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Influence of Bismuth dopants on the Microstructure of Tin metal

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ABSTRACT

Bismuth–tin alloys having different proportions of both elements were synthesized by the zone melting method. EDAX and X–ray diffraction (XRD) data of all prepared samples recorded at room temperature confirmed the formation of Bi-Sn alloy and its polycrystalline nature. Scanning electron microscopy (SEM) of transverse cross sections revealed dependence on composition in the microstructure of all samples. The micrographs show no voids or discontinuities at the cross sections of any sample. The existence of distinguishable Bi-rich α phase and Sn-rich β phase indicates significant diffusion during cooling of the melt. Bismuth is seen precipitated out of the tin rich matrix. With increase in Bi concentration the microstructure gradually evolves into a lamellar structure showing relationship to their dendritic origin and discrete grains of Bi-rich phase having smooth rounded corners are seen. The details of the SEM studies are discussed in this paper.

***Keywords*: Zone melting method, Bismuth – Tin alloys, Microstructure, EDAX, XRD, SEM.**

I. INTRODUCTION

The choice of any material for a particular application is mainly decided by its various properties and behaviour under a given situation. The property of any material in turn depends on its atomic and electronic structures, crystal structure, microstructure, as well as various other macrostructural features. During the synthesis of an alloy, solid state transformation structures are produced on cooling from the different phases present and the product structure can consist of one or more phases in a particular morphology. Morphology, i.e. the size, shape and distribution of the phases present in the microstructure, is one way of characterizing the microstructure. The importance of microstructure to the properties of metals and alloys have long been recognized [1–3].

Over the last few decades, various researchers have investigated the different characteristics of bismuth alloys as well as tin alloy system. Bismuth–tin system has mainly attracted attention as a potential lead–free solder for microelectronics[4]. The bismuth–tin alloy system has also been investigated for their different applications other than as a soldering material [5-10]. Binary alloys like bismuth–tin alloys have limited solubility and hence their micrographs usually consist of two phases. In such structures, the major phase is called the matrix or the base structure and the minor phase is called the second phase. The size, shape and distribution of the second phase play a very important role in determining the properties of the alloy. The detailed study of these features requires examination by electron microscopy [11–12].

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In the present work, bismuth – tin alloys having different composition of both elements were prepared using the zone melting method [8] and the influence of bismuth concentration on microstructure were studied.

II. EXPERIMENTAL METHOD

In the present work, the materials used were AR grade bismuth and tin, in the form of granules. The starting material were thoroughly washed with 10% nitric acid solution, acetone and distilled water to remove surface oxide layer and were dried in hot air. Samples were taken in a quartz tube and were evacuated to a pressure of 8.2 x 10^{-4} mbar before being sealed. The sealed tubes were kept at a temperature of 300^0 C for 15 minutes while being rocked vigorously to mix the constituent components. After cooling of the sealed samples, they were loaded into a horizontal zone melting apparatus which was equipped with one circular ring furnace. The temperature gradient maintained at the liquid – solid interface for all the passes was 250 ^{0}C cm^{-1}.

Transverse sections of the prepared specimen were cut using a saw blade and were mechanically polished with grade 2000 carborundum paper, rinsed with distilled water and acetone and dried in hot air. The quantitative elemental analyses of the specimen were done using energy dispersive X–ray spectroscopy (EDAX) at room temperature using a JEOL JSM–5610 LV Scanning Electron Microscope at an accelerating voltage of 15 kV in back scattered mode. The microstructural investigations were done using the same scanning electron microscope.

III. RESULTS AND DISCUSSION

Fig. (a) to (h) show the back scattered SEM images of the cross sections of the prepared alloys. The micrograph shows variation in the microstructure of each sample as the percentage composition of bismuth is varied. The white planar phase shows the bismuth-rich α phase and the grey region corresponds to the tin-rich β phase as is confirmed by EDAX. The micrographs show no voids or discontinuities at the cross sections of any sample. This is because unlike other metals, Bi expands on solidification. The existence of distinguishable Bi-rich α phase and Sn-rich β phase indicates significant diffusion during cooling of the melt.

