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# Growth nature of in-situ $Cu_6Sn_5$-phase and their influence on creep and damping characteristics of Sn-Cu material under high-temperature and humidity

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ABSTRACT

This paper describes the morphology and growth nature of in-situ $Cu_6Sn_5$ intermetallic compound (IMC) and their impacts on material properties of an environmental-friendly Sn-0. 7Cu (wt%) material when exposed to high-temperature (85 °C) and relative humidity (85%) environments. A detail microstructural characterization is carried-out by electron microscopy e.g., SEM, EBSD and TEM techniques. In as-cast Sn-Cu material, along with the fine matrix Sn grains, the in-situ $Cu_6Sn_5$ IMC in grain boundary and interior grain appear with a dimension of submicron size and more consistently spread in the matrix. Such fine and uniform dispersed IMC acted as a pinning effect that obstacle the dislocation movement and enhanced their hardness and creep performance. In contrast, after exposing at harsh environment, such in-situ IMC phase morphologies are changed and seemed to a coarse elongated-shaped IMC and reduced their aspect-ratio. These morphological changes negatively impact on the mechanical reliability of Sn-Cu material. A contrast between the as-cast specimen and specimen exposed to high-temperature and relative humidity presents that the electrical resistivity reduces to 12%, where their hardness degrades about 18.5%. However, it is worthy noted that the coarse IMC positively impacts on damping property of Sn-Cu material at given a strain and temperature.

## 1. Introduction

Trend towards miniaturization of modern electronic products with better functionality, higher-packing density and vital reliability concern creates new challenges in microelectronic industry [1,2]. These challenges are associated mainly for two reasons: (i) the advanced electronic products require new and more powerful functions with downscaling their dimensions that lead to increase the current density [2–4], and (ii) the application of environmental-friendly electronic interconnects in advanced electronics increases the difficulty of the interconnection and their reliability issues [5–7]. In general, the electronic interconnect materials provided mechanical and electronic support between the electronic components such as ICs, chips, transistor etc. and printed circuit boards in packaging systems [5,8,9]. As a result, in the last two decades extensive investigations have been going on for manufacturing suitable electronic interconnect material for green electronics that fulfil the requirement of health and environment concern associated with toxic traditional Pb-based electronic interconnect materials [10–12]. However, various type of Sn-based electronic interconnect materials doped with other elements for instance Sn-9Zn (198 °C), Sn-58Bi (139 °C), Sn-Sb (271 °C), Sn-Ag (221 °C), Sn-35Bi-1Ag (187 °C), Sn-0.7Cu (227 °C), Sn-3Ag-0.5Cu (217 °C) and Sn-Bi-In (143 °C) [13–21] are introduced to replace the traditional toxic Sn-38Pb (183 °C) alloy [22,23]. Among them, the lead-free eutectic Sn-0.7Cu electronic interconnect material has been considered as a suitable candidate in green electronic devices due to its various positive aspects e.g., commercially available and cheap, better mechanical property, moderate melting temperature and relatively slow reaction kinetics with Cu pads [9,24]. Furthermore, the in-situ formation of intermetallic compound (IMC) particles i.e., $Cu_6Sn_5$ and $Cu_3Sn$ provides an improvement of thermal cyclic fatigue and wetting behavior [24]. However, it is well documented that the materials properties of Sn-based electronic interconnect fluctuate with operating environments (e.g., temperature and humidity). This creates new challenges in miniaturized electronic product applications of this material, as some operating environments may change the temperature and relative humidity.

Moreover, with the advancement of micro-/nano-systems, electronic devices turn into thinner, lighter, smaller and more functional

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that lead to raise the current density across the interconnections. In packaging rule, each 100 μm or less diameter interconnection carry 0.2–0.4 A current which denoted that the average current density through an interconnection is approximately $2 \times 10^4$ A/cm$^2$ [25]. However, in near future solder bump size will reduce to 1 μm in diameter which will significantly increase the current density up to $1 \times 10^7$–$1 \times 10^8$ joint/cm$^2$ [26]. Additionally, miniaturized electronic interconnection may also raise the temperature gradient up to 3000 °C/cm [27] that value almost three times bigger that the present design of electronic interconnection [28]. This huge temperature gradient affects the mechanical reliability and leads to increase the classical migration-induced failures of electronic interconnection. Therefore, it becomes a critical task to correlate a structural-property relationship of bulk environmental-friendly Sn-based solder materials after exposing at harsh service environments. Numerous research works have been done to understand the chemical kinetic of IMC phase under special aging conditions [29–32]. It is also documented that solid-state aging leads to increase IMC layer thickness which leads to originate the micro-cracks and degrade the mechanical reliability of electronic interconnections [31,32]. Several research groups also investigated the oxidation and growth behavior of IMC phase on different surface-finished printed circuit boards after exposing at high-temperature/relative humidity (85 °C/85% RH) environment [15,33]. However, the microstructural changes and their effects on mechanical reliability (i.e., hardness, temperature/strain-dependence damping capacity, electrical resistivity and creep behavior etc.) are limited when the Sn-Cu electronic interconnect material is exposed into high-temperature/humidity (85 °C/85% RH) environment. This condition is widely used for reliability test of electronic packaging materials because of two main reasons firstly, IMC formations get facilitated at this environment and secondly industrially this environment is adopted for simulating harsh service applications. The present work aims to assess the material properties (i.e., microhardness, creep and damping capacity etc.) after exposing at high-temperature and relative humidity environment, and formulate structural-property relationship that can provide information of mechanical integrity of an electronic product during similar working environments.

