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Indentation creep study of lead-free Sn–5%Sb solder alloy

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Abstract

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 temperatures in the range 298–405 K. Based on the steady-state power law creep relationship, the stress exponents were determined for the cast and wrought materials. These exponents, depending on the testing temperature, were in the range 4.8–2.9 and 13.7–6.0 for the wrought and cast conditions, respectively. The activation energy of 46.8 kJ mol^{-1} which is very close to the activation energy for grain boundary diffusion of β-Sn, together with a very fine grain size of 4.5 μm and a uniform distribution of fine SnSb particles, may suggest that the dominant creep mechanism in the wrought condition is grain boundary diffusion. However, for the cast material with a coarse grain size of 280 μm, the *n*-values of 13.7 to 6.0 and the activation energy of 66.5 kJ mol^{-1} are indicative of a dislocation creep mechanism over the whole temperature range investigated.

Keywords: Indentation creep; Lead-free solder; Hardness; Stress exponent

1. Introduction

One of the key issues in the electronics packaging industries is the serious environmental problem associated with the lead-containing solders. This has resulted in extensive research work to develop lead-free solders with suitable soldering characteristics and mechanical properties. Many lead-free Sn-based alloy systems with different alloying elements such as; Ag, Zn, Cu and Bi have been developed and their microstructures, mechanical properties and solderability have been investigated [1–4]. Antimony has also been used as the alloying element in the tin-based systems to provide suitable substitutes for Sn–Pb solder alloys. One of these materials is the near-peritectic composition, Sn–5%Sb, with a melting point of 245 °C and a solder–substrate contact angle of about 43° [5,6]. Solders in joints usually work at high homologous temperature because of their low melting temperatures. In this temperature range, creep is the most important deformation mechanism, and the stress and strain concentrations at the solder joints must be effectively relaxed by creep to guarantee the solder joint reliability [7]. Accordingly, the creep study of tin–antimony alloys has received a great deal of attention [5,6,8,9]. It has been shown that while antimony atoms in solution have only a minor effect on the creep resistance, alloys with higher concentrations of antimony contain SnSb precipitates, which provide a significant strengthening effect that reduces the creep rate of the material especially at temperatures below 100 °C.

The indentation creep process can be defined as the time dependent penetration of a hard indenter into the material under constant load and temperature. The variation in the indentation size, expressed either as a change in diameter (Brinell test) or diagonal length (Vickers test), is monitored with dwell time. Thus, the time-dependent flow behavior of materials can be studied by these simple hardness tests. This can be particularly advantageous when the material is only available as small test pieces or there are some difficulties with the machining of samples made of very soft materials. Therefore, the indentation creep tests, regarded as a quick, simple and non-destructive procedure to extract information on the mechanical behavior of materials, greatly reduce the effort for sample preparation [10,11].

The high temperature indentation creep testing method has been widely used in the creep study of intermetallics and refractory materials. These tests referred to as "hot hardness tests" have been employed in the creep testing of different zirconium alloys [12–14], intermetallics [11,15,16] and special steels [17,18]. Creep characteristics of solder alloys and other soft materials

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such as Sn–37.8%Pb [19,20], Pb–9%Sn [21], Sn–3.5%Ag [22], Sn–40%Pb–2.5%Sb [23] and Pb–(1.25–4.5)%Sb [24] have also been investigated by indentation technique. On the other hand, the creep behavior of the lead-free Sn–Sb alloys is mostly studied by conventional creep tests [2,5,8], room-temperature indentation creep tests [25,26], impression creep [27] and in one case by the automated ball indentation (ABI) method [5]. Thus, the high-temperature indentation creep of Sn–5%Sb solder has not been studied before and would be attempted in this investigation.

2. Analysis of indentation creep

Various models have been proposed for the analysis of indentation creep data. Those suggested by Mulhearn and Tabor [28], Atkins et al. [29], and Li et al. [30] are among these models. More recently, Sargent and Ashby [31] carried out hot hardness tests on a wide range of materials and proposed a dimensional analysis for indentation creep. According to their model, for steady-state creep, the high temperature creep rate $\dot{\varepsilon}$ is described by the power law of the type:

$$\dot{\varepsilon} = A\sigma^n \exp\left(\frac{-Q}{RT}\right) \tag{1}$$

where σ is the applied stress, A a material parameter, n denotes the stress exponent, Q the activation energy, T the temperature, and R is the universal gas constant.

