



Effect of some processing parameters on microstructure and hardness profiles of Al-Al₂Cu functionally graded materials prepared in-situ via interaction between molten Al and solid Cu

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Received: 10 June 2024; Accepted: 24 June 2024

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ABSTRACT

Functionally graded materials (FGMs) are receiving excessive consideration as they provide optimum thermal and mechanical properties without a distinct interface between two materials. These materials are considered for many applications such as aerospace, nuclear, energy, biology, electromagnetism, optics, energy and other fields. In the present study, for the first time, Al-Al₂Cu FGMs produced by employing a casting method by utilizing the interaction between molten Al and solid Cu. Different weights of solid copper placed in a ceramic mold, pre-heated to the desired temperature and subsequently filled with molten Al at various temperatures. Consequently due to different proportions of generated Al-Cu solid solution (α -Al), Al-Al₂Cu eutectic and remained un-reacted Al in the molten alloy, a functionally graded material was formed. The specimens subjected to optical microscopy (OM), X-ray diffraction (XRD) studies and hardness measurements. It was found that the pouring temperature as well as the mold preheat temperature affected the resultant microstructure gradients in the samples via their effects on the cooling rates during solidification. Moreover, the increased weight of solid copper resulted in shifting the hardness profiles towards higher values, while the gradient remained almost constant. It was concluded that three phenomena including melt convection, gravitational macro-segregation, and normal segregation governed the distribution of Cu atoms towards different directions in the samples. However, due to the dominant effect of gravity, steeper microstructure and hardness gradients obtained along the vertical as compared with horizontal directions.

Keywords: Casting parameters, Functionally graded Al-Al₂Cu, In-situ composites, Microstructure, Hardness profiles.

1. Introduction

Aluminum matrix composites (AMCs) due to their low density and acceptable mechanical properties have a wide range of application in automotive and aerospace industries [1-4]. These composites are fabricated either by directly incorporating reinforcements into the matrix or by creating the reinforcements in situ in the metallic matrix [5]. The advantages of in situ methods over conventional processing include formation of small sized reinforc-

ing particles, clean interface, good wettability as well as homogeneous distribution of the reinforcement within the matrix [6-8]. Hsu et al. [9] fabricated in situ ultrafine-grained Al-Al₂Cu composites by friction stir processing. Aravind et al. [10] reported the fabrication and characterization of Al₂Cu and other Al-Cu intermetallics in Al (Cu) alloy matrix composites by reaction sintering. However, in situ processing by casting routes as compared to other methods such as powder metallurgy and spray

forming are more economical and practicable for large-scale production of composites [6].

Functionally graded materials (FGMs) belong to a relatively new class of inhomogeneous composite materials, in which the composition and structure gradually change resulting in corresponding alteration in the properties of material. Several processing techniques explored for fabrication of FGMs for utilization in different applications [11–14].

Copper is one of the few elements that its phase diagram with aluminum has been considered broadly and forms different phases and compounds [10, 15–22]. According to the binary phase diagram of Al/Cu, various phases and intermetallics may form depending on chemical composition and temperature [18]. The Al rich corner of this phase diagram demonstrates that maximum solid solubility of Cu in Al is 5.65 wt. % at 548.2 °C. For

Cu contents lower than 33 wt.% one can notice a eutectic transformation in a wide region resulting in formation of Al_2Cu (θ) and solid solution of Cu in Al.

Functionally graded Al- Al_2Cu composites due to their excellent properties have potential applications in some specific industries. Watanabe et al. [23, 24] successfully produced in situ functionally graded Al- Al_2Cu composites from not only hypoeutectic or hypereutectic alloys but also eutectic Al- Al_2Cu compounds by a centrifugal method. In this method, the applied centrifugal force during solidification of Al-Cu melt enables creation of a compositional gradient resulting in graded distribution of the primary intermetallic compounds in an aluminum matrix. They deduced that the graded structure in a eutectic alloy is created due to the different densities of Al and Cu in the liquid state. The fabricated FGMs using

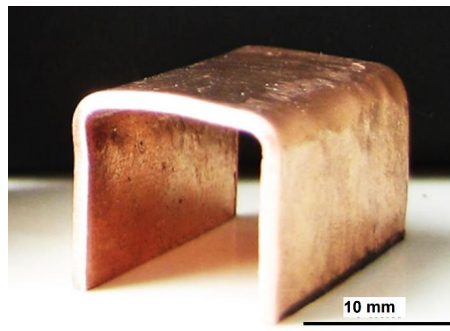


Fig. 1- The solid copper used in this study.