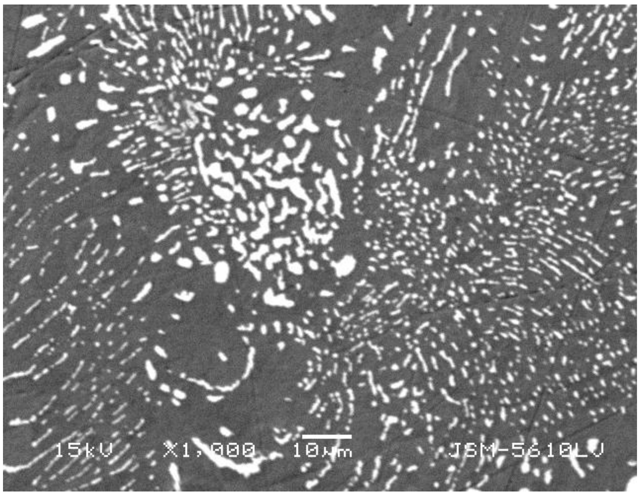

Fig.(a): SEM micrograph for 7.26 At. % Bi and 92.74 At.% Sn

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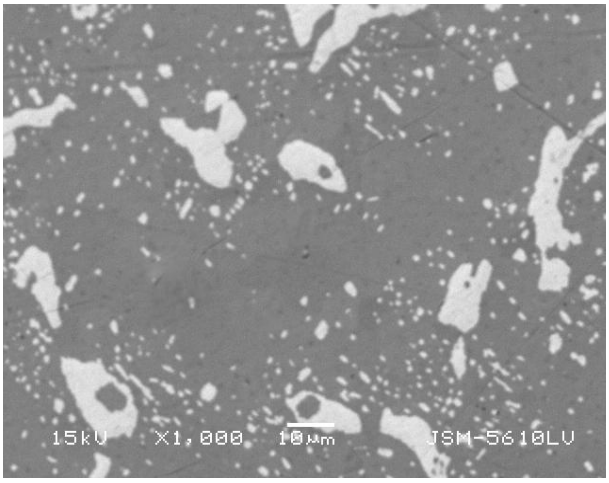

Fig. (b): SEM micrograph for 12.53 At. % Bi and 87.47 At.% Sn.

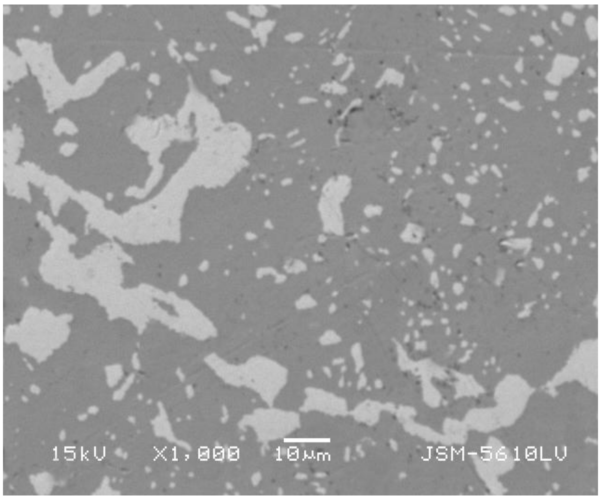

Fig.(c): SEM micrograph for 19.54 At. % Bi and 80.46 At.% Sn.