Generally, the electronic interconnect materials are normally experienced in harsh service conditions e.g., high temperature, thermal fluctuations, vibration and mechanical shock during their real-life applications [34]. In addition, electronic interconnect materials have relative low melting point ($T_m$), thus the creep behavior is one of the key factors for deformation mode when it exposed to homologous temperature ($T_H = T/T_m$) greater than 0.4. As a result, failure mechanisms associated with the creep become critical factors to design a reliable electronic product since their operating temperatures are usually higher than $0.4T_m$ [35]. Moreover, the miniaturized electronic devices are commonly exposed in a wide range of frequencies or vibrations and this creates a new challenge for sustainability of an electronic interconnects [36,37]. As a result, it is primary importance to understand the energy dissipation ability "so called damping capacity" of electronic interconnects material during temperature and vibration. Thus, a fundamental understand of the structure- property (e.g., hardness, strain-/temperature-dependence damping capacity, electrical resistivity and creep behavior) relationship of Sn-Cu interconnect material is essential when subjected to high-temperature and relative humidity environment.

The aims of this work are to understand growth nature and morphology of in-situ IMC phase and their effects on properties of an environmental-friendly Sn-0.7Cu interconnect materials after exposing at high-temperature (85 °C) and humidity (85%) environment with various durations. Therefore, the specific objectives are to: (a) investigate the microstructural changes of the as-cast and heat-treated specimens especially in high temperature and high humidity (85 °C/85%), (b) elucidate their electrical and mechanical properties with operating temperature, (c) assess the creep behavior and (d) measure damping capacity related to strain amplitude at various operating temperatures.

## 2. Experimental procedures

### 2.1. Starting interconnect material and microstructural observations

An environmental-friendly eutectic Sn-0.7Cu (wt%) electronic interconnect material (Shenzhen Jufeng Solder Company LTD, China) was produced by casting process. Then, the solder bar was sectioned into a cuboid-shaped with a dimension of 40.0 mm × 7 mm × 2.2 mm through a wire cutter with continuous supplying the coolant to minimize the artefacts through heat-generation. After that, samples were ground by metallographic process and finally the polished sample size was approximately 40.0 mm × 6.7 mm × 1.7 mm dimensions. After polishing, one set of samples was exposed in a high-temperature and relative humidity testing chamber (ESPEC-PL- 2FP) with a constant relative humidity (85%) and temperature (85 °C). Finally, structure and crystallographic information of the as-cast and heat-treated specimens were examined through a SEM Hitachi S3400 and EBSD (Carl Zeiss Auriga (FEG)) techniques, respectively. Then Channel 5™ software was used to process the EBSD data. Before investigating the crystallographic information through EBSD, the metallographic specimen surface was milled through a Hitachi IM4000 ion miller with 3.5 kV beam intensity. On the other hand, for TEM observation, the samples were thinned to 150 μm thickness through mechanical polishing. After that the thinned specimens were cut into a 3 mm diameter disk by a punch machine. Then, the disk was placed into a 3 kV argon ion beam (Gatan PIPs) to make a hole at the centre. Finally, TEM observation was carried out using a transmission electron microscopy (TEM, Philips CM200 FEG).

### 2.2. Property evaluations

Microhardness of the as-cast material and material exposed to high-temperature (85 °C) and relative humidity (85%) was measured by a DuraScan hardness testing machine. The average microhardness of each condition was conducted with ten points of 0.3 kg load and 5 s dwelling. Further, investigating the creep performance of as-cast material and material exposed to high-temperature and relative humidity specimens, a 2000 μN constant-load was used for 30 s dwelling with a series of six points by a nano-indentation machine (Hysitron TI-950 Tribo Indenter). Further, the measurement of electrical resistivity was performed by a precision LCR meter (Agilent E4980Az) with various operating temperatures (25 to 100 °C). Finally, temperature and strain amplitude-dependence damping characteristics were measured at 1 Hz frequency through a TA 2980 dynamic mechanical analyser (DMA) at different operating temperatures. The size of the damping characteristic specimen was about 17.5 mm × 6.8 mm × 1.92 mm.

## 3. Result and discussions

### 3.1. Microstructural characterization of Sn-Cu based interconnect material

Fig. 1 shows SEM images and elemental phase analysis of the Sn-Cu interconnect material when exposed at high-temperature and humidity environments at various exposure periods (in days) i.e., (a, b) 0 and (c, d) 40 and (e, f) 60. In the as-cast interconnect material, the in-situ $Cu_6Sn_5$ IMC phase with a dimension of submicrometer was found to be evenly distributed in matrix. Similar results also mentioned in the earlier research work [38,39] and thus, can be recognized from the microscopy analysis as such the distributions shown in Fig. 1(a, b). However, this particle size appeared with coarse structure in certain directions. Furthermore, after exposing at high-temperature and relative humidity of this material, the in-situ $Cu_6Sn_5$ IMC phases turned into a coarse structure as compare to the as-received interconnect material as shown in Fig. 1(c)–(f). It is worthy noted that after exposing at high-temperature and humidity for 60 days, some IMC phase become