p} + \left(\frac{3}{8} + \frac{9R}{4H}\right)\frac{a_2}{2p^2} - \left(\frac{63}{128} - \frac{45R}{64H}\right)\frac{a_4}{4p^4},$$

$$a_0 = (1-\Delta)\left[\frac{(1+\Delta^2)^{\frac{1}{2}}+1}{2}\right]^{\frac{1}{2}} - (1+\Delta)\left[\frac{(1+\Delta^2)^{\frac{1}{2}}-1}{2}\right]^{\frac{1}{2}},$$

$$a_2 = \left[\frac{(1+\Delta^2)^{\frac{1}{2}}+1}{2}\right]^{\frac{1}{2}} + \left[\frac{(1+\Delta^2)^{\frac{1}{2}}-1}{2}\right]^{\frac{1}{2}},$$

$$a_4 = \frac{a_2}{(1+\Delta^2)^{\frac{1}{2}}},\ p = \left(\frac{\pi\rho}{\eta T}\right)^{\frac{1}{2}} R,\ \Delta = \frac{\delta}{2\pi},\ H = \frac{W}{\pi R^2 \rho},$$

where W is the mass of the molten sample (kg). In the actual calculation, δ was derived using the difference between the values obtained from the crucible containing the melt and an empty crucible. η was experimentally determined from the oscillation via iterative calculations of p and Z.

Detailed information on the OC viscometer has been reported by Sato(9) and Nishi *et al.*(10) In the OC method, the sample volume must be estimated on the basis of $h/d > 3.5$, where h is the height of the molten sample in the crucible and d is the inner diameter of the crucible, because the effect of the edge cannot be ignored in Roscoe's equation.(11)

2.3 Calibration of sample temperature

Using the melting point of pure Sn metal (505 K), we performed a temperature calibration based on viscosity measurements obtained via the OC method using a 5N-Sn metal sample. In this method, the apparent viscosity decreases as the sample begins to solidify. Once fully solidified, the apparent viscosity becomes zero. The results are shown in Fig. 1, where the vertical and horizontal axes represent the apparent viscosity and the thermocouple reading, respectively. A sharp decrease in apparent viscosity was observed when the thermocouple reading reached 485 K. This indicated the onset of solidification. This observation suggests that, on the basis of thermocouple readings, the precipitation of the solid phase begins at a temperature approximately 20 K lower than the actual melting point. Therefore, a temperature difference of approximately 20 K was observed between the sample position and thermocouple readings. Because this difference remained constant over the temperature range investigated in this study, the measured temperature was defined as the thermocouple reading plus an offset of +20 K and treated as a temperature-independent correction.

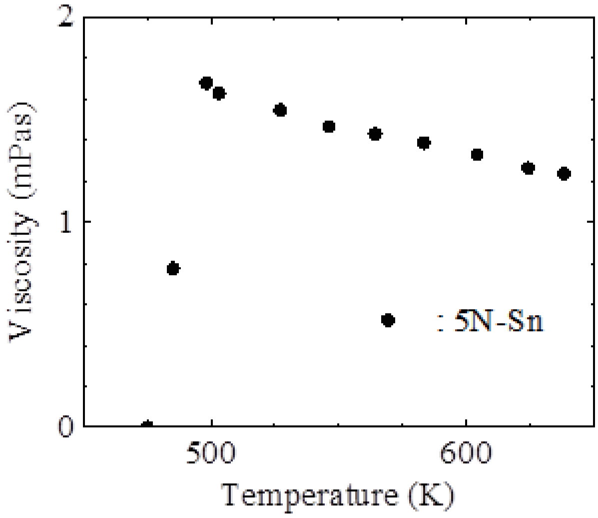

Fig. 1. Temperature calibration using molten pure Sn metal.

3. Results and Discussion

3.1 Viscosities of molten pure Sn metals

Figure 2 shows the measured viscosities of the pure 7N–Sn and 5N–Sn metal samples together with the reported values.(4,12–14) The measured values were close to the reported values and in particularly good agreement with those reported by Adachi *et al.*(4) Although the purities of the two samples differed, the variation between the two measurements was within approximately 2%, indicating good reproducibility.

