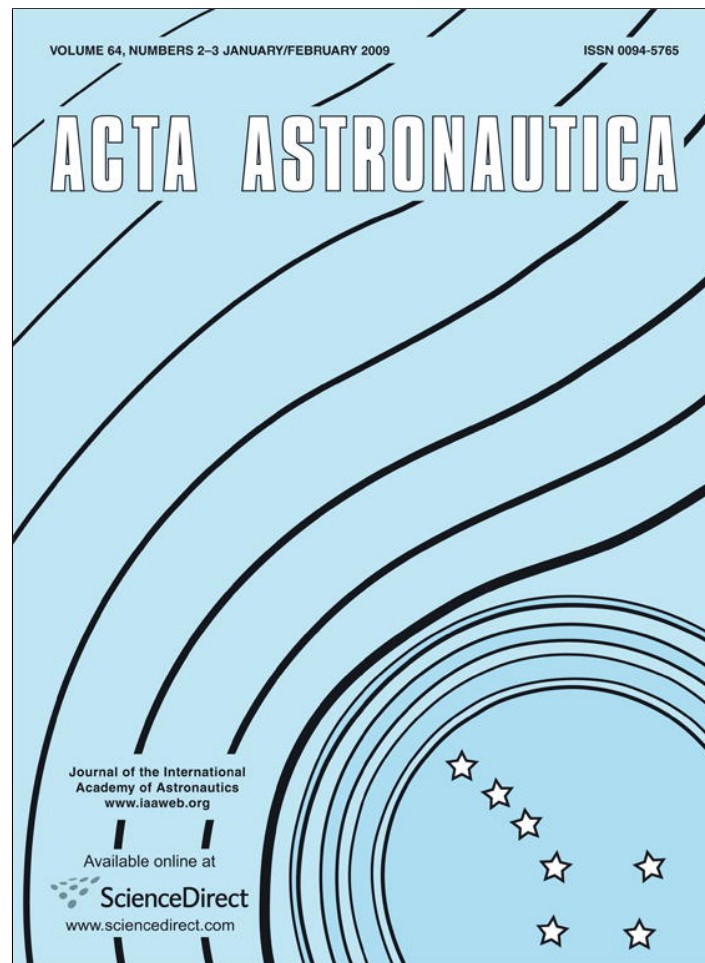


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Acta Astronautica 64 (2009) 256–263

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Analysis of the data obtained from liquid metal diffusion experiments conducted on the MIR space station

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Received 28 February 2008; received in revised form 26 May 2008; accepted 17 June 2008

Available online 8 August 2008

Abstract

Recent liquid metal diffusion experiments onboard the MIR space station have led to the proposal that suppression of g-jitter may change the temperature dependence of the diffusion coefficient from a square law to a linear dependence. It has also been claimed that the diffusion coefficients obtained under isolation mode are significantly lower than their non-isolated counterparts. A thorough reanalysis of the original data leads to three principal findings: (i) a linear temperature-dependence does not emerge when all available diffusion data obtained on MIR in the isolation mode are considered in the analysis. This finding suggests that the $D \propto T$ relation that was shown previously for a number of dilute alloy systems may simply arise because very limited numbers of data were considered for analysis for each system; (ii) the measured diffusion coefficients obtained with g-jitter suppressed are not reproduced in two replicated experiments; and (iii) the diffusion coefficient values are not consistently below the corresponding non-isolated microgravity data. These facts suggest there is an uncontrolled source of experiment variation that is much stronger than the effect of g-jitter on the experiments. On the basis of these new findings, it is proposed that the results from the liquid metal diffusion experiments conducted on MIR do not support the conclusions drawn.

