

# High-temperature shear strength of lead-free Sn–Sb–Ag/Al<sub>2</sub>O<sub>3</sub> composite solder

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## ABSTRACT

The lead-free Sn–1.7Sb–1.5Ag solder alloy and the same material reinforced with 5 vol.% of 0.3- $\mu$ m Al<sub>2</sub>O<sub>3</sub> particles were synthesized using the powder-metallurgy route of blending, compaction, sintering, and extrusion. The mechanical properties of both monolithic and composite solders were studied by shear punch testing (SPT) at temperatures in the range of 25–130 °C. Depending on the test temperature, the shear yield stress (SYS) increased by 4.8–8.8 MPa, and ultimate shear strength (USS) increased by 6.2–8.8 MPa in the composite material. The strength improvement was mostly due to the CTE mismatch between the matrix and the particles, and to a lesser extent to the Orowan strengthening mechanism of the submicro-sized Al<sub>2</sub>O<sub>3</sub> particles in the composite solder. The contribution of each of these mechanisms was used in a modified shear lag model to predict the total composite-strengthening achieved.

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## 1. Introduction

A great deal of efforts has been devoted to developing lead-free solders as substitutes for the Sn–Pb alloys used in the microelectronic packaging industry. Accordingly, many lead-free Sn-based alloy systems with different alloying elements such as; Ag, In, Cu, Zn, Bi, Ni and Sb have been developed and their microstructures, mechanical properties and wettability have been reported. Among the developed lead-free solders, those containing Sb and Ag could be of interest because of their suitable mechanical and creep properties [1]. The improved properties has been attributed to the solid solution hardening effects of Sb [2,3], formation of the SnSb particles [4], and the presence of Ag<sub>3</sub>Sn intermetallics [5] in the Sn matrix. It has also been shown that adding Sb can refine the Ag<sub>3</sub>Sn precipitate, and thus, improve the mechanical properties and thermal resistance of the Sn–Ag–Sb solders [2,3].

Composite-strengthening has been realized as an effective means for further improving the strength, creep, and fatigue resistance of the tin-based solder alloys [6]. This approach involves incorporating micro/nano sized metallic, intermetallic or ceramic particulates in the solder matrix. Metallic nano- and micro-sized Cu and Ag particles have been used in different Sn-based solders and in all cases significant improvements in the mechanical properties and creep resistance of the composite solders have been reported [7–10]. Nonmetallic oxide reinforcement particles such as Al<sub>2</sub>O<sub>3</sub> [11], SnO<sub>2</sub> [12], TiO<sub>2</sub> [13,14] and ZrO<sub>2</sub> [15] have also been added to

the monolithic solder alloys and their properties have been characterized. These studies have shown that the addition of ceramic particles significantly enhanced the hardness, tensile strength and creep resistance of composite solders. Recently, carbon nanotubes have also been incorporated into the soft solder alloys, leading to an improvement of microhardness, yield stress and tensile strength of solder joints [16,17]. The general conclusion is that, irrespective of the particle type and size, the particulate reinforcements hinder the movement of dislocations thereby increasing the resistance to deformation.

Among many possible ways to assess mechanical properties of solder materials, shear punch testing (SPT) is of some interest. This method 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 such as solder alloys. Further advantages of this localized test include detecting the distribution of shear strength, and avoiding size effects when studying mechanical properties of small bulk of materials such as solders. SPT involves penetration of a cylindrical punch with a flat end into the material against a receiving die at a constant temperature [18]. During the test, variation in punching load with displacement is continuously recorded, and thus the shear stress–shear strain deformation behavior of the material can be obtained. The shear strength of solder materials has been mostly studied by conventional single-lap shear tests [3,19], ring and plug shear test [20], and rarely by SPT. It is therefore, the aim of this paper to study the shear strength of a bulk composite Sn–1.7 wt.% Sb–1.5 wt.% Ag–5 vol.% Al<sub>2</sub>O<sub>3</sub> solder by the SPT method, to be compared with that of the monolithic base material.

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## 2. Experimental procedure

### 2.1. Materials and processing

The powder-metallurgy route was used to synthesize the materials. The composite solder was prepared by blending pre-weighed matrix solder powder with 5 vol.% (2.3 wt.%) of  $\text{Al}_2\text{O}_3$  reinforcement particles. The matrix material used for this study was a Sn–1.7Sb–1.5Ag powder with an average diameter of about  $70\ \mu\text{m}$  obtained from KHPMCO Ltd. The  $\text{Al}_2\text{O}_3$  powder with an average size of  $0.3\ \mu\text{m}$  was purchased from AMPCO Ltd. The base solder powder and the  $\text{Al}_2\text{O}_3$  powder were thoroughly blended in a double cone blender for 20 min to ensure a homogeneous distribution of the reinforcement particles in the solder matrix. The mixture was then hot pressed in a 40-mm diameter cylindrical die under 50 bar at  $145^\circ\text{C}$  and cooled to room temperature. The compacted bars having a melting point of about  $225^\circ\text{C}$  were sintered at  $150^\circ\text{C}$  for 1 h, before being extruded to  $10\ \text{mm} \times 10\ \text{mm}$  bars at the same temperature. The same procedures of compaction, sintering and extrusion were followed for the monolithic solder. The extruded bars were cut into  $1\ \text{mm} \times 10\ \text{mm} \times 10\ \text{mm}$  slices using an electrodischarge wire-cut machine.

