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Interdiffusion in Liquid Tin

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The interdiffusion coefficients of Zn, Ag, Sb, Pb, and Bi in liquid Sn were determined using both shear cell and long capillary techniques. These elements were chosen to provide a range of valences and atomic radii, variables that are expected to affect the interdiffusion coefficients. The results indicate that Sb and Ag diffuse in liquid Sn at the same rate as does Sn itself. Bi and Pb appear to diffuse more slowly in liquid Sn than does Sn. Zn appears to diffuse more rapidly in liquid Sn than does Sn itself. These results indicate that the atomic radius is an important variable for interdiffusion in liquid Sn. However, the results for the interdiffusion of Zn, Pb, and Bi, were more scattered than those for Ag and Sb, suggesting that some convective mixing, due possibly to transverse temperature gradients, may be occurring even in capillaries with only 1.5 mm diameters.

Keywords interdiffusion, liquid tin, long capillary, shear cell

1. Introduction

Atomic transport in liquid metals is an important phenomenon influencing the structure and properties of cast materials including metals, polymers, and ceramics. The main transport mechanisms are convective mixing and atomic diffusion in the liquid. Neither of these liquid transport processes is completely understood, and as a result the simulation of atomic transport processes during solidification for the purposes of establishing predictive models cannot be adequately accomplished.

Knowledge of the liquid diffusion process in metals and alloys is not nearly as well developed as that for solid diffusion, and as a result many theories have been advanced to explain the experimental data. The "hole" theory of liquid diffusion[1,2] is equivalent to the vacancy mechanism for solid diffusion and results in the standard Arrhenius equation for the determination of the diffusion coefficient. However, entropy considerations render the "hole" mechanism implausible.[3] "Fluctuation" theories propose that liquid diffusion occurs via the movement of atoms through small and variable distances as a result of local density fluctuations and that such transport occurs by the cooperative motion of five atom groups.[4,5] The fluctuation theory results in a T^2 temperature dependence for the liquid diffusion coefficient.

Several models for liquid diffusion that involve the viscosity of the liquid have been developed.[6-8] In these models, the activation energy for liquid diffusion is that for viscous flow. Other models for liquid diffusion assume the liquid atoms to behave as solid rigid spheres and that diffusion in liquids is more similar to diffusion in gases than in solids.[9-13] Additional theories for liquid diffusion include vibrational models,[14,15] a molecular friction model,[16] free volume theories,[17,18] and an entropy model.[19]

Unfortunately, different experimental investigations produce varying liquid diffusion coefficients, which invariably result in different theories for liquid diffusion. Further, much of the experimental data concerning liquid diffusion were obtained using the capillary reservoir technique, and it is now known that convective flow in the reservoir introduces "lid-driven" flow in the capillary that increases the "apparent" liquid diffusion coefficient.[20] The understanding of the liquid diffusion process remains incomplete.

A research program was conducted with the objectives of determining:

- The techniques best suited for obtaining the most accurate values for liquid diffusion coefficients in unit gravity experiments
- Accurate values for liquid interdiffusion coefficients in tin-base alloys
- The optimum conditions for subsequent definitive microgravity experiments

Sn was chosen as the solvent metal because:

- The solvent self-diffusion coefficient for liquid Sn is well established because six independent investigations (two conducted in microgravity) are in excellent agreement.
- Sn has a low melting point and is easy to work with.

The results for the solvent self-diffusion of Sn are presented in Fig. 1, which illustrates the good agreement of the six studies that were conducted with either the long capillary or shear cell techniques (two of these studies were conducted in microgravity). Also included in Fig. 1 are the results from three capillary reservoir studies that illustrate the problems resulting from the use of this technique.