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Keywords: Diffusion in metals; Microgravity; Microgravity vibration isolation mount (MIM); Thermotransport; MIR space station; g-Jitter

1. Introduction

Recent liquid metal diffusion experiments onboard the MIR space station have led to the proposal that suppression of g-jitter may change the temperature dependence of the diffusion coefficient from a square law to a linear dependence [1–6]. It has also been suggested that the diffusion coefficients measured in the isolated mode are significantly lower than Froberg's results [7] and those obtained in the non-isolated mode on MIR [1–4] and on the space shuttle flights (Space

Shuttles Endeavour Mission STS-47 and the Space Shuttle Columbia Mission STS-52) [8,9]. The microgravity experiments on the MIR space station were conducted using the Canadian Microgravity Vibration Isolation Mount (MIM) developed by the Canadian Space Agency (CSA). The MIM was intended to provide the opportunity for: (a) exposing the diffusion couples to the g-jitter, (b) isolating the diffusion couples from g-jitter and (c) subjecting them to a forced vibration superimposed on the isolating state. Unfortunately, it appears that the diffusion data obtained with MIM operating in various modes are unreliable because of serious errors in measuring the processing time and temperatures, and do not support the main general conclusions drawn. What makes the diffusion coefficients

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reported even more doubtful is the magnitude of the errors that occurred during the post-flight analysis of the solute concentration profiles. As pointed out by Garandet et al. [10], benchmark experiments should be thoroughly analyzed in terms of the sources of errors in order to allow an unambiguous conclusion to be reached. This is particularly true in the case of microgravity experiments performed on the MIR space station since it seems unlikely that other researchers will find the opportunity to independently replicate these experiments in the near future. At the time of writing, the results of the microgravity experiments conducted on MIR have been reported in numerous journal papers and conference proceedings by Smith [2,5], Smith et al. [1,6], Herring et al. [3], and Tryggvason et al. [4]. The conclusions drawn based on these experiments have called all other liquid diffusion experiments [11–14] into question but the sources of errors in the MIR experiments have not been adequately characterized. Because of the enormous interest in the subject, it is important that any uncharacterized sources of experimental variation or reasonable alternative explanations for the patterns in the experimental data are brought to light. That is what we have endeavoured to present in this analysis.

The analysis of the original microgravity data obtained on the MIR space station is now presented. One finds that within a set of data obtained under isolation mode, a $D \propto T$ relationship does not emerge when all available diffusion data obtained with g-jitter suppressed are considered in the analysis. Moreover, the measured diffusion coefficients obtained under isolation mode are not reproduced in two replicated experiments and are not consistently below the corresponding non-isolated microgravity data.

2. Sources of data

Data for the analysis was acquired from various sources. These included published journal papers [1–4], conference papers [5,6], thesis [15], and relevant reports including a report entitled “Results of liquid metal diffusion Experiments—Final Report” [16] that has been cited previously in a number of published journal papers by Smith et al. [17,18]. This report is cited here to draw attention to serious problems that were encountered during the space experiments on the MIR space station. This is deemed to be important because the conclusions reached in the published papers by Smith et al. appear to be questionable when one actually considers the scale of the problems reported in [16]. Critical, in many cases, was the availability of the tables in the cited thesis and reports that contained the original

data where it was possible to extract the precise, numerical data underlying the published figures. The numerical values of the diffusion coefficients in particular were considered to be important for the analysis of the temperature dependence of the diffusion coefficients for each dilute alloy system. These data are referred to in the discussion section as “original plotting data”.

3. Results and discussions

3.1. Variation of the diffusion coefficients with temperature in microgravity with g-jitter suppressed

The original proposal that suppression of g-jitter in microgravity may change the temperature dependence of the diffusion coefficient from a square law to a linear dependence was based on the experiments conducted on MIR for a number of dilute binary alloys including Pb–(Au, Ag, Sb), Sb–(Ga, In), Bi–(Ag, Au, Sb), Al–(Fe, Ni, Si), and In–Sb [1–4]. One reason for the lack of confidence in the proposed $D \propto T$ relationship is the relative paucity of the diffusion data that were considered for each system and the fact that in all cases the data appear to fit the linear relationship very well despite the serious problems that were encountered during experiments. Table 1 shows the number of data points that were used to establish the linear dependence for each binary alloy system. For example, for the lead–1 wt% silver diffusion couples, only five data points were considered to demonstrate the $D \propto T$ relationship [1–4]. In Fig. 1, the original plotting data points for the lead–silver system (open circles) are presented. It is seen that the linearity that can be claimed on the basis of these data points appears to be remarkable. The question that was asked was whether the reported $D \propto T$ relation could be explained as arising from the fact that only few data points were considered for the analysis. It was tempting to find out if an alternative relationship could be established when all available diffusion data for the lead–silver system obtained under isolation mode were included in the analysis. It was possible to identify at least two additional data points for the lead–silver system ($D = 2.46 \times 10^{-9} \text{ m}^2/\text{s}$ at 400°C and $D = 3.89 \times 10^{-9} \text{ m}^2/\text{s}$ at 600°C) (black circles) that were obtained under microgravity in the MIR space station with g-jitter suppressed and that were reported earlier by Smith in [5,16]. But in order to begin the analysis, the original plotting data were given the benefit of the doubt, and it was reluctantly assumed that all measured diffusion data reported in the past were valid and sufficiently accurate. It is seen in Fig. 1 that the two additional data points clearly do not fit the expected linear trend but they cannot be discarded since