The monolithic and composite materials were both studied by scanning electron microscopy (SEM) to examine the microstructure. The specimens were polished with  $0.25\text{-}\mu\text{m}$  diamond paste, followed by polishing on an abrasive-free microcloth. Etching was carried out for 10–15 s using a 5% HCl, 2%  $\text{HNO}_3$ , 3%  $\text{FeCl}_3$  and 90% ethanol solution at room temperature. Energy dispersive X-ray (EDX) analysis was carried out on selected samples to identify the phases.

### 2.2. Shear punch testing

In order to study the effects of nano-particles on the mechanical properties of the solders, the strength was assessed by the shear punch testing, the details of which are given elsewhere [21]. Tests were carried out in the temperature range of  $25\text{--}130^\circ\text{C}$ , corresponding to the homologous temperatures of 0.6–0.80. The 1 mm thick slices of the extruded materials were ground to a thickness of approximately 0.7 mm for the SPT. A shear punch fixture with a 3.175 mm diameter flat cylindrical punch and 3.225 mm diameter receiving-hole was used for this experiment. Shear punch tests were performed using a screw driven SANTAM material testing system with a load cell of 20 kN capacity and a crosshead speed of  $0.25\ \text{mm min}^{-1}$ . The shear punch fixture was located on an anvil below the loading bar and the assembly was accommodated by a three-zone split furnace. Then, the assembly was heated to the test temperature and held for 30 min to establish thermal equilibrium in the testing arrangement before the specimen was exposed to the punch. After application of the load, the applied load  $P$  was measured automatically as a function of punch displacement; the data were acquired by a computer so as to determine the shear stress of the tested materials using the relationship:

$$\tau = \frac{P}{\pi dt} \quad (1)$$

where  $P$  is the punch load,  $t$  is the specimen thickness and  $d$  is the average of the punch and die diameters. Three different samples were tested for each condition and it was observed that the variation in the measured ultimate shear strength values was small.

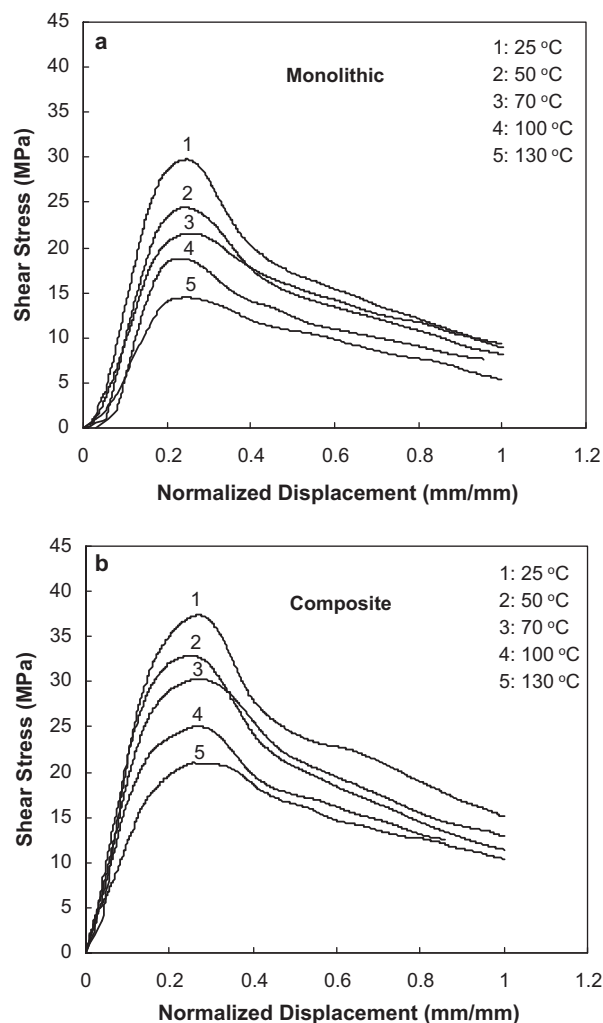


Fig. 1. Shear stress-normalized displacement curves at various temperatures for: (a) monolithic and (b) composite materials.

## 3. Results and discussion

The shear deformation behavior of both materials was investigated by SPT. Although all SPT specimens had similar thicknesses in the range of 0.70–0.75 mm, the punch displacement was normalized to the specimen initial thickness in order to eliminate gage effects. Using Eq. (1), the punching load was converted to shear stress and plotted against the normalized displacement, as shown in Fig. 1a and b for both monolithic and composite solders tested at different temperatures in the range of  $25\text{--}130^\circ\text{C}$ . As can be seen, similar to tensile stress–strain curves, each of the individual curves exhibits a linear part, after which a deviation from linearity is observed. Further loading of the samples results in the load-instability at which a maximum is observed in the stress–displacement curves. The deviation point is taken as the shear yield stress (SYS) and the stress corresponding to the maximum point is referred to as the ultimate shear strength (USS), as exhibited in Fig. 2. In all test conditions studied in this work, increasing the test temperature from  $25\text{--}130^\circ\text{C}$  results in lower SYS and USS values. To have an overall view on the variation of strength with temperature, the SYS and USS data are summarized in Fig. 3a and b, respectively. The results indicate that addition of  $\text{Al}_2\text{O}_3$  particles improves the shear strength of the base alloy at all test temperatures. It can be observed that after adding  $\text{Al}_2\text{O}_3$  particles, the SYS and USS of the monolithic solder increase, respectively,

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