Zn, Ag, Sb, Pb, and Bi were chosen as solutes because they represent a considerable range of valences and atomic

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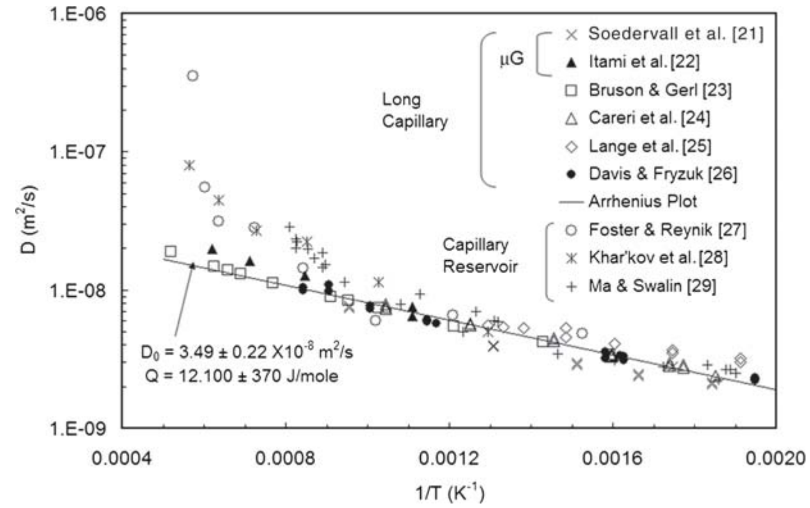

Fig. 1 Experimental results for the solvent self-diffusion of liquid Sn

radii, which are properties that could influence the liquid diffusion coefficient. Diffusion studies were conducted over the temperature range from 400 to 800 °C.

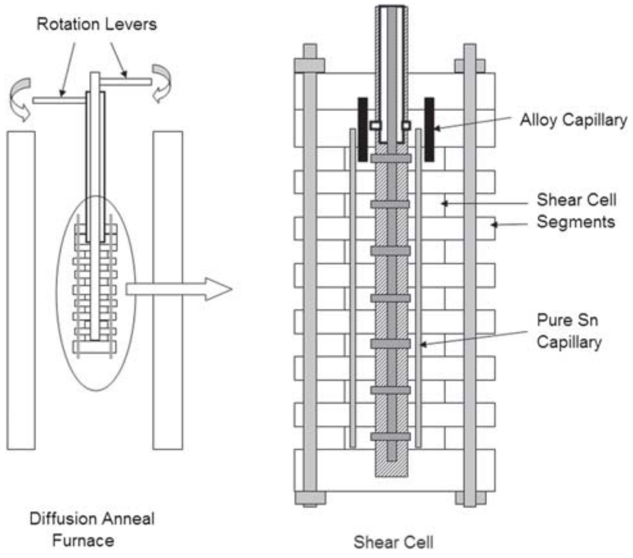

Fig. 2 Shear cell and furnace

2. Experimental

Sn wire, 1.5 mm in diameter obtained commercially, was used to make the pure Sn portion of the liquid diffusion capillaries. Capillary wires of Zn, Sb, Ag, Pb, and Bi alloys were prepared by melting a crucible of alloy containing 5 to 7 at.% of the alloying element under an argon atmosphere in a vacuum furnace in which four 50 mm long, 1.5 mm diam capillary tubes were suspended. After the alloy was molten and had achieved a superheat of about 200 K, the furnace was evacuated and the capillaries were immersed in the melt. Argon was then admitted into the vacuum furnace to fill the capillary tubes. The capillary tubes were then removed from the melt and allowed to solidify. All metals used were 99.99 mass% purity.