3.2 Viscosities of molten Sn–Pb, Sn–Cu, and Sn–Ag–Cu alloys

Figures 3–5 show plots of the viscosity of a molten Sn alloy (vertical axis) as a function of temperature (horizontal axis). The error bars at each measurement point represent the standard deviations of the viscosity. Figure 3 shows the viscosity of the molten Sn–37Pb alloy, along with reference values from the literature.(4,12,15) Figure 4 shows the viscosity measurements of the molten Sn–0.8Cu, Sn–1.0Ag–0.7Cu, and Sn–4.0Ag–0.5Cu alloys, together with literature values.(5,16–19) Figure 5 shows the viscosity data obtained in this study for each alloy. In these figures, the viscosities of the molten Sn–37Pb, Sn–0.8Cu, Sn–1.0Ag–0.7Cu, and Sn–4.0Ag–0.5Cu alloys tend to decrease with increasing temperature, consistent with literature values. Furthermore, the viscosity of the molten Sn–37Pb alloy was close to the values reported by Ejima *et al.*,(12) whereas the viscosities of the molten Sn–0.8Cu, Sn–1.0Ag–0.7Cu, and Sn–4.0Ag–0.5Cu alloys were in good agreement with the values reported by Yakymovych and coworkers.(16,18)

Figure 6 shows the analysis of the viscosity data for pure molten Sn metal and the Sn–37Pb, Sn–0.8Cu, Sn–1.0Ag–0.7Cu, and Sn–4.0Ag–0.5Cu alloys (Fig. 5) using an Arrhenius plot. Figure 6 shows that the viscosities of these alloys exhibited good Arrhenius-type linearity over the measured temperature range.

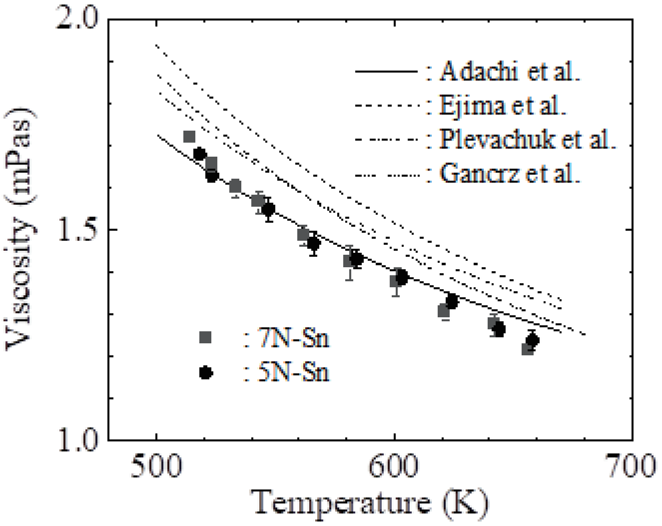

Fig. 2. Temperature dependence of the viscosities of molten pure Sn metals (measured values vs literature values).

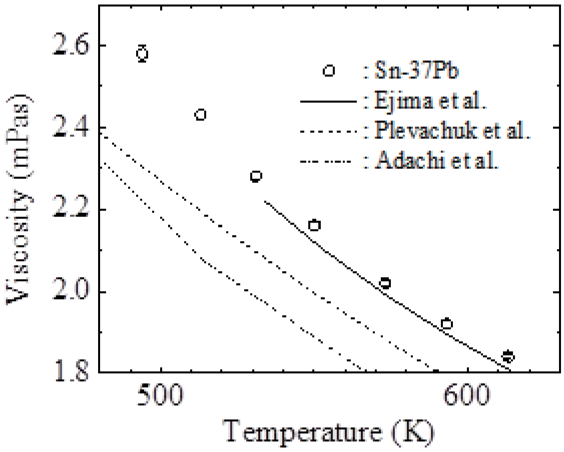

Fig. 3. Temperature dependence of the viscosities of molten Sn–37Pb alloys.