Table 1
Number of data points selected for each binary alloy to demonstrate the $D \propto T$ relation in microgravity with g-jitter suppressed [16].

System	Number of data points	System	Number of data points
Pb–1 wt% Ag	5	Bi–1 wt% Sb	3
Pb–1 wt% Au	4	Al–1 wt% Fe	2
Pb–1 wt% Sb	4	Al–1 wt% Ni	2
Sb–1 wt% Ga	3	In–1 wt% Sb	4
Sb–1 wt% In	3	Sn–1 wt% Au	2
Bi–1 wt% Ag	2	Sn–1 wt% Sb	4
Bi–1 wt% Au	2		

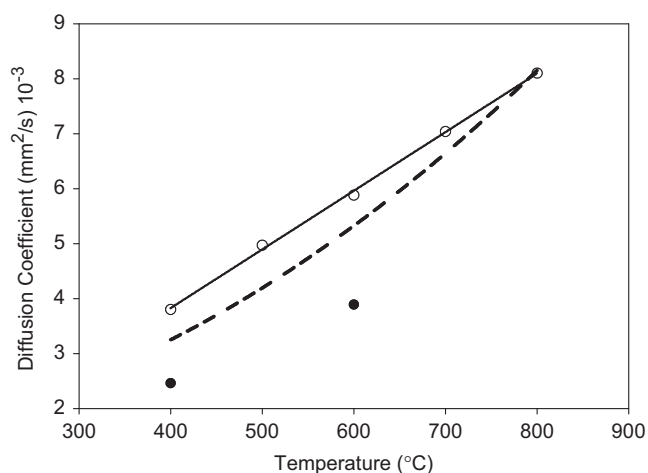


Fig. 1. Diffusion coefficients as a function of temperature for the Pb–1 wt% Ag system in microgravity with g-jitter suppressed. (○) Data points reported in [1–3]. (●) Data points reported in [5,16].

they were considered to be valid in the earlier publications [5,16] and they represent $\sim 30\%$ of the total data available for the lead–1 wt% silver system obtained under isolation mode. It can be seen in Fig. 1 that when one includes these earlier data in the analysis, a non-linear relationship can in fact emerge. This example shows why the limitation of the number of experimental data is so problematic. Clearly more data points help but the burden of proof of a $D \propto T$ relation is on those who propose it.

One of the supporting assumptions in the papers by Smith [2], Herring et al. [3], and Tryggvason et al. [4] has been that the $D \propto T$ relation can be demonstrated for a wide range of dilute binary alloys including lead–1 wt% gold system. But, again, it was found that the $D \propto T$ relation that was proposed for this system might simply arise because only very limited numbers of data were considered for the analysis. Fig. 2 shows the original plotting data (open circles) that have been frequently reported in the past [1,2]. The data set seems to agree better with a simple analytical expression than would be expected from the measurement accuracy. It

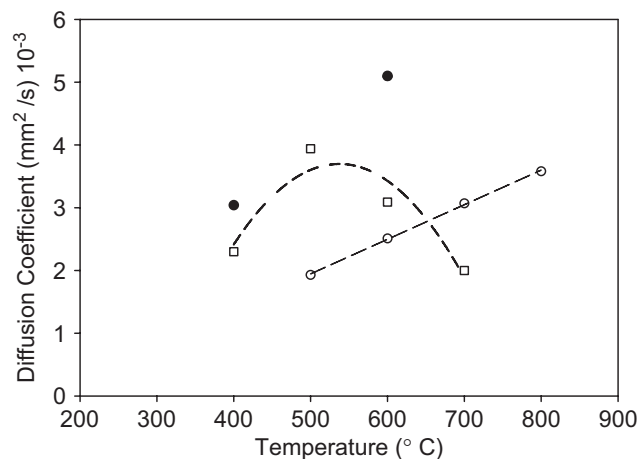


Fig. 2. Diffusion coefficients as a function of temperature for the Pb–1 wt% Au in microgravity. (○) Data points reported in [1,2] with g-jitter suppressed. (●) Data points reported in [15,16] with g-jitter suppressed. (□) Non-isolated data points reported in [16].