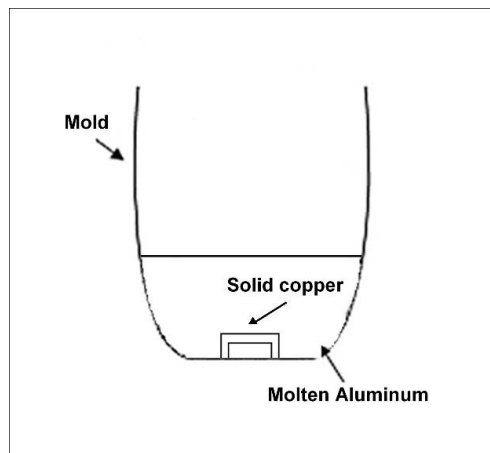


Fig. 2- Schematic representation of the setup used for casting.

Al-Cu melts with different Cu contents were tubular FGM specimens. The cross section of these tubes exhibited a number of rings with graded microstructure, hardness and density from the center towards the edge of samples [23, 24].

In the present study, for the first time, functionally graded Al–Al₂Cu composites produced by employing a casting method via interaction between molten Al and solid Cu. The microstructure and hardness gradients along different directions on the sectioned samples quantified and related to the employed casting processing parameters via their effects on the cooling rates during solidification.

2. Experimental Procedures

Commercially pure aluminum ingot served as the matrix of the composites, while a commercially pure copper sheet with dimensions of 30x10x1 mm³ used to create the reinforcing phase of composites. This rectangular copper sheet bent vertically from two sides along its width at a distance of 10 mm on each side of its length as shown in Fig. 1 to serve as the solid copper in our experiments. This bended copper sheet hereinafter referred to as solid copper in the present paper. In order to remove the oxide layer from the surfaces of the solid copper and achieve a true Al/Cu contact, its surfaces initially ground with 60-grid silicon carbide paper and subsequently treated with 15 % hydrochloric acid for 45 minutes at room temperature. Then in order to pre-

vention re-oxidation of copper, a thin layer of Al formed on the surfaces of this solid copper through a hot dipping process. For this purpose, the solid copper immersed in a bath of molten aluminum for 2 seconds.

The coated solid copper placed at the base of a ceramic mold (graphite crucible) and heated to the required temperature in an electric resistance furnace. Then, about 120g of commercially pure aluminum melt was prepared in a graphite crucible in another electric resistance furnace. The pre-heated mold filled with molten aluminum at the same temperature as the pre-heated mold temperature. Hereafter, cooling was carried out in a turned off furnace in air. A schematic representation of this setup is shown in Fig. 2.

The specimens sectioned from the center along their height (i.e. direction of gravity during solidification), polished by using standard metallographic techniques and subsequently etched with 0.5% HF at room temperature. The vertically sectioned sample is divided into four regions along the central axis of symmetry of sample being in the direction of gravity during solidification. These regions from top to bottom are designated by A, B, C and D. A macroscopic view of the polished surface of a sample exhibiting these four specified investigated areas is presented in Fig. 3.

The fabricated samples subjected to optical microscopy (OM) to investigate different microstructures of each sample at the specified areas shown in Fig. 3. Moreover, A

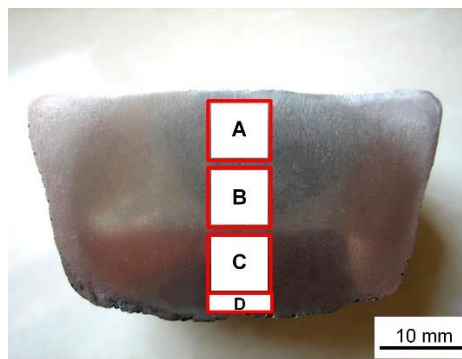


Fig. 3- Macroscopic view of the polished surface of a sample exhibiting the four specified investigated areas.

high-resolution X-ray diffractometer (Philips-X'pert,) with a rotating copper anode (Cu K α 1, $\lambda= 1.5406 \text{ \AA}$) was used for X-ray diffraction (XRD) analysis to study the phases formed at different parts of the samples. Scans were collected over 2θ range of $10\text{--}110^\circ$ with step size of 0.05° (counting time of 1s per step) and accelerating voltage of 40 kV. XRD patterns analyzed by using X'Pert High Score software (Philips-X'pert). Hardness measurements on the samples were carried out on a Vickers

hardness-testing machine (ESE WAY hardness tester device) with the accuracy of $\pm 5\%$ of the measured value, using a load of 30Kg, and the average values of at least 5 measurements conducted on different areas of each sample was considered.