Two experimental techniques were used to determine interdiffusion coefficients, shear cell and long capillary. The details of these techniques are discussed in the order mentioned. The shear cell comprised six graphite segments 50 mm diameter and 6.35 mm thick and seven graphite segments 25 mm diameter and 6.35 mm thick. A 14th segment 50 mm diameter and 9.12 mm thick was used to house the alloy portions of the diffusion capillaries. Each of the graphite elements had 1.5 mm holes drilled through them to accommodate the metal wire capillaries. Two graphite end plates, 50 mm diameter, completed the cell segments. The cell was assembled using two type 316 stainless steel bolts, four nuts, and graphite "felt washers" to accommodate thermal expansion. The shear cell is shown schematically in Fig. 2. A diffusion experiment was prepared by feeding two 1.5 mm diameter pure Sn wires through the 12 cell segments (6.35 mm thick), alternating the 50 mm diameter and 25 mm diameter segments, and placing the 14th segment over the protruding Sn wires (Fig. 2). Two alloy capillary wires were inserted into the 14th segment as illustrated in Fig. 2. (The shear cell could accommodate up to four capillaries, but assembly of the cell was extremely difficult with more than two). Attachment of the endplates and rotation levers completed assembly of the cell, which was then inserted into a controlled-atmosphere diffusion anneal tube furnace. The furnace tube was evacuated and filled with argon, and the temperature raised to the diffusion anneal temperature. When the temperature of the furnace and shear cell assembly had stabilized, the 14th (alloy) segment was rotated using the first rotation lever such that the alloy capillaries aligned with the pure Sn capillaries. This technique ensured that freshly sheared alloy and pure Sn surfaces came into contact and also ensured that no diffusion could occur before the diffusion temperature was reached. For alloys with a density greater than that of Sn, the alloy segment of the shear cell was located at the bottom of the cell.

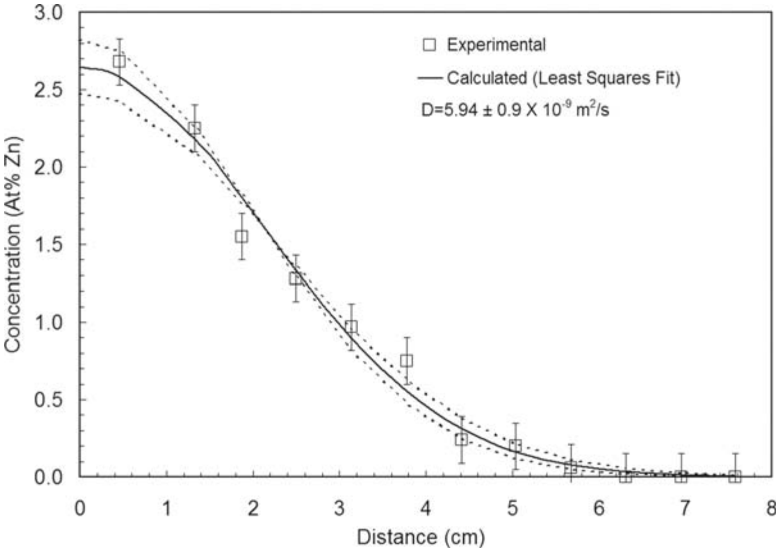

Fig. 3 Shear cell concentration penetration curve for Zn in liquid Sn at 400 °C

Upon completion of the diffusion anneal (3 to 10 h depending on the temperature), alternate graphite segments (25 mm diameter) were rotated using the second rotation lever, dividing each diffusion capillary into 14 pieces. The cell assembly was allowed to cool, removed from the furnace, and disassembled. Each capillary portion was removed from the graphite plates, mounted in plastic, then sanded and polished. The concentration-penetration curve was established by analyzing each portion on a JEOL JSM 5900V (JEOL, Tokyo, Japan) scanning electron microanalyzer by scanning the beam over the entire portion of the capillary. The diffusivity, *D*, was calculated from the least squares fit of the error function equation for diffusion from a finite length of alloy into a semi-infinite length of pure metal given by:

$$C = 0.5C_0\left\{\mathrm{erf}\left[\frac{h+x}{2\sqrt{Dt}}\right] + \mathrm{erf}\left[\frac{(h-x)}{2\sqrt{Dt}}\right]\right\} \qquad \text{(Eq 1)}$$

where *C* is the concentration at distance *x*, *h* is the length of the alloy portion of composition C_0, and *t* is the time of diffusion anneal. A typical concentration penetration curve for the interdiffusion of Zn in liquid Sn diffused at 400 °C for 10 h is presented in Fig. 3.