Accordingly, the equations for the viscosities of the molten Sn, Sn–37Pb, Sn–0.8Cu, Sn–1.0Ag–0.7Cu, and Sn–4.0Ag–0.5Cu alloys were derived using Andrade's equation, as shown as (20)

$$\ln\eta = \ln A + B / RT. \tag{5}$$

Here, constant A represents the intercept of the viscosity evaluation equation and directly reflects the magnitude of the viscosity of each sample.(20) By contrast, constant B corresponds to the apparent activation energy for a viscous flow. Using the Andrade's equation, A and B were evaluated; the resulting relations are given by Eqs. (6) and (10). The results corresponding values and the liquidus temperature of each sample are presented in Table 2.

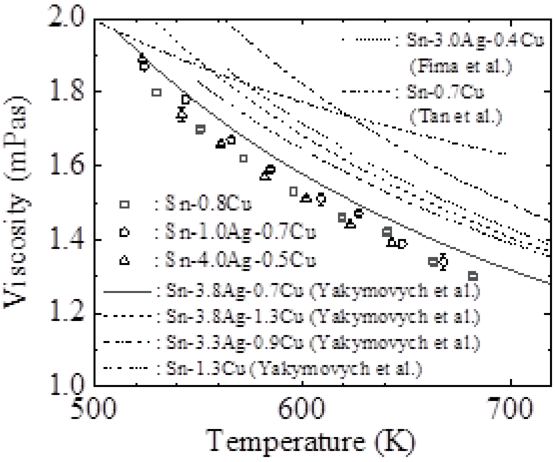

Fig. 4. Temperature dependence of the viscosities of molten Sn–Ag–Cu and Sn–Cu alloys.

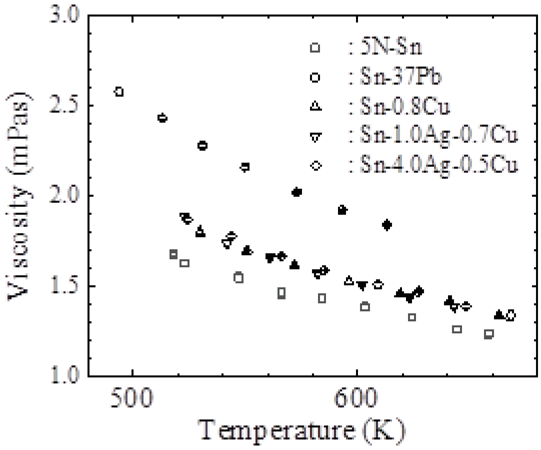

Fig. 5. Temperature dependence of the viscosities of molten Sn–Pb, Sn–Ag–Cu, and Sn–Cu alloys and molten pure Sn metals.

$$\ln \eta_{\mathrm{Sn}} = -0.8635 + 711.9 / T \tag{6}$$

$$\ln \eta_{\mathrm{Sn-37Pb}} = -0.8160 + 872.0 / T \tag{7}$$

$$\ln \eta_{\mathrm{Sn-0.8Cu}} = -0.8564 + 765.3 / T \tag{8}$$

$$\ln \eta_{\mathrm{Sn-1.0Ag-0.7Cu}} = -0.9176 + 810.3 / T \tag{9}$$

$$\ln \eta_{\mathrm{Sn-4.0Ag-0.5Cu}} = -0.9793 + 836.9 / T \tag{10}$$

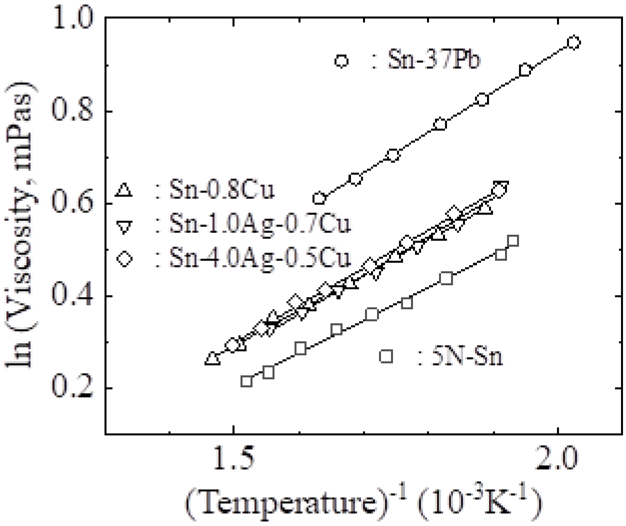

Fig. 6. Viscosities of molten Sn–37Pb, Sn–Ag–Cu, Sn–0.8Cu alloys and molten Sn metal plotted in an Arrhenius-type plot.