was of interest to find out if an alternative relationship could be established when all available diffusion data for the lead–1 wt% gold system obtained under isolation mode on MIR were included in the analysis. It was possible to identify at least two additional data points for the lead–1 wt% gold system ($D = 5.1 \times 10^{-9} \text{ m}^2/\text{s}$ at 600°C and $D = 3.04 \times 10^{-9} \text{ m}^2/\text{s}$ at 400°C) (black circles) that were obtained on MIR with g-jitter suppressed and that were reported earlier in [15,16], respectively. These two data points represent $\sim 30\%$ of the total data points reported for the lead–1 wt% gold system in isolation mode. It is seen in Fig. 2 that when one considers the new data points in the analysis, a $D \propto T$ relationship cannot be claimed with a reasonably high level of confidence due to the large scatter of the data points. Again, as noted earlier, more data would certainly help. But until there are additional and confirmatory experiments one can only analyze the data at hand. The additional diffusion data for gold and silver reported here could also be added to the limited data presented in Table I–II in [17,18]. If this is done, it will cast doubt on

the reliability of the conclusions that were drawn based on the limited data that were selected for the analysis in [17,18].

3.2. Sensitivity of the diffusion experiments to the ambient g-jitter of MIR

In the original papers published by Smith [2], Herring et al. [3] and Tryggvason et al. [4], the sensitivity of the diffusion experiments to the ambient g-jitter of MIR was demonstrated by comparing the diffusion data obtained for the lead–1 wt% gold system in isolation mode with the data obtained under non-isolated (“latched”) condition. The results from g-jitter isolation experiments were shown to be significantly lower than the diffusion coefficients obtained under non-isolated conditions and those reported by Froberg et al. [7]. Based on these results, it was concluded that the long capillary diffusion experiments conducted on MIR were sensitive to vibration at levels that were generally below the current ISS specification for ARIS isolated racks [4]. Smith et al. seem to suggest that their diffusion measurements on MIR were so precise that they could actually detect and measure the experiment sensitivity to g-jitter. Their findings, however, cannot be reliable because very limited numbers of data points were considered in the study and the sources of errors in the experiments were not discussed. In Fig. 2, the available data obtained under non-isolated conditions (square symbols, extracted from [16]) are compared with the results obtained under isolation mode. The findings in Fig. 2 are important because they show that (i) the data obtained in isolation mode on MIR vary quite significantly in two replicated experiments and (ii) the diffusion data obtained in isolation mode are not consistently below the data obtained in non-isolated (“latched”) mode as has been claimed in the past [1–4]. One may even argue that over the range of temperature studied, the data obtained with MIM operating in isolation mode are on average slightly higher than those obtained in non-isolated condition. The findings presented in Fig. 2, and in particular the large scatter of the diffusion data, suggest that there is an uncontrolled source of experiment variation that is stronger than the innate variation of the diffusion experiments with g-jitter. What might be the source of the variations in the data? As reported earlier [19], it appears that there were serious problems with the operation of the QUELD II furnace onboard the MIR space station that imposed serious limitations on the accurate measurements of time and temperatures during the processing of diffusion couples. Apparently, the processing times and temperatures were deduced by visually examining the

color of an oxide that formed on the samples’ container tubes [16]. One possibility that should be considered is the fact that the subjective and unusual procedure that was used to deduce the processing time and temperatures could bring about uncontrolled variations in the measured values of the diffusion coefficients. Thus, the apparent reduction of the measured values of the diffusion coefficients that was observed in few isolated experiments might simply arise from the subjective procedure and other variations unrelated to g-jitter. There are, as seen below, major errors and uncertainties associated with the thermal treatment of the samples, measurement of temperature, diffusion time, and the solute concentrations that could all bring about significant variations in the measured values of the diffusion coefficients.

