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# Effect of silver and indium addition on mechanical properties and indentation creep behavior of rapidly solidified Bi–Sn based lead-free solder alloys

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ABSTRACT

Mechanical properties and indentation creep of the melt-spun process Bi–42 wt%Sn, Bi–40 wt%Sn–2 wt%In, Bi–40 wt%Sn–2 wt%Ag and Bi–38 wt%Sn–2 wt%In–2 wt%Ag were studied by dynamic resonance technique and Vickers indentation testing at room temperature and compared to that of the traditional Sn–37 wt%Pb eutectic alloy. The results show that the structure of Bi–42 wt%Sn alloy is characterized by the presence of rhombohedral Bi and body centered tetragonal β-Sn. The two ternary alloys exhibit additional constituent phases of intermetallic compounds $SnIn_{19}$ for Bi–40 wt%Sn–2 wt%In and ε-$Ag_3Sn$ for Bi–40 wt%Sn–2 wt%Ag alloys. Attention has been paid to the role of intermetallic compounds on mechanical and creep behavior. The In and Ag containing solder alloy exhibited a good combination of higher creep resistance, good mechanical properties and lower melting temperature as compared with Pb–Sn eutectic solder alloy. This was attributed to the strengthening effect of Bi as a strong solid solution element in the Sn matrix and formation of intermetallic compounds β-SnBi, ε-$Ag_3Sn$ and $InSn_{19}$ which act as both strengthening agent and grain refiner in the matrix of the material. Addition of In and Ag decreased the melting temperature of Bi–Sn lead-free solder from 143 °C to 133 °C which was possible mainly due to the existence of $InSn_{19}$ and $Ag_3Sn$ intermetallic compounds. Elastic constants, internal friction and thermal properties of Bi–Sn based alloys have been studied and analyzed.

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## 1. Introduction

It has been established [1] that rapid solidification can produce high-strength structural materials, tool and bearing materials; corrosion-resistant materials; catalytic and storage material and finally electrical and magnetic materials. These properties actually depend on the structural changes produced in each particular case. For example, high mechanical strength is attainable in microcrystalline materials as a result of refined microstructure combined with increased alloying [2]. Rapid solidification has several effects on the structure, the most important effects are; formation of metastable crystalline phases, formation of amorphous phases, extension of solid state solubilities, refinement of the as-cast grain size, modification of the segregation pattern and achievement of high point defect concentrations.

Lead bearing solders and especially the eutectic or near eutectic Sn–Pb alloys have been used extensively in the assembly of modern electronic circuits. However, increasing environmental and health concerns about the toxicity of lead, as well as the possibility of legislation (as described in the WEEE proposal [3]) limiting the usage of lead bearing solders, have stimulated substantial research and development efforts to discover substitute, lead-free solder alloys for electronic applications [4].

Creep behavior of the lead-free Sn–Bi solder alloys with bismuth contents in the range of 1–5 wt% was studied by long time Vickers indentation testing at room temperature [5]. Creep behavior of the lead-free Sn–5%Sb solder alloy in the cast and wrought conditions was investigated by long time Vickers indentation testing under a constant load of 15 N and at temperature in the range 298–405 K [6].

There have been many attempts to investigate the possibility of gaining information on creep properties by the use of impression tests [7–11]. In addition to solder alloys, this technique has been widely used in the study of creep behavior in superplastic materials [12–14]. The majority of published work on the impression creep behavior of solder alloys has focused on the eutectic Pb–62.8Sn alloy [11,12,15]. However, other solder materials, such as Pb–10Sn [14], and more recently Sn–3.5Ag [16–18], Sn–Bi alloys [19] and Sn–0.7Cu and Sn–9Zn [16] have also been studied

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by impression testing technique. On the other hand, the creep behavior of the lead-free Sn–Sb alloys is mostly studied by conventional creep tests [20–22] and room temperature indentation creep tests [23,24]. Effect of cooling rate on the room temperature indentation creep of cast lead-free Sn–Bi solder alloys has been studied by Mahmudi et al. [25]. The indentation creep process can be defined as the time dependent penetration of a hard indentation the material under constant load and temperature. The indentation maintains its load over a period of time under well-controlled conditions and the changes in the size of the indentation are monitored during the experiment. The indentation test provides a simple and non-destructive method of investigating the mechanical properties of solids. This can be particularly advantageous when the material is only available as small test pieces or there are difficulties with the machining of samples made of very soft materials [6,26]. Furthermore, the tensile strength and creep resistance of the eutectic Sn–Bi solder are higher than that of the eutectic Sn–Pb solder [27]. The majority of published data on the indentation creep behavior of solders alloys have focused on the eutectic Sn–37Pb alloy [28–30]. Thus, in the present study, the effects of In and Ag additions on the structure, mechanical, creep properties, melting point, internal friction and activation energy of ordering of Bi–Sn lead-free solder alloys were investigated and compared with traditional Sn–Pb eutectic alloy.

## 2. Experimental procedures

### 2.1. Materials and processing

The samples used in the present work are Sn–37 wt%Pb, Bi–42 wt%Sn, Bi–40 wt%Sn–2 wt%In, Bi–40 wt%Sn–2 wt%Ag and Bi–38 wt%Sn–2 wt%In–2 wt%Ag. These samples were melted in a muffle furnace using tin, lead, bismuth, indium and silver of purity better than 99.9%. The examined solder alloys are listed in Table 1.

