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Indentation depth dependent micromechanical properties and rate dependent pop-in events of $(Au,Cu)_5Sn$

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ABSTRACT

The indentation depth dependent micromechanical properties of $(Au,Cu)_5Sn$ were investigated using nanoindentation. A correction model was proposed to eliminate the overestimation of the hardness for small indentation around the nano-characteristic depth (h_{nano}) by the Nix–Gao model. The indentation hardness of $(Au,Cu)_5Sn$ around h_{nano} is proved to be constant by nanoindentation experiment and our analytic model. Pop-in events of $(Au,Cu)_5Sn$ exhibit obvious rate dependent: pop-in becomes inconspicuous as the loading rate increases, while the first pop-in load and the maximum shear stress increase with the loading rate. Potential mechanism of the rate dependent pop-in events is analyzed based on our experimental results.

1. Introduction

Eutectic Au–20Sn (20 wt%Sn) is an important solder in optoelectronics packaging due to its high thermal conductivity (57.3 W/m °C), excellent high temperature yield strength (165 MPa at 150 °C) and fluxless soldering [1–3]. $(Au, Cu)_5Sn$ intermetallic, ζ(Cu) for short, is ready to form in the reflow process of Au–20Sn on Cu metallization substrates and coarsens seriously during the subsequent service process [1,2]. The intermetallic layers at the solder/substrate interfaces are always considered to be the weakest region in the solder joints [4]. Thus, the evaluation of the micromechanical characteristics of ζ(Cu) is a fundamental issue for the reliability evaluation of Au–20Sn/Cu chip-scale packaging based on Finite Element (FE) simulation. Recently, nanoindentation has been widely used to study the micromechanical properties and micro deformation behavior of Cu_6Sn_5 and other intermetallics in solder joints [4–6]. However, little attention has been paid to the micromechanical characterization of ζ(Cu). Extensive nanoindentation experiments have shown that micromechanical properties of metal materials are indentation depth dependent, i.e. the indentation size effect (ISE) [6–8], which can be understood by Nix–Gao's geometrically necessary dislocation (GNDs) model [7]. But the Nix–Gao model always overestimates the hardness for small indentation [8,9]. Previously, we have modified the Nix–Gao model to describe the ISE of homogeneous duplex Au–20Sn solder based on the maximum allowable density of GNDs, ρ_G^{max}, and enlarged effective storage volume for GNDs [10]. But our previous model does not deal with the case of the hardness for indentation depth around h_{nano} (the nano-characteristic depth). On the other hand, pop-in events in the load–displacement (*L*–*D*) curves are considered to be the elastic–plastic transition and are extensively used to study the micro deformation characterization of metals and ceramics [11,12], which have great significance in improving the accuracy of FE simulation in micro deformation behavior of solder joints [13,14]. Although the influence of the tip radius and the dislocation density on pop-in events has been investigated [11], the effect of loading rate on pop-in events in crystal metal materials has not yet been explored.

In this study, we investigate the indentation depth dependent micromechanical properties of ζ(Cu) and found that pop-in events are loading rate dependent. A revised ISE model was proposed to describe the depth dependent H of ζ(Cu) for multi-scale indentation depth. Potential mechanism of the loading rate dependent pop-in was explained based on our experimental results.

2. Experimental

Eutectic Au–20Sn solder were reflowed on oxygen-free cooper substrates in a vacuum furnace (air pressure less than 1×10^{-2} Pa) without any flux. The reflow process was conducted with a typical peak temperature of 320 °C for 300 s. The interfacial morphologies and the phase compositions were characterized by a JEOL JXA8530F electron microprobe analyser (EPMA). Room temperature nanoindentations were carried out on an Ultra Nanoindentation tester using Berkovich indenter (tip radius $R \approx 100$ nm)