The absolute accuracy of the concentrations measured on the microanalyzer is normally about ±10% of the amount present, but the relative accuracy is about 5% of the amount present since systematic errors are similar for all compositions and will not substantially affect the concentration-penetration curves. However, for the lower concentrations, the background becomes an increasing source of error as the concentration approaches zero due to the increasing noise-to-signal ratio, and therefore one assumes that the analytical error is constant at ±5% of the largest concentration, ±0.15 at.%. These error limits are shown by the error bars in Fig. 3. *D* is assumed constant over the concentration range, and the calculated value of *D* should be within ±15% indicated by the dotted lines in Fig. 3, since these error limits result in limiting concentration-penetration profiles that pass within the error limits of essentially all of the experimental concentrations.

For the long capillary technique, Al_2O_3 thermocouple wire insulators 75 mm long with 1.5 mm diam holes were used. A diffusion couple was made by inserting a 60 mm length of pure Sn wire into the 75 mm long capillary and placing a 10 mm length of alloy capillary at the top or bottom of the Sn wire, depending on the density of the alloy. The diffusion couple was then annealed for an appropriate time in the diffusion tube furnace.

Upon completion of the anneal, the diffused capillary was solidified, removed from the furnace, and cut into three lengths that were then mounted in plastic. The mounted specimens were sanded, polished, and analyzed on the scanning electron microanalyzer by scanning a 1.5 by 1.0 mm electron beam over the entire width of the capillary sections. Analyses were conducted every 2 mm to provide a concentration penetration curve. A typical long capillary curve for the interdiffusion of Zn in liquid Sn diffused at 400 °C for 10 h is presented in Fig. 4. The error bars in Fig. 4 are again ±0.15 at.% and the dotted lines are *D* ± 15%.

The results for the interdiffusion of Zn, Pb, Bi, Ag, and Sb in the temperatures range 400 to 800 °C are compared with the results for the solvent self-diffusion of pure Sn in Fig. 5 to 9. Due to the variation in the results for the solute diffusion data, activation energies were not calculated.

3. Results and Discussion

Examination of Fig. 5 through 9 shows that the shear cell results agree reasonably well with the long capillary results, but, in general, the shear cell technique appears to give interdiffusion coefficients about 50% higher than the long capillary technique. Possible reasons for this are discussed later in this article.

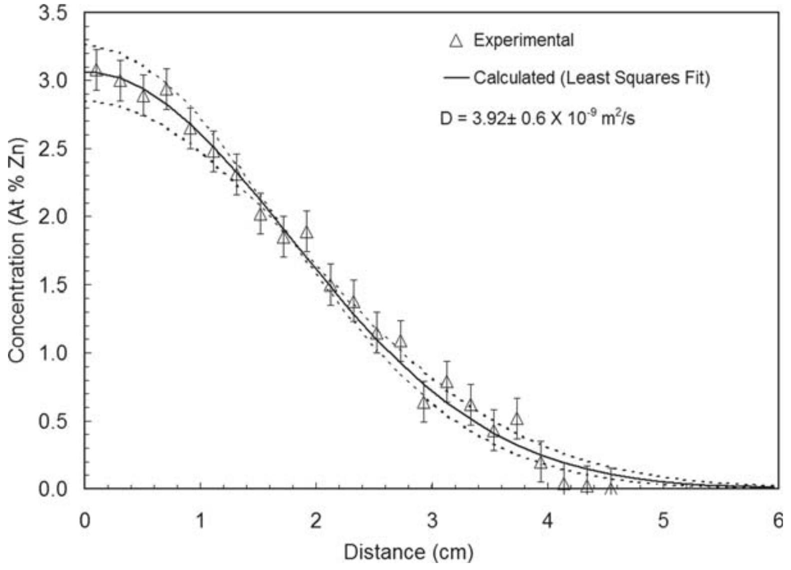

Fig. 4 Long capillary concentration penetration curve for Zn in liquid Sn at 400 °C