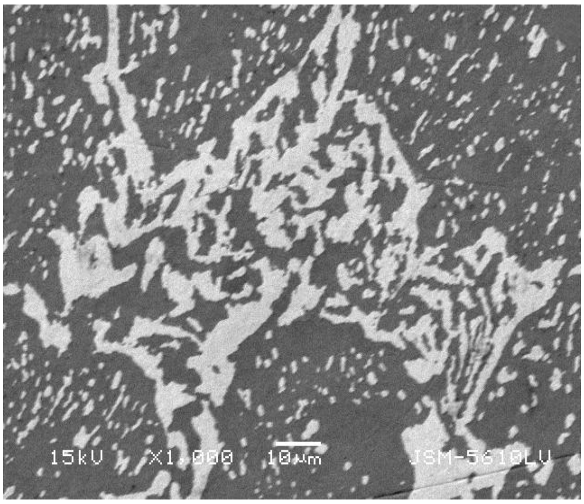

Fig.(d): SEM micrograph for 26.79 At. % Bi and 73.21 At.% Sn.

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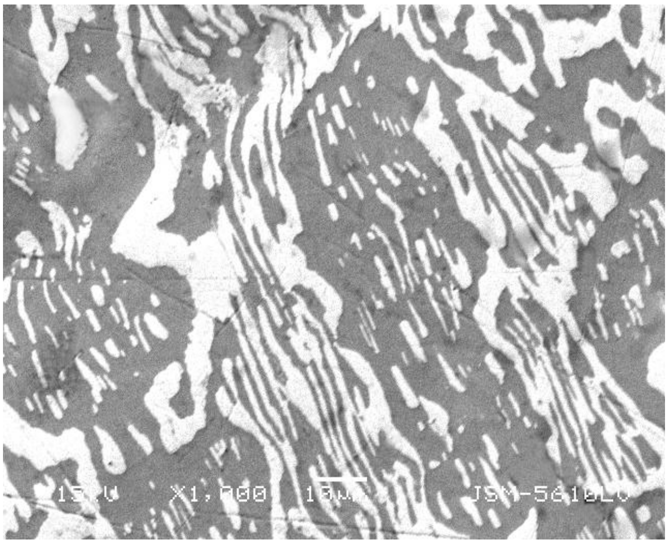

Fig.(e): SEM micrograph for 28.35 At. % Bi and 71.65 At.% Sn

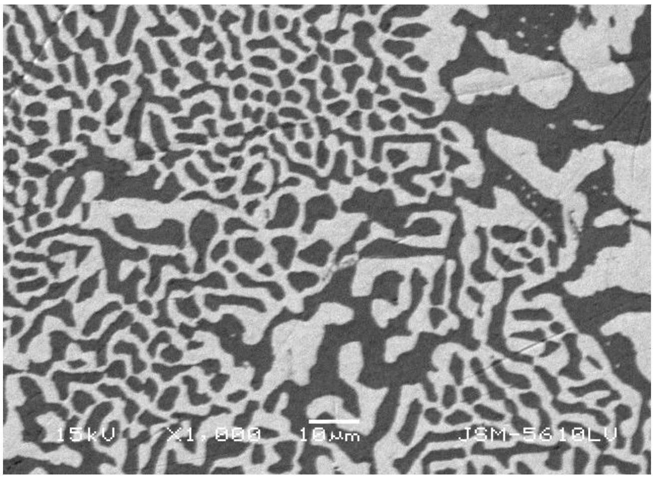

Fig.(f): SEM micrograph for 37.99 At. % Bi and 62.01 At.% Sn.