3. Results and Discussion

3.1. Effect of pouring temperature

The effect of four pouring temperatures of 660, 700, 800, and 900 °C on the induced

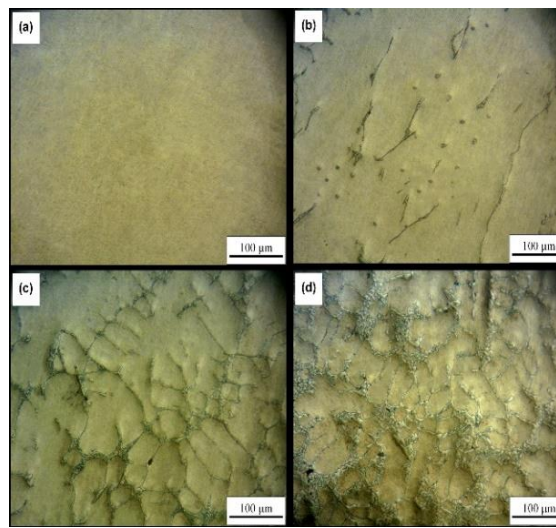


Fig. 4- Micrographs of the four specified regions (a) A, (b) B, (c) C, and (d) D along the height of the sample (as specified in Fig.3) produced at pouring temperatures of 660 °C.

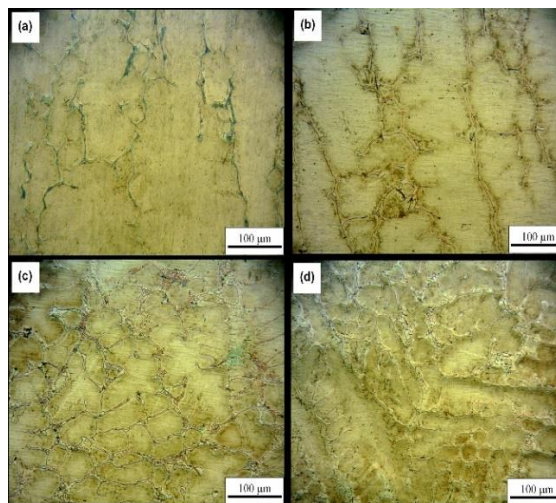


Fig. 5- Micrographs of the four specified regions (a) A, (b) B, (c) C, and (d) D along the height of the sample (as specified in Fig.3) produced at pouring temperatures of 800 °C.

microstructure and hardness gradients along the height of samples investigated. The solid copper for each sample preheated to the same temperature as the selected pouring temperature. The micrographs taken from four specified regions along the height of samples (regions A to D of Figure 3), produced at pouring temperatures of 660 °C, 800 °C and 900 °C are shown in Figs. 4, 5 and 6 respectively.

It is clear that the amount of eutectic decreases and the grain size as well as the sec-

ondary dendrite arm spacing (SDAS) increase from bottom towards the top of all the samples. In addition, the increased pouring temperature resulted in increased vol.% of eutectic at the top regions, accompanied with increased grain size. Actually, the microstructure gradient is evident from these images. The XRD patterns of different regions of the sample in which the pouring temperature was 800 °C is shown in Fig. 7. It is clear that at the top of the sample (region A), only Al peaks detected. At region

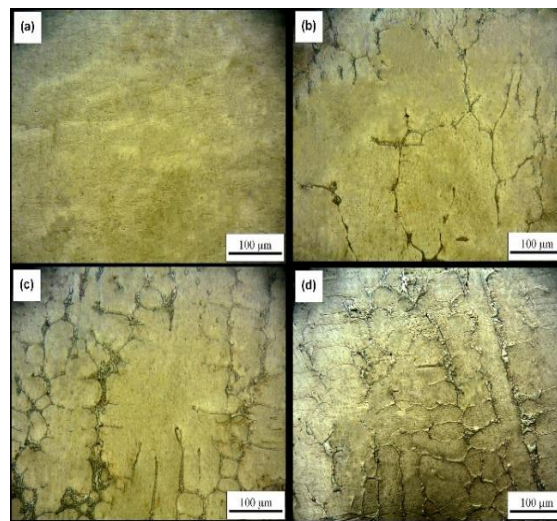


Fig. 6- Micrographs of the four specified regions (a) A, (b) B, (c) C, and (d) D along the height of the sample (as specified in Fig.3) produced at pouring temperatures of 900 °C.

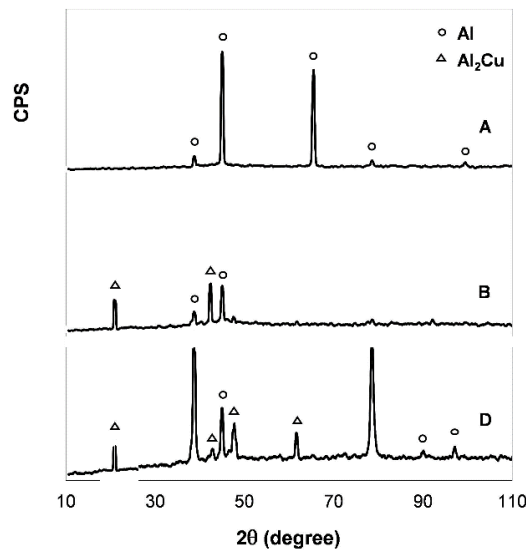


Fig. 7- XRD patterns of the 800 °C produced FGM corresponding to three regions along the height of the sample as specified in Fig.3.