The displacement rate of an indenter has been derived as

$$\frac{du}{dt} = \left[\frac{\dot{\varepsilon}_0}{C_2}(\sqrt{A})\right]\left[\left(\frac{C_1}{\sigma_0}\right)\left(\frac{P}{A}\right)\right]^n \tag{2}$$

where A is the projected area of indentation, C_2 a constant, $\dot{\varepsilon}_0$ the rate at a reference stress σ_0, n the stress exponent and P is the applied load. For a pyramid indenter the penetration is proportional to $\sqrt{A}$, i.e.,

$$u = C_3\sqrt{A} \tag{3}$$

Differentiating Eq. (3) with respect to time and substituting into Eq. (2) gives:

$$\frac{dA}{dt} = C_4\dot{\varepsilon}_0 A\left(\frac{P}{A}\sigma_0\right)^n \tag{4}$$

where C_3 and C_4 are constants. When P is held constant, Eq. (4) can be rewritten as

$$\left(\frac{1}{H_V}\right)\left(\frac{dH_V}{dt}\right) = -C_4\dot{\varepsilon}_0\left(\frac{H_V}{\sigma_0}\right)^n \tag{5}$$

From Eq. (5), a plot of $\ln[(-1/H_v)(dH_v/dt)]$ versus $\ln H_V$ at a constant temperature has a slope of n. A plot of $\ln[(-1/H_v)(dH_v/dt)]$ versus $\ln t$ can superimpose all the data for a single material onto a single master curve having an intercept of $1/n$ at $t = 1$ min. Sargent and Ashby have also derived the following relationship between indentation hardness and dwell time:

$$H_V(t) = \frac{\sigma_0}{(nC_4\dot{\varepsilon}_0 t)^{1/n}} \tag{6}$$

where $H_V(t)$ is the time dependent hardness. Therefore, from Eq. (6) the slope of a plot of $\ln H_V$ against $\ln t$ at a constant temperature is $-1/n$. The activation energy is calculated from the plot of $\ln t$ against $1/T$ at constant hardness, the slope of which provides Q/R.

3. Experimental procedure

3.1. Materials and processing

The material used was a Sn–Sb alloy containing 5 wt.% Sb. It was prepared from high purity tin (99.99%) and a Sn–20Sb master alloy melted in an electrical furnace under inert argon atmosphere, and cast into 120 mm × 30 mm × 12 mm slabs. In order to ensure that the slabs had similar initial microstructures, the slabs were homogenized at 450 K for 24 h. Some of the homogenized cast slabs were cut into 2 mm × 30 mm × 12 mm slices using an electrodischarge wire cut machine and some others were rolled to a 83% reduction at room temperature ($T > 0.5T_m$) in order to generate a homogeneous fine grained material with a grain size of about 5 μm and without the initial as cast structure. The structure of the cast and wrought materials was examined by optical microscopy and the average grain size was measured. Image analysis was carried out on selected samples to measure the particle volume fractions for both experimental conditions.

3.2. Indentation creep tests

Square samples with edges of 20 mm and a thickness of 2 mm were cut from both cast and rolled materials. These samples were polished and tested in a home-made high-temperature Vickers hardness testers. The testing arrangement for the indentation creep test is shown in Fig. 1. The Vickers indenter was mounted in a holder which was positioned in the center of the vertical loading bar. The specimen was located on an anvil below the loading bar. The stainless steel container was almost filled by ethylene glycol solution covering the whole specimen and indenter. The bath was then heated by resistive coil heating and stirred by argon gas bubbling through the liquid. The bath temperature was continuously monitored with a thermocouple and maintained to within ±2 °C. Indentation hardness measurements were made

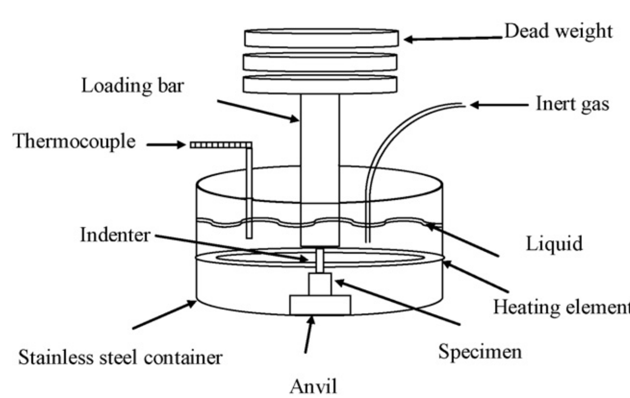

Fig. 1. Schematic illustration of the indentation creep apparatus.