Table 2
Liquidus temperatures and B values of Sn, Sn–37Pb, Sn–0.8Cu, Sn–1.0Ag–0.7Cu, and Sn–4.0Ag–0.5Cu alloys for viscosity measurements.

Sample	Temperature range (K)	Liquid temperature (K)	A ($\times 10^{-1}$ mPa·s)	B (kJmol^{-1})
Sn	495–658	505	4.22	5.92
Sn–37Pb	494–613	456	4.42	7.25
Sn–0.8Cu	530–682	502	4.25	6.36
Sn–1.0Ag–0.7Cu	524–668	497	3.99	6.74
Sn–4.0Ag–0.5Cu	523–643	502	3.76	6.96

Hirai proposed a relationship between the *B* value and liquidus temperature.(21) This relationship is illustrated in Fig. 7, where the vertical and horizontal axes represent the *B* value and liquidus temperature, respectively. According to Hirai, the *B* value tends to decrease as the liquidus temperature decreases.

This relationship was confirmed for the molten Sn, Sn–37Pb, Sn–0.8Cu, Sn–1.0Ag–0.7Cu, and Sn–4.0Ag–0.5Cu alloys. A lower *B* value implies that the minimum energy required for viscous flow is reduced, leading to more frequent particle movement within the material and, consequently, a decrease in viscosity. Because the melting points of the samples used in this study did not differ significantly, it was presumed that the *B* values also showed minimal variation.

The relationship between the Ag content and viscosity of the Sn–Ag–Cu alloys was investigated. In Fig. 8, the viscosity and Ag content are plotted on the vertical and horizontal axes, respectively. As shown in Fig. 8, no significant change was observed in the slope of viscosity across various temperature ranges with different Ag contents. This result indicates that the Ag content has virtually no effect on viscosity. Within the practical composition range of

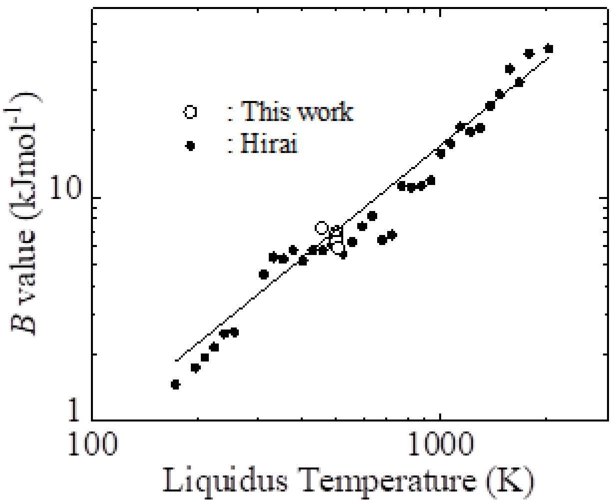

Fig. 7. Relationship between the activation energy and liquidus temperature of molten Sn, Sn–37Pb, Sn–0.8Cu, Sn–1.0Ag–0.7Cu, and Sn–4.0Ag–0.5Cu alloys.

