

# The Study of Creep Properties of Sn-5wt%Sb Lead-Free Solders with Annealing Temperature

## ABSTRACT

Sn-5wt%Sb is one of the materials considered for replacing Pb-bearing alloys in electronic packaging. The creep tests were conducted to investigate the effect of the heat treatments on the mechanical properties of Sn-5wt%Sb lead-free solders. Samples were heat treated at temperatures of 313, 333 and 353 K to produce a range of different grain sizes. **Tow-load creep tests were carried out at each temperature for the wire samples to alloys.** The results obtained show that there is a relationship between the heat-treatment temperature and the microstructure and that this, in turn, affects the creep properties of the alloys. From the steady state creep rate the stress exponent is described in terms of the heat treatment temperatures. The stress exponent (n) were determined to clarify the deformation mechanism. Based on the n values, it is suggested that the rate controlling creep-deformation mechanism is dislocation climb. Comparisons are made with all alloys on the creep resistance of solder alternatives. **The effect of annealing temperature against the strain rate indicated that in increasing annealing temperature, the strain rate is increasing, might be due to the increasing in the grain size.** This study revealed that the solder alloy Sn-5wt%Sb have potential to give a good combination of higher creep resistance and rupture time.

**Keywords:** *Lead-free solder, Sn-Sb alloy, Heat Treatments, Creep deformation, Stress exponent, Microstructure*

## 1. INTRODUCTION

Lead-tin alloys are the dominant solders used widely in manufacturing because of their unique combination of material properties and low cost. But, due to the toxicity of lead present in these solders, environmental and legislation trend to reduce or eliminate the use of lead from a wide variety of uses. So, many studies tend to intensify the work to form a new lead-free solder alloy with improved properties [1–2]. There is a set of the binary alloys chosen as candidates for lead-free solder: Sn–Bi, Sn–Ag, Sn–Zn, Sn–Cu and Sn–Sb, in which Sn is the principal component. Alloys of tin with antimony have received the amount of attention because of their high specific gravity and low melting point. These alloys contain between 3.5 and 15% antimony, much of which combines with some of the tin presents to form an intermetallic compound SnSb that improves its mechanical properties [3–4]. This alloy system was subjected to many studies [5–6], including the effect of rapid solidification as well as the effect of bismuth addition on structure and properties of this alloy system. Heat treatment technique of alloys has a great effect on the resulting grain size [7]. It is highly

desirable to have a solder that has a low melting point, high creep resistance, mechanical strength, ductility, and so on. Determinations of these properties are necessary for structural analysis and evaluation of thermal fatigue life of the solder joint. In these fatigue processes, the creep mechanisms play an important role, because of high homologous temperatures involved [8,9].

The antimonial tin solder (Sn95Sb5) alloy with a near peritectic composition has the advantages of good room-temperature creep resistance and mechanical strength, although its melting point is high 518 K compared to that of the popular lead – tin eutectic 456 K [10, 11]. Therefore, the knowledge of the creep behaviour of tin–antimony alloys under long term mechanical loading is essential for the evaluation of their service lifetime.

The present work aims to study the effect of heat treatment on the creep deformation behaviour of the Sn-5wt.%Sb.

## 2. EXPERIMENTAL WORK

Sn-5wt%Sb lead-free solder alloy was examined. The compositions were prepared from high purity

(99.99%) component materials. All components were melted under vacuum. The casting was done in a graphite mould and the ingot (rod of 1 mm diameter) was swaged to form wire samples of 1 mm diameter and 80 mm in length. Each alloy samples were annealed for 4 hr at a temperature of 393K, and then leave in a furnace to cold slowly into the temperature of 298K. Tensile creep tests were carried out with a computerize tensile testing machine. Creep tests were carried out at temperatures (313-333-353 K) under different applied stresses ranging from 6.24 to 9.98MPa.

## 3. RESULTS AND DISCUSSION:

### 3.1 Creep Results

In order to determine the temperature dependence of the deformation behaviour of the tested solder alloy, the creep properties of the Sn-5wt%Sb solder alloys was investigated at temperatures in the range temperature (313-333-353 K) with a range of applied stress (6.24 -9.98 MPa). Fig. 1 (a, b) shows a typical set of creep curves for lead-free solder alloy wire specimens annealed at 373K, all tested solders show the expected creep characteristics. The creep curves consist of a transient stage, which is characterized by a creep rate that decreases with time followed by a steady-state stage, where the creep rate is essentially constant. The figure shows that the creep rate increases with increasing the annealing temperature, However, the results show that the Sn-5wt% Sb alloys had the lowest creep resistance and highest creep rate with increasing annealing temperature due to the lowest strain hardening processes.