B of the sample, the Al and the Al₂Cu peaks co-exist. XRD pattern of the region D exhibits increased number as well as increased intensity of some Al₂Cu peaks. These XRD patterns confirm that the identified intermetallic phase detected on grain boundaries of microscopic images is Al₂Cu compound.

Fig. 8 shows the hardness profiles along the height of four samples prepared at different

pouring temperatures. For all the four pouring temperatures, the hardness values gradually decrease from bottom towards the top of the samples. These hardness gradients attributed to different contents of Cu and Al₂Cu intermetallic compound at different regions of these samples.

According to Fig. 8, the increased pouring temperature resulted in decreased hardness

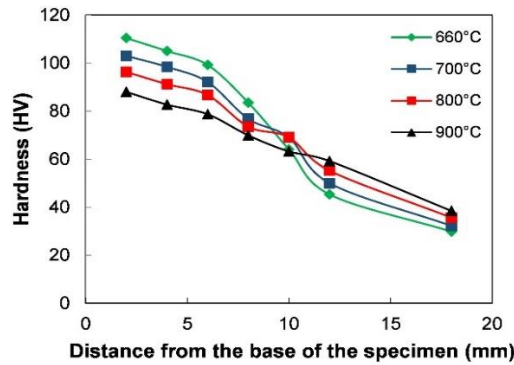


Fig. 8- Hardness profiles along the height of four samples prepared at different pouring temperatures.

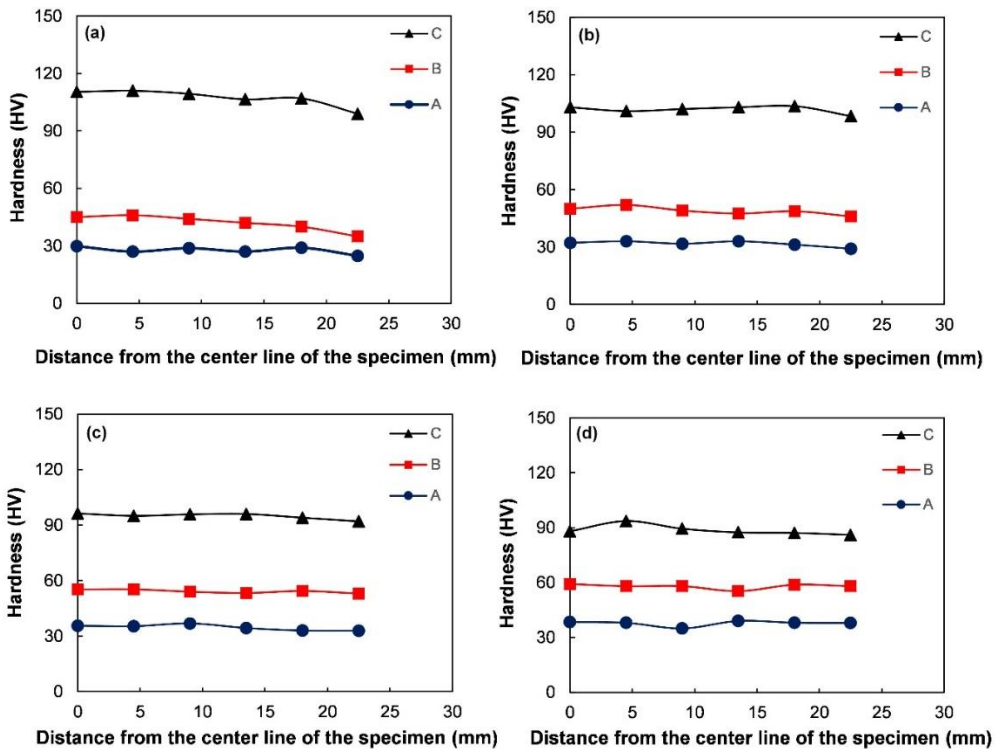


Fig. 9- Hardness profiles of samples produced at four different pouring temperatures. (a) 660 °C, (b) 700°C, (c) 800°C and (d) 900°C quantified along the horizontal direction, i.e. perpendicular to the gravity direction during solidification of samples and at different heights specified in Fig.3 by A, B, and C.

at the lower regions of the sample but at the same time the hardness at the upper regions increased resulting in decreased slope of the hardness profiles.

Fig.9 shows the hardness profiles of samples produced at four different pouring temperatures, quantified along the horizontal direction, i.e. perpendicular to the gravity direction during solidification of samples and at different heights specified in Fig.3 by A, B, and C.

