Journal of Alloys and Compounds 731 (2018) 504–514

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: http://www.elsevier.com/locate/jalcom

Real time resistance monitoring during sintering of silver paste

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ARTICLE INFO

Article history:
Received 4 August 2017
Received in revised form
2 October 2017
Accepted 10 October 2017
Available online 12 October 2017

Keywords:
Silver sintering paste
Non-destructive signals
Sintering process
Real time monitoring

ABSTRACT

Real time resistance monitoring technology is used to study the silver sintering process. Signals of joint resistance show events such as resistance increase to >10 GΩ, abrupt resistance drop from >10 GΩ to <1 kΩ, and gradual resistance drop to <1 mΩ. Based on cross-sectioning of samples at various stages of sintering and differential scanning calorimetry (DSC), we propose a correlation between resistance signal and solvent evaporation, capping agent degradation, and silver sintering. We identified distinct clusters of sintered silver of samples removed from the oven when the resistance drops to ~2.94 Ω.

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1. Introduction

Silver sintering pastes (SSPs) are used as die attachment material in microelectronics packaging, especially for power devices [1]. Some SSPs have lower bonding temperature than most solders, and can survive a higher temperature than some solder joints once bonded [1]. In some work [2–4], SSPs are added to epoxy based isotropically conductive adhesives (ICAs) to adjust the material property such as the conductivity.

Silver sintering pastes are typically mixtures of solvent, capping agent, and silver micro and/or nano particles. The silver particles are usually covered with a capping agent to avoid low temperature sintering. The silver sintering process is, for example in Ref. [5], a sequence of solvent evaporation, capping agent degradation, and silver sintering in elevated temperature according to the thermal gravimetric analysis (TGA). Generally, the smaller the particle sizes, the lower the sintering temperature needed [6]. Magdassi et al. have achieved room temperature sintering of 10 nm silver particles [7].

Despite the advantages of low process temperature, challenges remain for the current SSPs, such as the requirement for pressure to densify the joint, which increases the risk of cracking the brittle semiconductor, and thus can reduce the production yield [8]. To overcome such challenges, better SSP sintering process knowledge is required.

To improve the understanding of SSP sintering processes, a common way is to prepare samples at various sintering temperatures and times [5–7]. The samples are analyzed with microscopic images [5–7], thermal/electrical conductivity measurement [5], and shear/tensile test [6].

For example, Hu et al. [6] studied the tensile strength of silver sintering paste joint between copper wire to copper pad obtained at five temperatures between 100 °C and 300 °C. They found that the tensile strength increases with temperature for 250 μm wire, and remains unchanged with 50 μm wire.

Alternatively, in-situ monitoring can provide real-time information for the sintering process, and transient phenomena can be studied with a fast enough sampling rate. A common in-situ monitoring technology to study SSP sintering process is TGA [1,5,9,10].

For example, Lu et al. [5] used derivative thermogravimetric analysis to study six SSPs with different capping agents, and the same solvent α-Terpineol. For all the SSPs studied, the capping agent degradation happens after or in the end of solvent evaporation. Specifically, silver 2-ethylhexanoate decomposes at the lowest temperature of 190.3 °C. After 350 °C/30min processing, sintered silver is observed if the capping agent degradation temperature is lower than 250.2 °C.

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https://doi.org/10.1016/j.jallcom.2017.10.077
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Lately, resistance monitoring technology has been applied to study the curing process of epoxy-based ICA [11–14], while such study on SSP sintering process is not found. Inoue et al. [11,13,14] found that the change of electrical conductivity can not only be caused by curing reaction of the binder molecules, but also the conductivity change of the inter-filler chemicals, by comparing resistance signals with results from free-damped oscillation method.

Xiong et al. [12] obtained resistance-time curve with ICAs of 65 wt% silver fill loading. The resistance value becomes detectable (<2.0 MΩ) at 27 min. In general, the resistance drops gradually, and it reaches 1.4 kΩ at 40 min, and 8.8 Ω at 80 min, respectively. After 80 min the resistance remains unchanged. The resistance decrement is explained with the epoxy resin shrinkage leading to an increase in the contact area between the fillers, which promote the formation of conductive pathways between the filler particles during the curing process.

In summary, in previous studies with resistance measurement for ICA curing process, the resistance values are measured at a few (<20) curing times and/or temperatures [5,11,12,14–17], and/or monitored with a slow sampling rate [12]. Some studies [3,11–14] have missing resistance values because of the range limit of the technology applied. For example in Ref. [3], the maximum measurable resistance was 100 MΩ for the method used. When monitoring the ICA specimen resistance during curing, it exceeded the limit of 100 MΩ before the temperature reached 90 °C, and valid data was therefore not detected.