Figure 1(a &b) shows the creep curve for Sn-5wt.% Sb alloy at different temperature annealing. It shows an increase in the strain as the temperature increases in different amounts. The same behaviour was observed in all samples. The variations in creep rate  $\dot{\epsilon}$ , suggest a fundamental change in the internal stress of the alloy sample during the time. This implies that the hardening of the matrix was recovered immediately and balanced at an extended deformation rate [12].

The steady-state creep rate,  $\dot{\epsilon}$ , was determined from the slopes of the linear portions of the strain time curves of the type shown in Fig. 1(b), the results of creep tests conducted at 313, 333 and 353 K over the range of stress. The minimum creep rate

is related to the applied stress,  $\sigma$ , and temperature,  $T$ , by the following relation [11]:

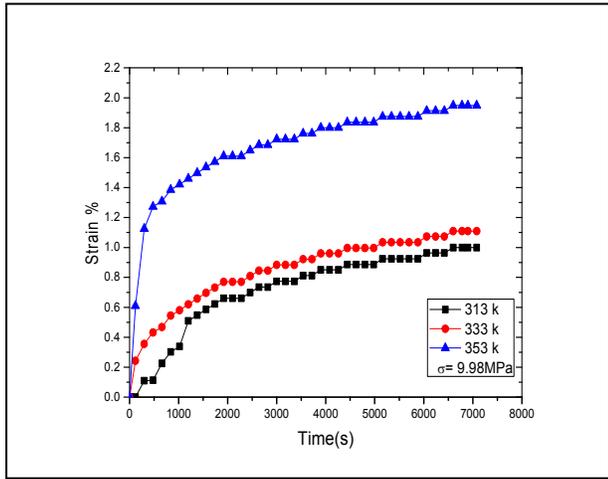


Figure 1. (a.): Comparison creep curves at constant applied stress  $\sigma = 9.98 \text{ MPa}$  for Sn-5wt%Sb,  $T_a = 313 \text{ K}, 333 \text{ K}, 353 \text{ K}$

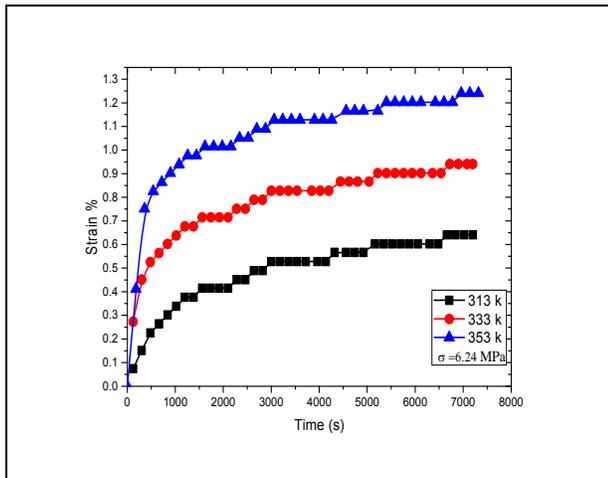


Figure 1. (b): Comparison creep curves at constant applied stress  $\sigma = 6.24 \text{ MPa}$  for Sn-5wt%Sb  $T_a = 313 \text{ K}, 333 \text{ K}, 353 \text{ K}$

$$\dot{\epsilon}_s = A\sigma^n \quad (1)$$

where  $A$  is the material-dependent constant, and  $n$  is a parameter of the material that could give useful information on the creep controlling mechanisms. ( $n$ ) can be evaluated from the slope

of the  $\ln \dot{\epsilon}_s$  against  $\ln \sigma$  as plotted in Figure 2 and according to the following equation,