For each sample, the hardness on the center line of the specimen at the middle of three regions A, B, and C (Fig.3) measured and this measurement continued along horizontal direction towards the edge of that sample. It is clear that while the increased distance of the hardness measurement lines from the bottom of samples resulted in decreased hardness values, these hardness profiles exhibited no significant gradient. In addition, the increased pouring temperature resulted in decreased hardness of the lower regions of the samples and consequently hardness profiles got closer to each other.

In order to discuss these results, it is necessary to mention the following points. The melting point of pure copper is 1085 °C and it is obvious that in none of the experiments performed in this research the temperature of solid copper has reached this limit and therefore copper has not been melted. However, free copper (i.e. undissolved Cu) was not detected by neither microscopic nor XRD studies performed on all the samples. In fact, when solid copper comes in contact with molten aluminum, the aluminum atoms diffuse into the copper and the copper atoms diffuse into the aluminum at interface. Consequently, the copper atoms gradually separate from the solid surface and dissolve in the aluminum. Finally by continuing this process, the solid copper disappears due to its complete dissolution in aluminum melt before start of solidification. The formation of this solution is accelerated via mechanical mixing, possibly as a result of convection inside the melt [25]. These copper atoms distribute in the melt through the diffusion and convection process accompanied with gravitational macrosegregation, resulting in creation of different copper concentration gradients in different directions in the melt. However, due to the higher density of copper

(8.96 g/cm³) as compared to that of aluminum (2.7 g/cm³), Al-Cu alloys with different percentages of copper that initially formed at the lower regions of the mold are more likely to remain in the same regions, provided that not a long time remains before the start of solidification. In fact, due to the higher density of copper atoms compared to aluminum, the gravitational macrosegregation phenomenon contributes to the migration of copper to the lower parts of the mold. On the other hand, due to Al-Cu phase diagram, with increasing the percentage of copper in the alloy (before reaching the eutectic point), the melting point decreases and the melt fluidity increases. This effect enhances moving of the Cu atoms dissolved in Al to the other parts of the mold by the abovementioned mechanisms. With the onset of solidification and nucleation of α -Al phase, the copper atoms that are pushed by the solidification front and accumulated at the dendrite borders and grain boundaries can form Al-Al₂Cu eutectic compound provided that sufficient Cu concentration at the last freezing liquid as shown in Figs. 4-6. The decreased amount of eutectic from bottom towards the top of the samples as shown in Figures 4-6 as well as the XRD pattern (Fig. 7) confirm the decreased Cu content in the alloy towards the top of samples.

The increased pouring temperature accompanied with the increased mold pre-heating temperature results in decreased melt cooling rate. Consequently, the alloy remains in the liquid state for a longer time enhancing the migration and distribution of the Cu atoms in the melt. Therefore, by considering Figs. 4-6, the increased pouring temperature from 660 °C to 900 °C resulted in increased vol.% of eutectic at the top regions of the samples poured at the higher temperatures. It is well known that the increased solute content in alloys results in refining the microstructure via its effect on increased constitutional supercooling induced on solidifying melt [26]. Therefore, the increased both the SDAS and grain size from bottom towards the top of specimens (Figs. 4-6) is attributed to lower Cu content at the upper regions of these the samples. In addition, the increased pouring temperature accompanied with the increased mold pre-heating temperature, as was mentioned before, in

turn contribute in generation of coarser microstructure [27] observed in all the regions of the sample poured at 800 °C and 900 °C (Figs. 5 and 6) as compared with their counterparts poured at a lower temperature (Fig. 4).

Addition and increased amount of Cu as an alloying element to Al and its incorporation in the alpha phase as a solid solution, increases the hardness of the alloy [28, 29]. Also when

the eutectic compound of Al-Al₂Cu appears in the microstructure, due to the relatively high hardness of the Al₂Cu-based phase (588.4 HV) as compared to that of the α -Al phase (147.9 HV), much higher hardness values are achieved [30, 31]. Therefore, the gradually decreased hardness values from bottom towards the top of the four samples poured at different temperatures (Fig.8) is attributable to gradual