3.3. Problems associated with the design of the QUELD II furnace

One problem with the diffusion experiments conducted on the MIR space station appears to be associated with the non-isothermal treatment of the diffusion couples in the QUELD II furnace. This furnace was developed at Queen’s University (Kingston, Canada) with support from the CSA. The furnace was apparently tested on ground to ensure that the isothermal processing of the diffusion couples on MIR could be reliably achieved. But the results obtained from the ground-based tests reported in [16] show that during operation, the furnace temperature was fairly constant only within a short segment that was 35 mm long. Test results in [16] show, for example, that beyond the short segment the furnace temperature dropped continuously to temperatures as low as $\sim 320^\circ\text{C}$ for a set point of 500°C , and temperature gradients as high as $\sim 60\text{ K/cm}$ could be established within the furnace. It should be noted that for the experiments on the MIR space station, the diffusion couples ($\sim 40\text{ mm}$ long) were enclosed in stainless steel tubes that were over 100 mm in length, much longer than the constant temperature zone in the furnace. Test results reported in [16] show that significant temperature differences, as high as 20 K, could exist in the diffusion couples under normal operating conditions. The likely influence of the non-isothermal treatment of the samples on the measured values of the diffusion coefficients has not been discussed by Smith et al. [1–4] but in [16], it is argued that the temperature gradient in the samples is “a necessary component to obtain sound specimens using the quenching method employed in QUELD II”. Thus it appears that even under normal conditions, when the samples are fully inserted into

the furnace, there is always a significant temperature gradient in the samples, particularly at high temperatures because of the larger heat fluxes towards the cold sinks. However, as will be seen below, a large number of samples processed on the MIR space station were not in fact fully inserted into the furnace because of the malfunction of the QUELD II furnace during the diffusion experiments. This appears to have compounded the problem associated with the non-isothermal treatment of the samples and to have imposed serious limitations on the accurate determination of times and temperatures. It is also interesting to note that the numerical model recently reported by Smith et al. [17] predicts significant temperature variations along the length of the diffusion couples. It is seen in [17] that the temperature in the middle of the sample seems to be approximately 200 °C greater than the temperature at each end. One would think that under such non-isothermal conditions, thermotransport would significantly distort the results of a liquid metal diffusion experiment that is performed in microgravity. In the ground-based experiments, the buoyancy-driven transport is expected to distort the results even further. Thus, if in fact what the numerical model suggests is meaningful and applicable, then one needs to be very concerned with the assumption that the diffusion coefficients on earth and in space were obtained under isothermal conditions.

3.4. Experiment variations caused by the improper functioning of the QUELD II furnace onboard the MIR space station

As reported recently [19], it appears that the long capillary diffusion couples processed on the MIR space station encountered some difficulties due to the malfunction of the QUELD II furnace that made it impossible to accurately measure the processing times and temperatures. According to [16], the astronaut performing the experiments on MIR noted that a large number of samples were not fully inserted into the furnace while they were being heated. Apparently, one of the sample mounting arms became bent and “the tube containing the sample was entering the furnace at an angle and were catching on the furnace walls”. In [16], it is stated that “it was hoped that the oxide film on the tubes would provide specific information about the furnace temperature, the treatment time, and the degree of container insertion into the furnace”. The processing time and temperatures were apparently deduced by visually examining an oxide film that formed on the sample containers [16]. The arbitrary procedure that was employed for deducing time and temperature has

not been described in [1–4] but in [16], it is stated that “if it is determined that a quantitative and significantly more accurate measurement of the oxide thickness should be made in order to validate the results that will be obtained from these methods, it will be necessary to physically measure the depth of this thin film”.