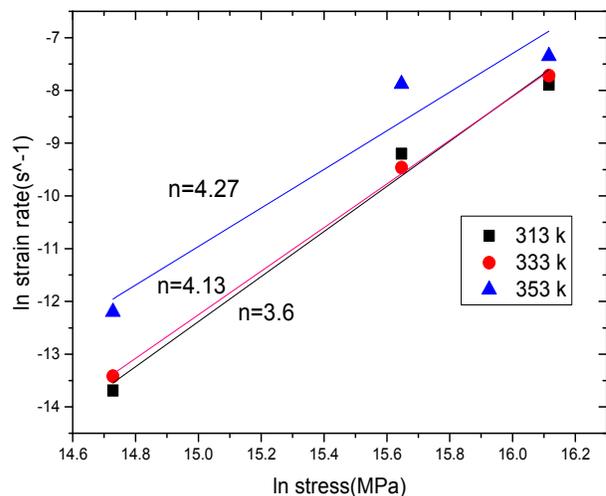
$$n = (\partial \ln \dot{\epsilon}_s / \partial \ln \sigma) \quad (2)$$

The values of the stress exponent are given in Fig. 2 shows the relationship between  $\ln \sigma$  and  $\ln \dot{\epsilon}_s$  (sec-1) for the alloy at different annealing temperature (313K, 333K, and 353K). Figure 2 shows the resulting stress exponent ( $n$  creep) vs. The stress exponent, which gives an indication of the rate controlling mechanism, that, as the annealing temperature increases from 313K to 353K a significant increase in the stress exponent occurs which has been related to the dissolution of the  $\beta$ -the phase during high temperature. At all annealing temperature (313-333-353 K) the stress exponent ( $n$ ) is in the range (3.65-4.27) for all alloys. These values of the stress exponent ( $n$ ) suggest that the creep deformation mechanism is due to the dislocation climb mechanism dominated this well within the range for dislocation climb control where is (3 -8) this agreement with [14 -15].

The  $n$  value also as the annealing temperature increases from 313K to 353 K significant increase in the stress exponent occurs which has been related to the dissolution of the  $\beta$ - phase during high temperature. The difference in the stress exponent values suggests that the creep deformation in the solder alloy is influenced by the formation of the IMCs that could restrict the dislocation climb at the high annealing temperature. Furthermore, creep resistance was more evident at lower testing temperatures.

### Strain rate sensitivity index $m$

The strain rate sensitivity index is a very important



parameter in characterizing

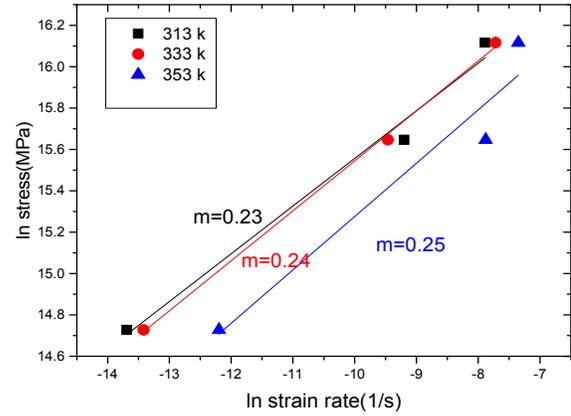
Figure 2. The relationship between  $\ln\sigma$  and  $\ln\dot{\epsilon}_s$  (sec-1) for alloys at different temperature.

structural superplastic deformation. The  $m$  value also reflects the inhibition ability of local necking, the larger the  $m$  value the stronger the inhibition ability of necking, so elongation is much higher and ductility is better. The strain rate sensitivity index is a very important parameter in characterising structural superplastic deformation. There are various methods of measuring  $m$  but all these methods lead to a variation of  $m$  value. This parameter is calculated using the equation (3). [16].

$$m = (\partial \ln \sigma / \partial \ln \dot{\epsilon}) \quad (3)$$

As depicted in the experiment procedures, the strain rate sensitivity index,  $m$ , can be calculated from the designed **Stressed Receiver Conformance Test (SRCT)**. According to the test results, the  $m$  value is not a constant during the completely tensile process. Generally, with the increase of the elongation deformation, the  $m$  value becomes **higher**, that means the  $m$  is strain-dependent. Figure 1(b) illustrated the procedure of how to calculate the SRSI, i.e. the  $m$  value, using a typical load-strain curve obtained from the SRCT test. The values of the strain rate exponent show the relationship between  $\ln \sigma$  and  $\ln \dot{\epsilon}$  (sec-1) for the alloy at a different annealing temperature (373K, 423K, and 448K) (Fig. 3). **The  $m$  values** are obtained using instantaneous values measured in the steady-state region of  $\ln \sigma - \ln \dot{\epsilon}$  curves. The slope of the  $\ln \sigma - \ln \dot{\epsilon}$  plot is measured using a curve-fitting procedure and carrying out **differentiation (Fig. 3)**. On the other hand, the slight increase for the  $m$ -value with temperature might be due to easier dislocation movement and diffusional processes at higher temperatures. During deformation, the  $m$  value represents the capacity of the material to resist necking and affects the overall deformation and stability [17]. Hence, the Sn-5wt%Sb solder has **high** resistance to necking. Generally, superplasticity is observed when  $m$  value is equal to or greater than 0.3. The  $m$  values for Sn-5wt%Sb are less than 0.3 in the temperature rang investigated in this study. There for, superplasticity behaviour did not occur in the Sn-5wt%Sb solder