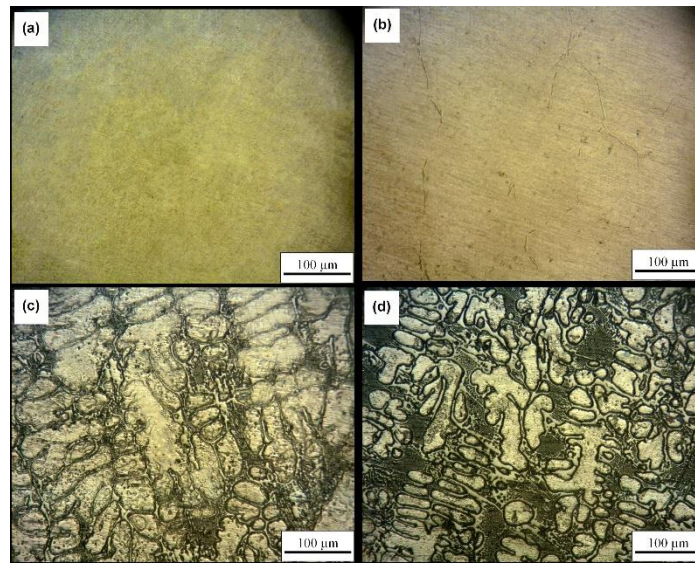


Fig. 10- OM micrographs of the four specified regions (a) A, (b) B, (c) C, and (d) D along the height of the sample (as specified in Fig.3) produced at pouring temperature of 660 °C without mold preheating.

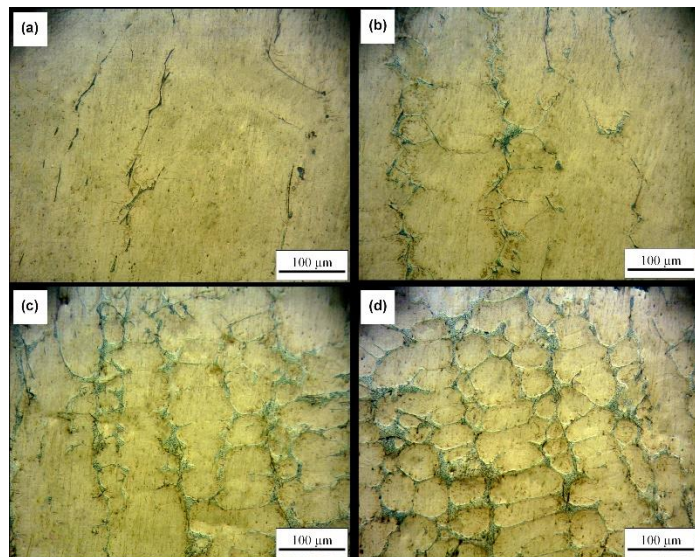


Fig. 11- OM micrographs of the four specified regions (a) A, (b) B, (c) C, and (d) D along the height of the sample (as specified in Fig.3) produced at pouring temperature of 800 °C without mold preheating.

decreas in Cu content in that direction.

As mentioned earlier, the increased pouring temperature results in suppressing the Cu concentration gradients in the melt towards reaching lower slopes. Therefore, the increased pouring temperature results in respectively increased and decreased hardness of the upper and lower parts of the sample consequenting the decreased slope of the hardness porfiles.

The results presented in Fig. 9 reveal that for all the pouring temperatures explored, dispersion of Cu atoms in the melt occurred throughout the entire mass of the melt causing the increased hardness. However, according to the mechanisms mentioned earlier, the increasd pouring temperature resulted in partial homogenization in Cu distribution so that

closer hardness values obtained for three vertical lines on the sectioned sample. The nearly zero slope of all the hardness profiles shown in Fig. 9 reveal that the gravitational macrosegregation was the dominant mechanism affecting the Cu distribution in the melt before the onset of solidification. Also the identical hardness values obtained for the edge and centre on each hardness profile confirms that the impact of normal segregation was not so prominent.

3.2. Effect of mold preheating temperature

In order to study the effect of mold preheating temperature on the microstructure and hardness profiles, two samples prepared at different pouring temperatures of 660 °C and 800 °C but no mold preheating. The other casting

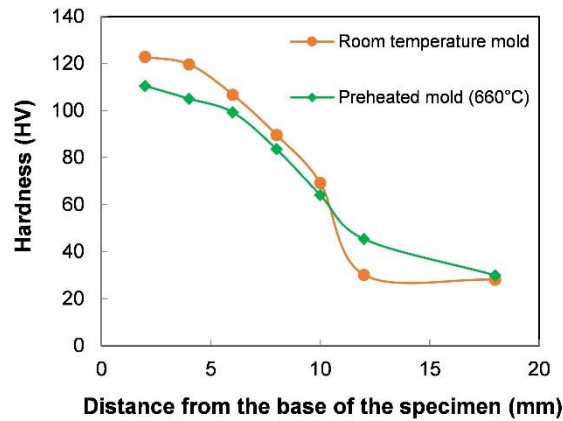


Fig. 12- Hardness profiles along the height of two samples prepared at pouring temperature of 660 °C but different mold preheat temperatures.