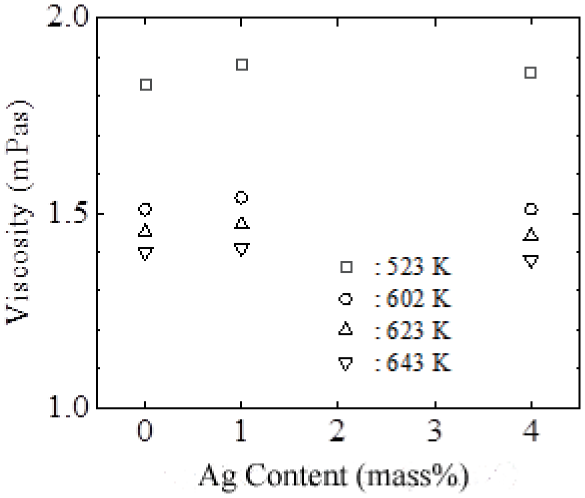

Fig. 8. Ag content dependence of the viscosities of molten Sn–Ag–Cu alloys.

Sn–Ag–Cu alloys, near the eutectic composition of Sn–3.5Ag–0.75Cu, the viscosity remained nearly constant, even at low Ag contents. This result suggests that reducing the Ag content in Sn–Ag–Cu alloys does not cause viscosity issues. This finding is significant from an industrial perspective.

4. Conclusions

Viscosity measurements were conducted via the OC method to obtain the experimental viscosity data of molten Sn–Ag–Cu alloys—required for fluid analysis to improve the precision and efficiency of processing and manufacturing technologies for Pb-free solder alloys. The measured samples included pure molten Sn and the molten Sn–37Pb, Sn–0.8Cu, Sn–1.0Ag–0.7Cu, and Sn–4.0Ag–0.5Cu alloys. The sample temperatures were calibrated using pure Sn. The viscosity measurements revealed that, within the investigated temperature range, all the samples exhibited a clear Arrhenius-type temperature dependence, with viscosity decreasing as the temperature increased. The viscosity of the pure molten Sn metal was in good agreement with the values reported in the literature. Similarly, the viscosity of the molten Sn–37Pb alloy was consistent with that reported by Adachi *et al.*,(4) and the viscosities of the molten Sn–Ag–Cu alloys were similar to the values for Sn–3.8Ag0.7Cu reported by Yakymovych and coworkers.(16,18) Viscosity evaluation equations were constructed on the basis of these measurements to express the viscosity of the molten Sn alloys as a function of temperature. Using these equations, we confirmed the correlation between the liquidus temperature and apparent activation energy for viscous flow (*B* value). In addition, we found that within the practical compositional range of Sn–Ag–Cu solder alloys, the amount of Ag added had a minimal effect on the viscosity.

Appendix

To address the recent surge in helium gas prices, we investigated the possibility of using argon as an alternative to atmospheric gas. First, the logarithmic attenuation rates were compared using He and Ar as the atmospheric gases. Figure 9 shows the obtained results; the vertical axis represents the logarithmic attenuation rate and the horizontal axis represents the temperature. The error bars in the figure represent the standard deviations of the logarithmic attenuation rates. A previous study(22) also showed that the attenuation rates of He and Ar differ by a factor of two. Therefore, the data obtained using He tended to exhibit less variation than those obtained using Ar. Therefore, He was selected as the atmospheric gas for these measurements.

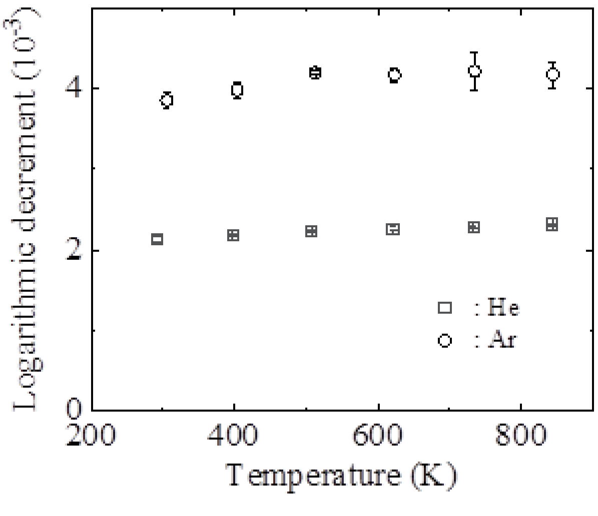

Fig. 9. Temperature dependence of logarithmic decrement.

Acknowledgments

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