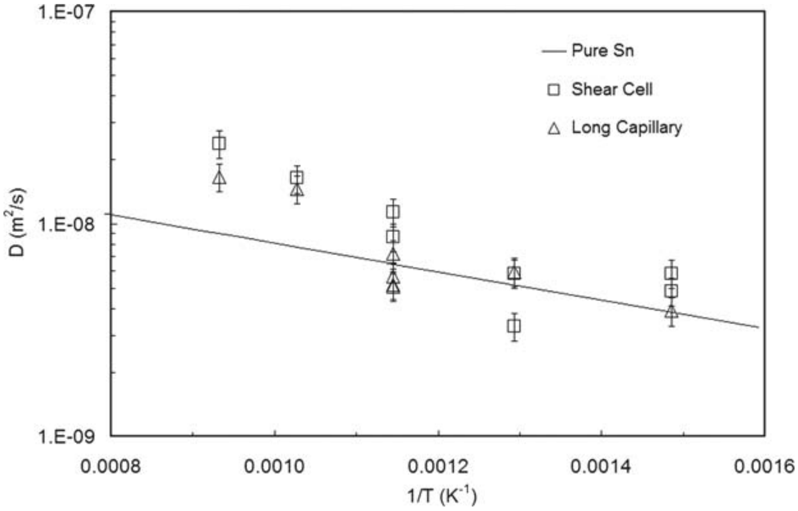

Fig. 5 Arrhenius plot for the interdiffusion of Zn in liquid Sn

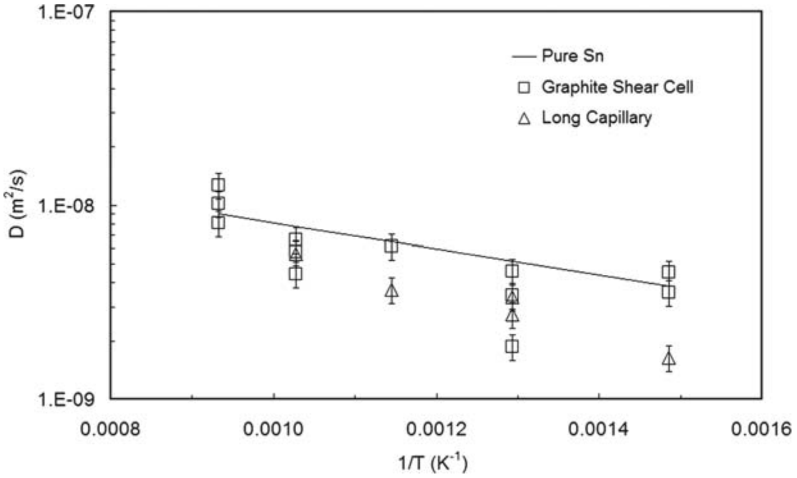

Fig. 6 Arrhenius plot for the interdiffusion of Pb in liquid Sn

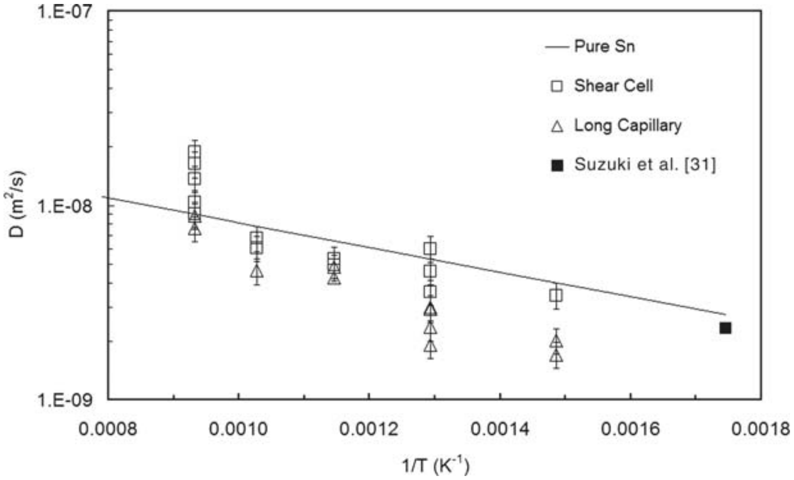

Fig. 7 Arrhenius plot for the interdiffusion of Bi in liquid Sn

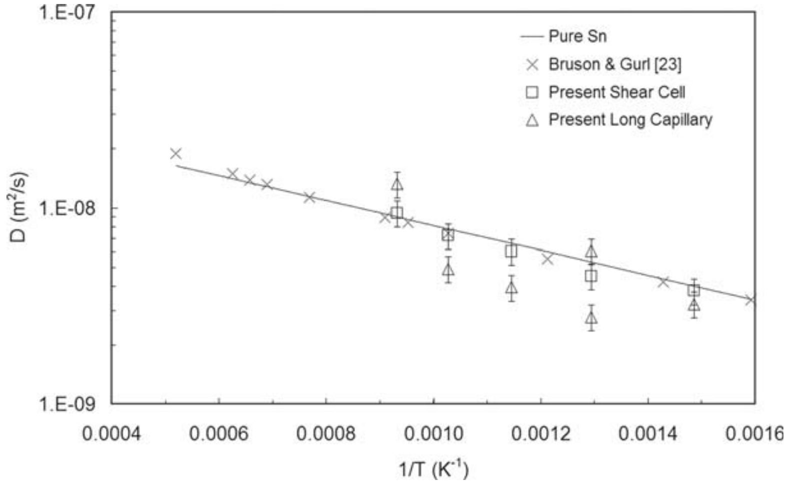

Fig. 8 Arrhenius plot for the interdiffusion of Ag in liquid Sn

Despite the inconsistency of some of the results, it is possible to draw some conclusions concerning the interdiffusion of solutes in liquid Sn. First, Fig. 9 shows that the interdiffusion coefficient of Sb in liquid Sn is essentially the same as that for solvent self-diffusion in liquid Sn. This is confirmed by the results of the present investigation, that of Bruson and Gerl,[23] and that of Smith et al.,[30] which were obtained in microgravity (Fig. 9). Also, it appears that Ag diffuses at about the same rate as does Sn in liquid Sn as confirmed by present investigation as well as that of Bruson and Gerl[23] (Fig. 8).

Although the experimental results for the diffusion of Zn in liquid Sn exhibit considerable scatter (Fig. 5), it appears that Zn may diffuse more rapidly in Sn or at least as fast as does Sn itself. Further, Fig. 6 and 7 indicate that Pb and Bi may diffuse more slowly in Sn than does Sn, but again the inconsistency of the results prevents a firm conclusion.

The obvious variables to consider when attempting to explain interdiffusion in liquids are differences in atomic radius and valence. Values of radius and valence for the elements considered in this section are presented in Table 1 where the solute valences have been adjusted for differences in zero-point energy.[34]

For solute diffusion in solids, the valence difference between solvent and solute (relative valence) is an important factor. For a vacancy mechanism of diffusion, the electrostatic interaction between solute atoms and vacancies alters the local concentration of vacancies near solute atoms and alters the local elastic properties near solute atoms that produce a change in the energy required for the solute to move into the vacancy. The net result is that solutes with a positive relative valence have a lower diffusion activation energy than the solvent and therefore diffuse faster than the solvent, whereas solutes with a negative relative valence have a higher diffusion activation energy than the solvent and therefore diffuse more slowly than the solvent.[34,35] The interdiffusion coefficients in liquid Sn at 400 °C for the elements included in this study are plotted versus relative valence in Fig. 10, which shows that the interdiffusion coefficients do not exhibit the expended trend with relative