According to [16] “only 11 of the 37 samples were fully inserted into the furnace and so could be processed as desired”. According to [16], the lead–1 wt% gold samples processed on MIR in isolated mode (sample nos. 1, 2, 3, and 4 in Table VI-3 [16]) were among those samples that were not fully inserted into the furnace and, therefore, did not experience the desired processing times and temperatures. Interestingly, as seen in Fig. 2, the diffusion coefficients for the above samples reported in [1,2] fit remarkably well with the expected $D \propto T$ relationship. According to [16], the experimental results from lead–gold diffusion couples “should be of particular concern”. Despite this warning, the experimental results for the lead–gold diffusion couples were used in [1–4] to draw the conclusion that the diffusion coefficient measured in the isolated mode in space are significantly smaller than the Froberg’s results [7] and most likely reflect the true intrinsic diffusion coefficient. These findings and conclusions do not appear to have much scientific value because the processing times and temperatures that were so vital for precise determination of diffusion coefficients were not scientifically measured. It is particularly difficult to understand how the diffusion times could be deduced for samples that were not fully inserted into the furnace. According to [16], those samples that were not fully inserted into the furnace were inserted “to the position in the furnace where the blockage prevented further ingress and so the current demanded by electric sample translation motor rose sharply to its cut-out limit and turned off the supply to the motor”. It is not clear how long it did took to finally pull out the samples from the furnace in each case and how it was possible to figure out the processing times from the color of an oxide that apparently formed on the container tubes. Table 2 shows an example of how much the diffusion times reported could vary for one binary alloy system (lead–antimony).

3.5. Problems associated with the post-flight analysis of the solute concentration profiles

3.5.1. Microgravity experiments onboard the Space Shuttle Endeavour Mission STS-47 and the Space Shuttle Columbia Mission STS-52

In recent studies reported by Smith [2], Herring et al. [3], and Tryggvason et al. [4], the measured diffusion

Table 2

Variations in the diffusion times and temperatures reported in [15,16] for the lead–1 wt% antimony.

Sample number	Temperature (°C) (Ref. [15])	Holding time (h) (Ref. [15])	Temperature (°C) (Ref. [16])	Holding time (h) (Ref. [16])	Diffusion coefficient (mm ² /s) 10 ⁻³
82	400	1.75	390	1.70	2.74
83	550	1.30	542	1.52	4.21
84	700	0.98	684	1.15	5.91
85	800	0.83	785	0.97	6.75

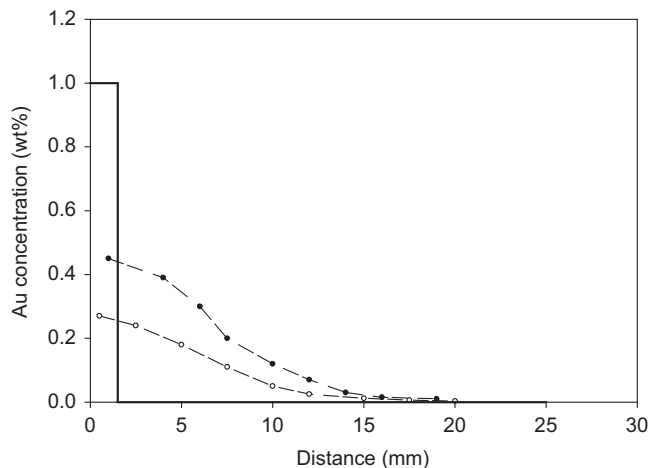


Fig. 3. Concentration profiles for the Pb–1 wt% Au diffusion couples processed in the space shuttle and on ground [8,9]. The solid line represents the initial concentration profile. (●) Space Shuttle, 383 °C. (○) Ground-based results, 670 °C.

coefficients for the lead–1 wt% gold samples processed on space shuttle flights have been compared with the results of the g-jitter isolation experiments on MIR to demonstrate the usefulness of the MIM in reducing the vibratory accelerations. However, one problem with the diffusion coefficients measured on the Space Shuttle flights appears to be associated with the post-flight analysis of the solute concentration profiles. Using the original data extracted from [8,9], typical solute concentration profiles for the lead–1 wt% gold are shown in Fig. 3 together with the initial solute concentration profile in the samples. Using these results, it is possible to calculate the area under the curves and determine the amount of the solute (gold) that diffused into the specimen during the diffusion experiments. Assuming an initial gold concentration of 1 wt% in the alloy section of the diffusion couples, the area under the top concentration profile in Fig. 3 shows that the amount of gold that diffused into the diffusion specimen in space was more than 2 times the original mass of gold in samples. Thus the error associated with each point appears to be on average more than 100%. The concentration data

that were used to determine the diffusion coefficients under microgravity conditions in the space shuttle are so improbable that it seems impossible that they represent experimental data that were simply overestimated by an error associated with the atomic absorption spectroscopy (AAS). The concentration profiles obtained for the diffusion couples processed on the space shuttle and on the ground can also be compared in Fig. 3. It is seen that apparently more gold was found in samples processed in space than the ones processed on earth despite the fact that the initial concentration of gold was the same in both experiments. One may argue that perhaps the initial gold concentration in the samples varied from one experiment to the next experiment but this is unlikely since the diffusion couples were apparently prepared simultaneously by casting in one mould using the same molten lead–gold alloy that contained 1 wt% gold [15]. Considering the significant variations in the amount of gold detected in the samples during the post-flight analysis, it seems futile to use the diffusion coefficient values derived from these measurements to draw any conclusion with respect to usefulness of the MIM in reducing the vibrations.