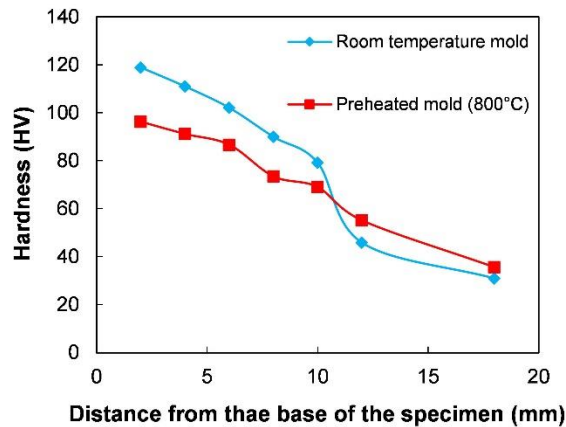


Fig. 13- Hardness profiles along the height of two samples prepared at pouring temperature of 800 °C but different mold preheat temperatures.

conditions for preparation of these two samples are identical to those described so far. The OM micrographs taken from four specified regions along the height of samples (regions A to D of Fig. 3), produced at initial mold temperature of 25 °C (i.e. room temperature) and pouring temperatures of 660 °C and 800 °C are shown in Figs. 10 and 11 respectively. A comparison between the microstructures pre-

sented in Figs. 4 and 10 in which both samples poured at identical temperature of 660 °C, reveals the role of mold preheating temperature. In the microstructure shown in Figure 10, due to using a non-preheated mold and low pouring temperature, the opportunity to propagate copper to the upper parts of the mold was not provided and therefore copper was mainly accumulated in parts C and D. In addition,

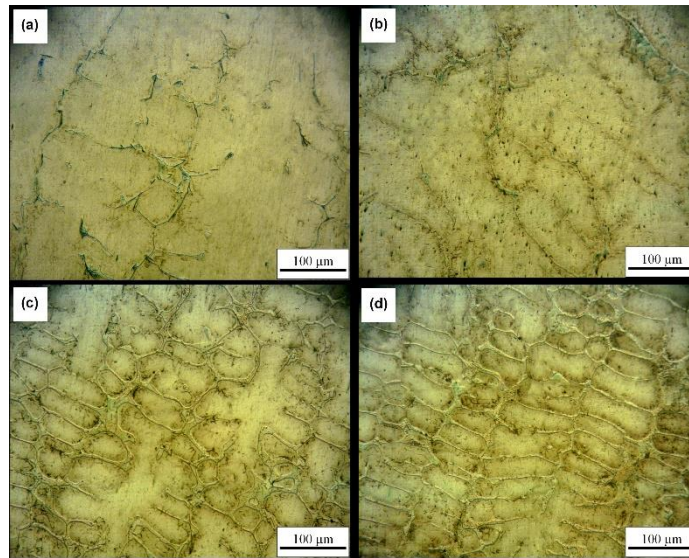


Fig. 14- OM micrographs of the four specified regions (a) A, (b) B, (c) C, and (d) D along the height of the sample (as specified in Fig.3) produced at pouring temperature of 900 °C using solid copper weighing 6g.

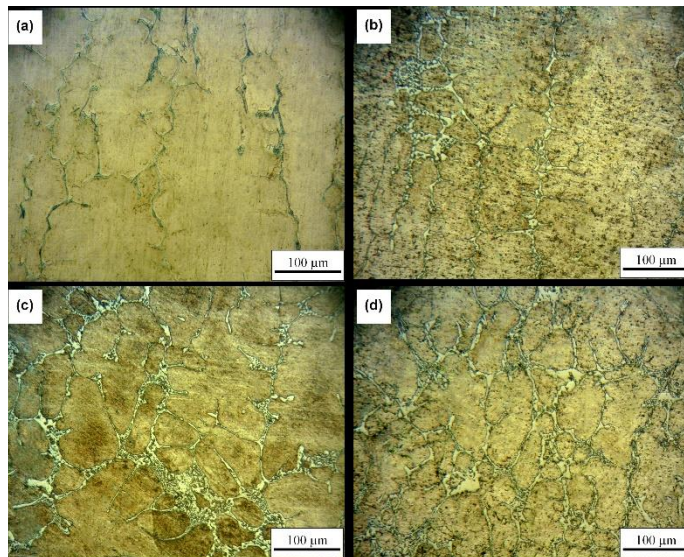


Fig. 15- OM micrographs of the four specified regions (a) A, (b) B, (c) C, and (d) D along the height of the sample (as specified in Fig.3) produced at pouring temperature of 900 °C using solid copper weighing 9g.

the size of the SDAS in each part of Fig. 10 is smaller and the eutectic percentage is higher as compared to its counterpart regions in Fig. 4. Similarly, by comparing Figs. 5 and 11 the same trend is noticeable. Comparing Figures 10 and 11, reveals that although in both cases the mold was not preheated, but the higher pouring temperature (Fig. 11) resulted in formation of eutectic at entire parts of the sample.

Figs. 12 and 13 show the hardness profiles along the height of samples prepared at pouring temperatures of 660 °C and 800 °C respectively. In each of these two Figs., one profile belongs to a sample in which no mold preheating was used while for the other one mold was preheated to a temperature identical to pouring temperature..