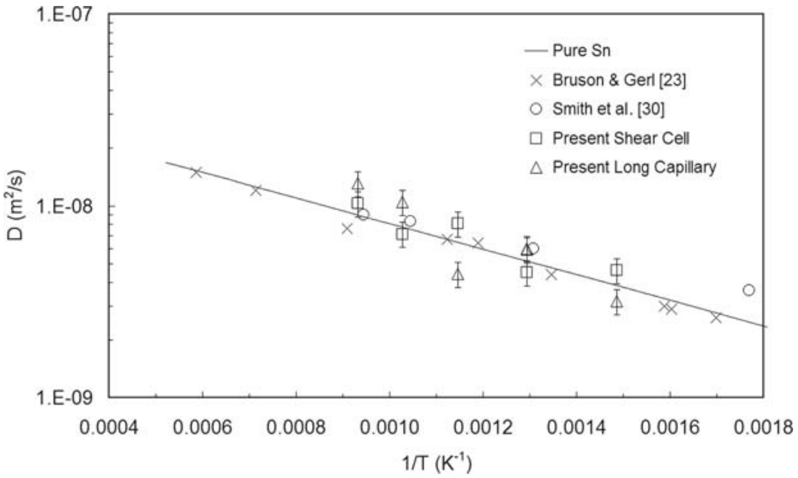

Fig. 9 Arrhenius plot for the interdiffusion of Sb in liquid Sn

Table 1 Atomic radii and valences for solute interdiffusion in liquid Sn

Element	Atomic radius (a), mm	Engel Brewer valence(b)	Valence in Sn corrected for E_0(c)	Relative valence in Sn
Sn	0.141	4	4	0
Zn	0.133	2	2.25	−1.75
Pb	0.175	3	4.25	+0.25
Bi	0.155	5	5.1	+1.10
Ag	0.141	3	2.59	−1.41
Sb	0.145	5	4.74	+0.74

Valences adjusted for zero-point energy. (a) Ref 32. (b) Ref 33. (c) Ref 34

valence. Of particular note are the diffusion coefficients for Ag and Sb, which should show significant differences from the coefficient for pure Sn but which actually have very similar values. If anything, the plot of diffusion coefficients versus relative valence exhibits a trend opposite to that expected. With respect to atomic radius, intuitively it would seem that solute atoms with a smaller radius than the solvent atoms would diffuse more quickly than the solvent itself and solute atoms with a larger radius than the solvent atoms would diffuse more slowly. A plot of interdiffusion coefficient versus atomic radius is given in Fig. 11.

Figure 11 indicates that atomic size may be an important factor in solute diffusion in liquid Sn because Zn with a radius smaller than Sn diffuses more rapidly than Sn, whereas Bi and Pb with larger radii diffuse more slowly than does Sn. Further, Ag and Sb with radii similar to that of Sn diffuse at essentially the same rate as Sn. It must be emphasized, however, that the experimental values for the interdiffusion coefficients of Zn, Bi, and Pb in liquid Sn are not as accurate as those for Ag and Sb.

As indicated previously, while there is reasonable agreement between the shear cell and long capillary techniques, there is a tendency for the shear cell results to be about 50% higher. Two possible sources of Marangoni convection could contribute to mass transfer in the capillaries:

- Surface energy differences caused by concentration and temperature gradients could cause Marangoni convection at the capillary wall. Different surface energies between liquid alloy and Al_2O_3 or graphite could cause different amounts of Marangoni convection.
- Surface energy differences in the vicinity of the interface between graphite segments could cause Marangoni convection in the shear cell, which would be absent in the long capillaries, according to Müller and Müller-Vogt.[36]

The results of Müller and Müller-Vogt [36] also indicate that transverse temperature gradients as small as 0.1 K/mm can cause significant convective mixing in capillaries of 1.5 mm diameter that double the apparent diffusion coefficient. Further, recent solidification results[37] have shown that small transverse temperature gradients can cause convective mixing in capillaries larger than 0.8 mm diameter, although the authors do not give an estimate for the magnitude of the gradients. For the 1.5 mm capillaries used in the present investigation, the larger mass of the shear cell compared with that of the long capillaries could result in different transverse gradients and thus differing amounts of convection even though the stable configuration (heavy element on the bottom) was always used.

Despite the problems in the experimental determination of liquid interdiffusion coefficients, it is apparent that differences in coefficients for different solutes are probably going to be small and quite close to the solvent self-diffusion coefficient. Very accurate determinations of diffusion coefficients are obviously required, and at the present time it appears that a microgravity environment is required. The use of 0.8 mm diameter (or less) capillaries may limit convective mixing,[36] but could cause convection due to increased surface energy forces.