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calibrated by fused silica. The contact area A_c was modified to eliminate the influence of tip radius on H by [15],

$$Ac = 24.56(hc + 0.06R)^2 \tag{1}$$

where h_c is the indentation contact depth. Continuous multi-cycle loading (CMC) test was conducted with 20 different peak loads ranging from 0.5 mN to 15 mN with a hold time of 10 s. Load-control (LC) tests were performed with the peak loads of 2 mN and 10 mN at loading rate ranging from 5 μN/s to 800 μN/s. Displacement-control (DL) tests were performed at different penetration velocities (ranging from 1 nm/s to 9 nm/s) to the maximum depth of 150 nm. The microstructure of the indents was evaluated by a Veeco NanoMan atomic force microscopy (AFM). Each test was conducted at least 5 times to guarantee the repeatability of the results.

3. Results and discussion

Fig. 1(a) shows the cross-section microstructure of the Au–20Sn/Cu joints. A mushroom-shaped layer formed at the interface with a composition of (71.2–70.3) at% Au–(15.5–14.8) at% Cu–(13.3–14.9) at% Sn. Since Cu and Au are chemically alike in nature, Cu atoms diffused into the liquid solder, entered into the Au_5Sn (ζ) lattices and substituted for the Au atoms during reflowing [1,2], forming ζ(Cu) as identified by EPMA. A close analysis of Fig. 1(a) shows that there is a continuous Au-/Cu-rich thin layer next to the Cu side. But the composition of this layer could not be measured precisely due to its thickness less than 0.2 μm. The dimension of ζ(Cu) is large enough to performance nanoindentation tests, as shown in Fig. 1(b). The AFM micrograph (Fig. 1(c)) conforms that no pile-up or sink-in occurred around the indents, implying that Oliver and Pharr's method would not overestimate or underestimate H and the Young's modulus, E, of ζ(Cu) [6].

Fig. 2 shows the result of CMC test. It can be seen that E decreases with the indentation depth (h) until a stable platform, E_0=79.5 GPa, is reached at 450 nm, while H decreases with the indentation depth continuously, indicating an apparent ISE. As can be seen from Fig. 2(b), the Nix–Gao model agrees well with the linear relation between H^2 and $1/h$ for h larger than 275 nm. But the model overestimates H^2 for large $1/h$, namely, small indentation depth. This overestimation can be explained by $\rho_G^{\max}$ [16] and the enlarged effective storage radius fr for GNDs [9]. Previously, we have modified the density of GNDs as follow [10],

$$\rho_G = \rho_G^{\max}\begin{cases} 1 & h < hnano \\ \frac{1}{f^3}\left(\frac{3h_{nano}}{2h} - \frac{h_{nano}^3}{2h^3}\right) & h \geq hnano \end{cases} \tag{2}$$

where $h_{nano} = \tan^2\theta / b\rho_G^{\max}$ (on the order of 50 nm), and $f = 1 + e^{-a/h}$. But the exponential parameters, a, adds extensive fitting flexibility and significantly affects the calibrated value of the characterization length [17]. Thus, we simplify Feng and Nix's method [9] and take f as a constant parameter [8,12]. Based on Nix and Gao's method, we have:

$$\left(\frac{H}{H_0}\right)^2 = 1 + \frac{1}{f^3}\begin{cases} \frac{2h_{micro}}{3h_{nano}} & h < h_{nano} \\ \left(\frac{h_{micro}}{h} - \frac{h_{micro}h_{nano}^2}{3h^3}\right) & h \geq h_{nano} \end{cases} \tag{3}$$

where $h_{micro} = \frac{27M^2}{2} b\alpha^2 \tan^2\theta \left(\frac{G}{H_0}\right)^2$, is the micron-characteristic depth. For indentation depth around h_{nano} ($h \ll_{micro}$), H is a constant value,