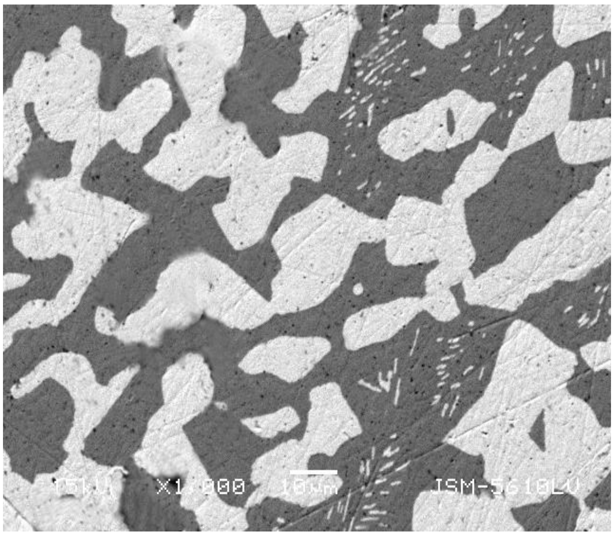

Fig.(g): SEM micrograph for 41.42 At. % Bi and 58.58 At.% Sn

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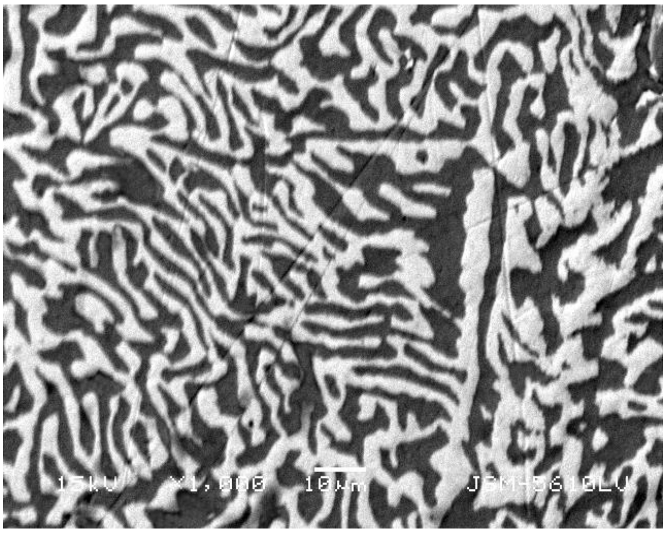

Fig.(h): SEM micrograph for 47.20 At. % Bi and 52.80 At.% Sn

The Bi-Sn phase diagram as calculated by J. Vizdal *et al* [13] indicates limited solubility of Bi and Sn at room temperature. The maximum solubility of Bi in Sn is ~ 6 at. % at the eutectic temperature of 138^0C . In the present study, since the bismuth content in all the alloy samples exceeds this limit, bismuth is seen precipitated out of the tin rich matrix, resulting in the observed structure. For the alloy with 12.53 at.% Bi, fine rounded particles of Bi are seen precipitated in the Sn-rich matrix. Sn with lower freezing temperature (~ 231.96 ^{0}C) is seen segregated to the edges of the solidifying Bi which has a higher freezing temperature of ~ 271.44 ^{0}C.

With increase in Bi concentration (Fig. c to e), the microstructure gradually evolves into a lamellar structure. In Fig. f, the Bi-rich phase shows relationship to their dendritic origin and discrete grains of Sn-rich phase having smooth rounded corners are seen. The randomly arranged phase regions of irregular shape in binary alloys have been reported by other researchers when simultaneous growth of both phases are not possible [14].

Fig. (g) and (h) show that increased Bi concentration has favoured the Bi dendrites to have filled out somewhat into large idiomorphic particles. Possibly, during the cooling of the melt in the equilibrium condition, Sn is getting dissolved and the microstructure consists of only Bi grains. Upon further cooling, the microstructure will no longer represent equilibrium conditions, but instead will be supersaturated with Bi atoms. Therefore, in order for the alloy to return to equilibrium, Bi atoms will tend to congregate in various regions of the sample to form colonies of Bi. The Bi atoms in some of these colonies will drift apart, while other colonies will grow but not form distinct particles. Such coarsening of microstructure in Bi–Sn alloys has also been observed by Liu *et al* [15]. It has been reported that the bismuth-rich phases grow by combination of previously formed and precipitated bismuth-rich phases through thermally activated diffusion during thermal aging since large bismuth particles grow at the expense of the smaller ones [16].

IV. CONCLUSIONS

Hypereutectic and hypoeutectic Bismuth–tin alloys beyond the solubility limit of each elements could be successfully alloyed using the zone melting method. SEM study reveals dependence of the alloy composition on the microstructure of all the prepared alloys. The observed variation in the microstructure would have a profound effect on the mechanical and other properties of the alloy which should be investigated.

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