3.5.2. Post-flight analysis of the solute concentration profiles for samples processed on MIR

Similar errors were also identified for a large number of diffusion couples processed on the MIR space station, including lead–1 wt% silver and antimony–1 wt% indium. In Fig. 4, the solute concentration profiles for the antimony–1 wt% indium samples processed at 700, 750, and 800 °C in isolation mode are re-plotted together with the initial concentration profile in the samples by extracting the original data from [15]. Using these results, it is possible to calculate the area under the curves and determine the amount of the solute (indium) that diffused into the samples during the diffusion experiments at various temperatures. It is seen that there is a significant variation in the total amount of indium detected in samples despite the fact the initial concentration of indium in all samples was the same (1 wt%). Moreover, at

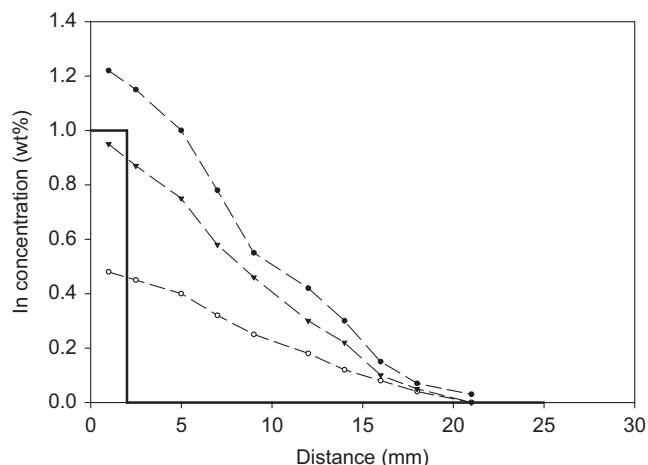


Fig. 4. Concentration profiles for the Sb–1 wt% In couples processed on MIR space station with g-jitter suppressed at various temperatures. The solid line represents the initial concentration profile. (●) 700 °C. (○) 750 °C. (▼) 800 °C.

700 °C, the area under the concentration profile shows that apparently the amount of indium detected in the diffusion specimen was more than 5 times the original mass of indium used in the alloy section of the sample. Again, one may argue that perhaps the diffusion couples had significantly different initial concentrations but this is unlikely since the diffusion couples were made simultaneously by casting in one mould using the same molten antimony–indium alloy that contained 1 wt% indium [15]. These findings place further doubt on the reliability of the solute concentration profiles that were used to determine the diffusion coefficients.

4. Concluding remarks

Various groups of researchers are planning to conduct microgravity experiments on the international space station (ISS) to examine the effects of the residual accelerations and vibrations. It was one of the purposes of the analysis presented here to bring to light the uncharacterized sources of experimental variation in the MIR experiments and offer alternative explanations for the patterns in the experimental data, and so perhaps guide future studies of the liquid metal diffusion in microgravity.

The analysis was framed as a response to three main questions. First, it was asked whether the linear temperature–dependence ($D \propto T$) that has been claimed for a range of binary alloys is valid when all available diffusion data measured in the isolated mode on MIR are considered in the analysis. The answer to this question was found to be no, meaning that one

should be highly skeptical about the limited data that were considered to demonstrate the $D \propto T$ linear relationship. Then it was asked whether the measured diffusion coefficients obtained with g-jitter suppressed are reproduced in two replicated experiments and whether the diffusion coefficient values obtained with g-jitter suppressed are consistently below the corresponding non-isolated microgravity data. The answers to these two questions were also no, suggesting that the apparent differences in the measured values of the diffusion coefficients may have arisen from the measurement errors and variations unrelated to g-jitter.

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