alloy. In addition, them value of sample solder alloy increased significantly with the increases of the temperature, this indicates that the necking resistance of the sample alloy increased with



increasing temperature.

Figure 3. The relationship between  $\ln\dot{\epsilon}_s$  and  $\ln\sigma$  for alloys at different temperature.

### The Effect of the Heat Treatments on the Grain Size

The overall properties of the solder depend on the formed IMCs and the microstructure of the solder. It is therefore important to determine the types and morphology of formed IMCs.

$$\beta \cos \theta = \frac{K\lambda}{D} + 4\epsilon \sin \theta \quad (4)$$

Where ( $\beta$ ) is the diffraction ray's breadth, ( $\theta$ ) is the Bragg's angle and ( $\lambda$ ) is wavelength of used X-ray.[16]. In order to obtain ( $D$ ) and ( $\epsilon$ ), the relation between  $\beta \cos \theta$  and  $4\epsilon \sin \theta$

should be plotted. The values of  $\beta \cos \theta$  on y-axis were plotted as a function of  $4\epsilon \sin \theta$  on x-axis, and from the linear fit of the data.

The slope of the fitted lines represents average crystallite size ( $D$ ) and the y-intercept represents the microstrain ( $\epsilon$ ). In order to verify effects of annealing temperatures on the microstructure of Sn-Sb solder alloys. The variations of grain size were calculated from X-ray data using equation (4). The effect of

increasing temperature on the particles size shown in Fig (4) It can be seen that the particle size increases gradually with increasing temperature the increase in grain size can be attributed to the dissolution of pinning particles from the grain boundary at a higher temperature which allowed grain growth to occur. In the heating process when the particles are formed they collide and either coalesce with one another to form a large particle. The process, which occurs, depends upon the temperature and available energy that is why particle size increases with increasing temperature. On the other hand, since annealing temperature was set beyond the desired temperature of recrystallization, the new strain-free grains are heated that leads to a progressive increase in grain size. Furthermore, at the higher annealing temperature, second-phase particles, which inhibit grain growth, will dissolve progressively and results to coarsening of grain [18,19, 20].

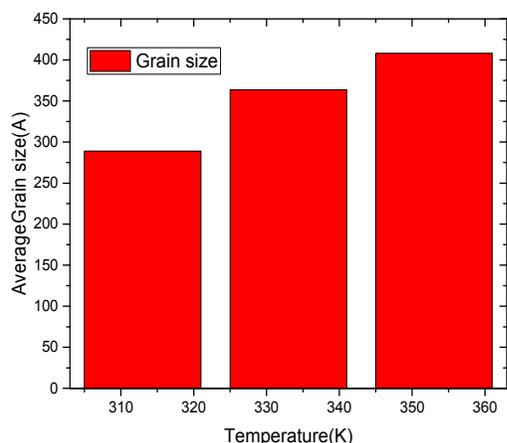


Fig (4). Variations of grain size at a different temperature.

#### 4. CONCLUSIONS

The effect of annealing temperature on the structure, creep behaviour, of the Sn-5wt%Sb lead free solder alloy, was investigated, the result showed that: -The creep rate for all alloys was increased continuously with increasing the annealing temperature. Sn-5wt%Sb at the lowest temperature had the highest creep resistance this is due to the fine grain size (small grain size), on the other hand by increasing the annealing temperature the grain size increases which may be induced through the cold drawing process. From XRD patterns showed that the alloy

was polycrystalline for all temperatures and the cubed SbSn intermetallic compound is precipitated within the -Sn matrix in the Sn-5wt%Sb-based lead-free solder alloys.

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