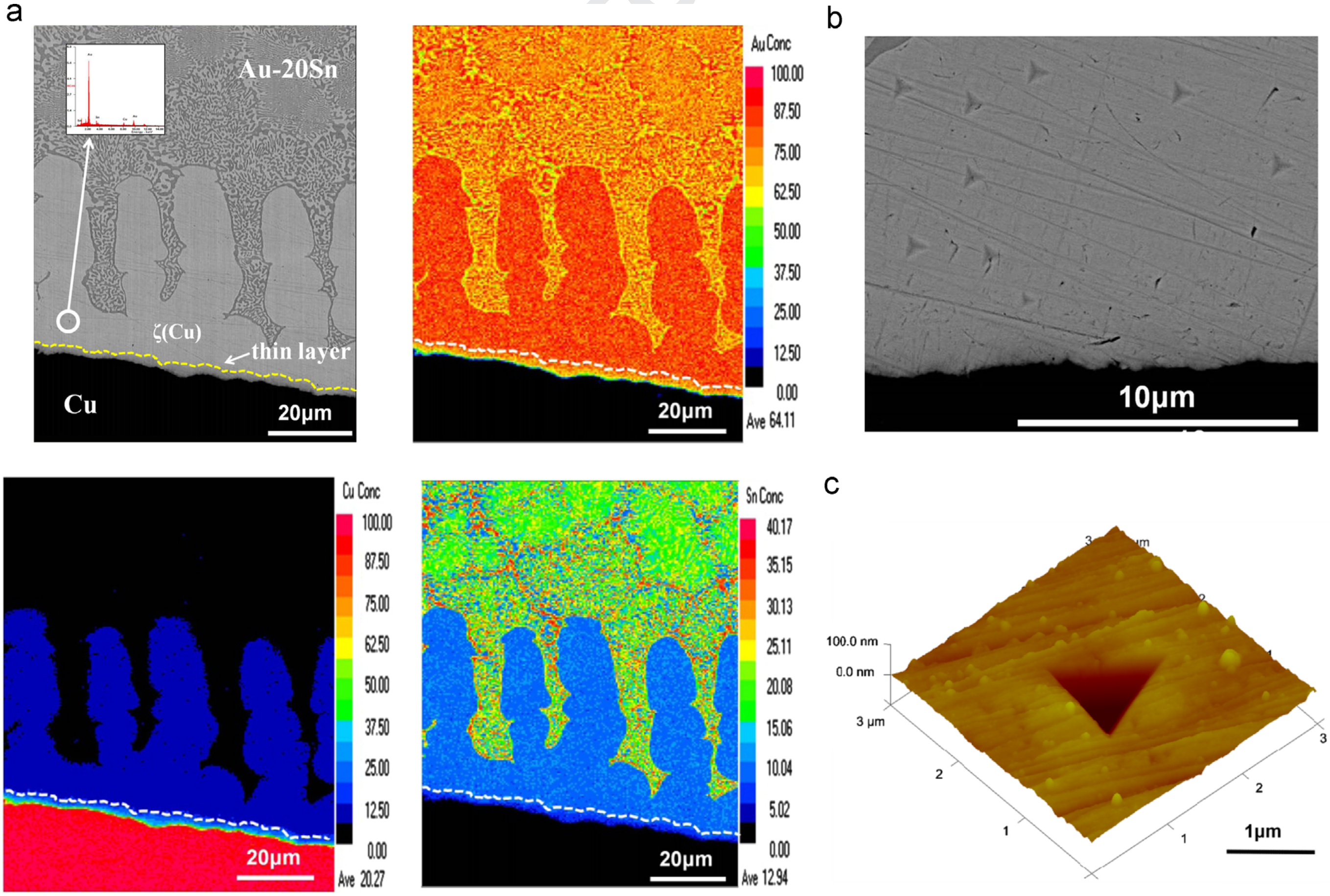

Fig. 1. (a) Elemental maps of the Au–20Sn/Cu interface; (b) SEM image of indentation spots on ζ(Cu); and (c) a typical AFM micrograph of the indents on ζ(Cu) after nanoindentation with the peak load of 10 mN.

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