There was no evidence that any significant segregation occurred during melting and solidification of the capillaries. The use of a complicated shear cell, which in itself could contribute to Marangoni convection at the segment interfaces, may not be necessary.

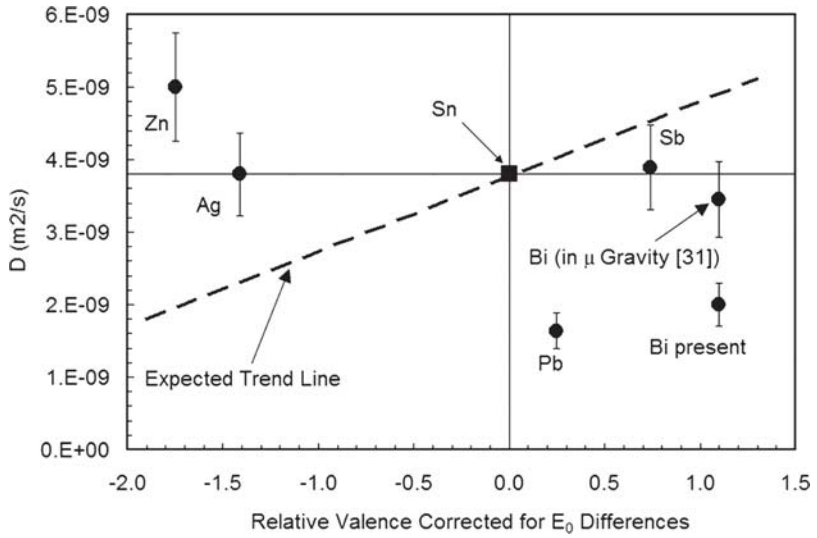

Fig. 10 Interdiffusion coefficients in liquid Sn at 400 °C versus relative valence

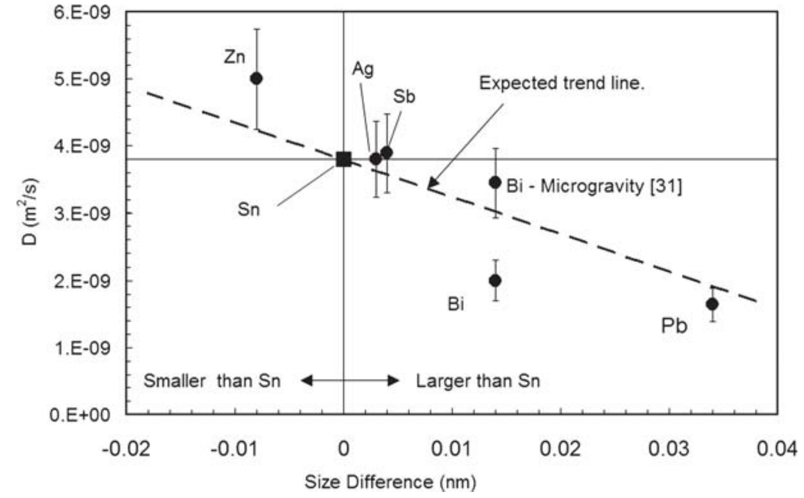

Fig. 11 Interdiffusion coefficients in liquid Sn at 400 °C versus atomic radius

4. Conclusions

- A comparison of the long capillary and shear cell results shows reasonable agreement between the two techniques. However, there is a tendency for the shear cell results to be about 50% higher than long capillary results.
- The discrepancy between shear cell and long capillary results may be due to differences in the amount of convection (Marangoni or otherwise) between the two techniques.
- The long capillary technique is probably as accurate as the shear cell technique.
- Ag and Sb diffuse in liquid Sn at essentially the same rate as does Sn itself. Zn appears to diffuse faster than Sn, whereas Bi and Pb appear to diffuse more slowly than Sn.
- At the present time, it appears that a microgravity environment is necessary to obtain accurate liquid diffusion coefficients.

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