The raw materials were placed in muffle furnace and melted at 500 °C. In order to homogenize the solder alloy, mechanical stirring was performed using graphite rod. Long ribbons of about 4 mm width and ~60 μm thickness were prepared by a single roller method in air by melt spinning technique. The surface velocity of the roller was 31.4 m/s giving a cooling rate of $\sim 3.7\times 10^5$ K/s. The samples then cut into convenient shape for the measurements using double knife cuter. The internal friction $Q^{-1}$ and the elastic moduli were determined using the dynamic resonance method. The value of the dynamic Young's modulus $E$ is determined by the following relationship [31–33]:

$$\left(\frac{E}{\rho}\right)^{1/2} = \frac{2\pi L^2 f_0}{kz^2} \quad (1)$$

where $\rho$ is the density of the sample under test, $L$ is the length of the vibrated part of the sample, $k$ is the radius of gyration of cross section, $f_0$ is the resonance frequency and $z$ is the constant depends on the mode of vibration and is equal to 1.8751. Plotting the amplitude of vibration against the frequency of vibration around the resonance $f_0$ gives the resonance curve, the internal friction $Q^{-1}$, of the sample can be determined from the following relationship:

$$Q^{-1} = 0.5773 \quad \Delta f/f_0$$

where $\Delta f$ is the half width of the resonance curve and $f_0$ is the resonance frequency. The structure of these alloys was examined by X-ray diffraction (XRD, X′ pert PRO, PANalytical using $CuK_\alpha$ target with secondary monochromator). Differential scanning calorimetry is carried out by [(DSC-16, Setaram, France) with a heating rate 10 K/min.

**Table 1**
Compositions of the solder alloys (wt%).

| Solder alloy series | Element content (wt%) | | | | |
|---|---|---|---|---|---|
| | Sn | Pb | Bi | In | Ag |
| Sn63Pb37 | 63 | 37 | | | |
| Bi58Sn42 | 42 | | 58 | | |
| Bi58Sn40In2 | 40 | | 58 | 2 | |
| Bi58Sn40Ag2 | 40 | | 58 | | 2 |
| Bi58Sn38In2Ag2 | 38 | | 58 | 2 | 2 |

### 2.2. Indentation creep tests

The polished specimens were tested in a Vickers hardness tester where the applied load and testing time were the only variables. In the Vickers test, a diamond pyramid with square base is used and the Vickers hardness number is given by $H_v = 0.185\, F/d^2$, where $F$ is the applied load in $N$ and $d$ is the average diagonal length in mm. Indentation creep measurements were made on each sample using 10, 25 and 50 gf load for dwell times up to 90 s. Each reading was an average of at least 15 separate measurements taken at random places on the surface of the specimens. The mechanical properties of metallic materials at homologous temperatures higher than 0.5 can be fairly expressed by the power-law creep in a wide range of strain rates. Thus, for steady-state creep, the high temperature creep rate ε is described by the power law of the type

$$\dot{\varepsilon} = A\sigma^n \exp\left(-\frac{E_a}{RT}\right) \quad (2)$$

where $\sigma$ is the applied stress, $A$ is a material constant, $n$ denotes the stress exponent, $E_a$ is the activation energy, $T$ is the temperature, and $R$ is the universal gas constant.

The relationship between indentation hardness and dwell time was done using Sargent–Ashby. The expression of Sargent–Ashby [34]:

$$H_v(t) = \frac{\sigma_0}{(nC_4\dot{\varepsilon}_0 t)^{1/n}} \quad (3)$$

where $H_v(t)$ is the time dependent hardness, $\varepsilon_0$ is the rate at a reference stress $\sigma_0$, $n$ is the stress exponent, $C_4$ is constant. Therefore, the slope of a plot of $\ln(H_v)$ against $\ln(t)$ at a constant temperature is $-1/n$.

## 3. Results and discussion

### 3.1. Structure

The X-ray diffractometry (XRD) patterns of the melt-spun Sn–37Pb, Bi–42Sn, Bi–40Sn–2In, Bi–40Sn–2Ag and Bi–38Sn–2In–2Ag alloys are shown in Fig. 1. The X-ray analysis of the Sn–37Pb eutectic alloy shows the phases of eutectic structure, body centered tetragonal β-Sn and face centered cubic α-Pb. For Bi–42Sn eutectic alloy indicates two phases of the eutectic structure rhombohedral Bi and body centered tetragonal β-Sn. After addition indium at 2 wt% to Bi–42 wt%Sn alloy the same phases appeared in addition to the two peaks observed for intermetallic compounds β-SnBi at $2\theta = 60.98°$, 62.23° and $InSn_{19}$ at $2\theta = 63.5°$, 72.20°. For Bi–40Sn–2Ag alloy Fig. 1(c) the same phases appeared in addition to the two intermetallic compound are observed, ε-$Ag_3Sn$ at $2\theta = 39.69°$, 72.99°, 76.60° and β-SnBi at $2\theta = 61.14°$, 62.21°. The results of X-ray diffraction analysis show that the quaternary alloy Bi–38Sn–2In–2Ag melt-spun alloy form

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