As it can be seen in Figs. 12 and 13, preheating the mold has reduced the hardness gradient in both samples. In fact mold preheating brings more heat in the system and hence decreases the cooling rate and provides more time for moving Cu atoms in the melt by convection before onset of solidification. As mentioned earlier, this results in decreased both the slope of Cu concentration and hardness profile along the height of the sample.

3.3. Effect of solid copper weight

The weight of the solid copper (Fig. 1) used in these experiments described so far was about 3 grams. In order to investigate the effect of copper weight on the hardness profiles

of produced samples, two experiments performed using 2 and 3 of the solid copper pieces that were stacked on top of each other and thus samples with 6 and 9 g copper produced. These experiments performed at a pouring temperature of 900 °C and the same mold preheating temperature.

The OM micrographs taken from four specified regions along the height of samples (regions A to D of Fig. 3), of these samples produced using different solid copper weights of 6, and 9g are shown in Figs.14 and 15 respectively. These micrographs can be compared to Fig. 6 that was produced under identical conditions as those of Figs. 14 and 15 but by using 3g of copper. The hardness profile along the height of these three samples are shown in Figure 16.

According to Figs. 6, 14 and 15, the increased weight of the solid copper, as was expected, resulted in increased vol.% of eutectic accumulated at SDAS borders and grain boundaries in all the four investigated areas of the samples. The increased Cu content in the Al-Cu alloys created at different parts of samples contribute either in the form of solid solution or eutectic structure. Therefore, the increased hardness of Al-Cu solid solution with increased Cu content as well as the greater hardness of Al₂Cu as compared with that of α -Al, resulted in shifting of the hardness profiles towards higher values as shown in Figure 16. It should be noted that the basic requirement in produc-

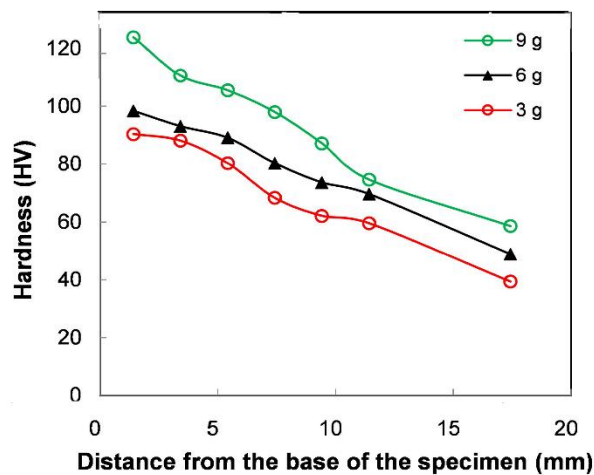


Fig. 16- Hardness profiles along the height of two samples prepared at pouring temperature of 900 °C using solid coppers with different weights.

tion of Al-Al₂Cu FGMs via the present method is complete interaction between solid copper and molten Al at onset of solidification. Therefore, copper weight should be chosen in such a way that the heat content of the molten Al be enough to insure complete solution of the solid Cu.

4. Conclusions

In present study, for the first time, a cost-effective casting method benefiting from utilization of conventional casting equipments employed for fabrication of Al-Al₂Cu FGMs by utilizing the interaction between molten aluminum and solid copper. The melt pouring temperature, mold pre-heat temperature and weight of the used solid copper all affected the microstructure and hardness profiles. The solidified alloy consisted of Al-Cu solid solution dendrites and grains with Al-Al₂Cu eutectic accumulated at dendrite borders and grain boundaries evidenced by microscopic images and XRD studies. Based on the present study, the following conclusions were made.

A gradient microstructure and hardness in vertical direction (parallel to gravity direction during casting) of all the solidified specimens achieved.

The amount of eutectic decreased and the grain size as well as the secondary dendrite arm spacing (SDAS) increased from the bot-

tom towards the top of all the samples.

The increased pouring temperature resulted in increased vol.% of eutectic at the top regions of the samples, accompanied with increased grain size.

For all the four pouring temperatures explored, the hardness values gradually decreased from bottom towards the top of the samples. These hardness gradients attributed to different contents of Cu and Al₂Cu intermetallic compound at different regions of these samples.

The increased pouring temperature resulted in decreased slope of the hardness profiles.

In order to find horizontal hardness profiles, hardness of different points on horizontal lines on the sectioned sample having different distances from the bottom of sample quantified. While the increased distance of these horizontal lines from the bottom of samples resulted in decreased hardness values, they exhibited no significant gradient. In addition, the increased pouring temperature resulted in decreased hardness of the lower regions of the samples and consequently hardness profiles got closer to each other.

Preheating the mold reduced the hardness gradient.

The increased weight of the solid copper resulted in shifting of the hardness profile towards higher values.

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