In this work, we study the sintering process of silver paste with an improved real time resistance monitoring technology based on the method used previously for a solder reflow study [18]. We present a larger range of resistance measurement (10^{-5}-10^{11} Ω), and provide a more continuous measurement than previous methods [3,5,10].

2. Experimental

2.1. Materials

The sample material is a commercially available SSP, Loctite Ablestik SSP 2020 [19–21]. The ingredients are shown in Table 1 [22]. The SSP is a silver sintering paste containing a mixture of silver particles, 2-(2-butoxyethoxy)ethyl acetate as capping agent, and 1,1′-oxydipropan-2-ol as solvent [23]. The optimal storage temperature is ≤40 °C [24].

2.2. Sample preparation

The SSP is dispensed with a Nordson Ultimus I automatic dispenser, with parameters shown in Table 2. The substrate is ceramic side-brazed dual in-line packages (Spectrum CSB02801). Fig. 1 shows the top view optical image of the as dispensed samples. After dispensing, to minimize sample change such as room temperature sintering, or solvent vaporization, the sample is either used immediately, or covered and stored in a freezer at −40 °C. To minimize thawing issues such as the condensation of the moisture from air on the samples, the cover is always kept on the substrate before the substrate reaches room temperature. The cover is ceramic, and a double sided Kapton tape is used to attach the cover to the substrate.

Fig. 2 shows the sample design. Terminal numbers are indicated and the 12 resistance signals R1-R12 are defined.

Table 1
SSP ingredients [22].

Chemical name	wt.%
silver flakes and spheres	balance
2-(2-butoxyethoxy)ethyl acetate (capping agent, boiling point 245 °C, flash point 102 °C)	1–5
1,1′-oxydipropan-2-ol (solvent, boiling point 232.8 °C, flash point 138 °C)	1–5

Table 2
Dispensing parameters.

Tip inner diameter [mm]	Speed [mm/s]	Pressure [psi]
0.33	4	15

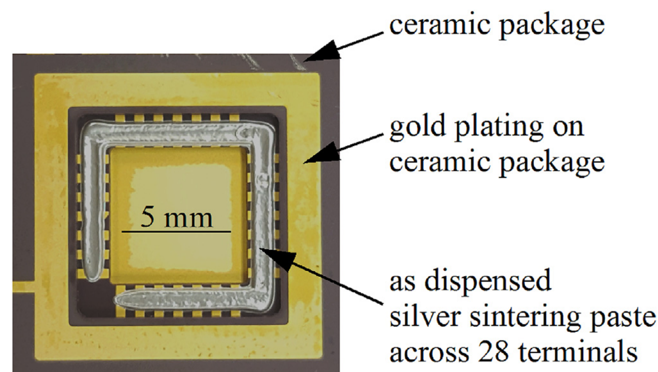

Fig. 1. A top view optical image of the ceramic package with the as dispensed SSP.

2.3. Sintering and resistance measurements

The sintering profile recommended by the manufacturer is 4 h at 175 °C. The oven is preheated to the sintering temperature. After the sample is connected, the resistance measurement starts running for 9 min in room temperature, with cover removed at 5 min, before the sample unit is placed into the oven. Four hours after placing the sample into the oven, the oven stops heating, and the samples are left inside the oven to cool down.

For the test setup, the oven, multiplexer, connecting materials, and temperature measurement are the same as in Ref. [18]. In addition, an Agilent B2911A precision source/measure unit and an

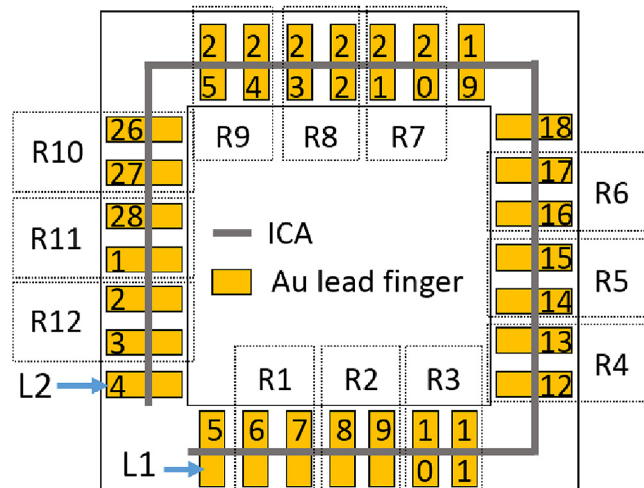

Fig. 2. SSP resistance measurement scheme.

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