

## Creep and Structure Parameters Near the Transformation Temperature of Sn-1 wt% Pb Alloy

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*Sn-1 wt % Pb alloy is studied in tensile creep at different temperatures between 323 and 383K and constant stresses varying between 14.1 and 16.0 MPa. Both the creep and the microstructure parameters show one transformation temperature at 353K. The transient creep parameters  $\beta$  and  $n$  are affected by both the applied stress and the working temperature and have values ranging from  $1.36 \times 10^{-4}$  to  $34 \times 10^{-4}$  and from 0.878 to 0.975, respectively. The activation energies of the transient creep around 353K are found to be  $(50 \pm 4)$  and  $(86 \pm 7)$  kJ/mol, which identify dislocation mechanisms. The activation energy of the steady state creep has values of  $(79 \pm 5)$  and  $(106 \pm 9)$  kJ/mol around the transformation temperature which characterizes Coble creep and viscous grain boundary sliding mechanisms, respectively. The X-ray diffraction analysis shows that both the ratio  $(c/a)$  and the integral intensity  $(I)$  of the Sn-rich phase have minima at the transformation temperature.*

**Introduction:**

The low hardness and consequent low mechanical strength of pure tin make it unsuitable for use as a bulk material for construction unless strengthened by hardening alloying elements [1]. Alloying tin with lead is of particular importance to soft solders specially its eutectic composition so, many workers have studied the physical properties of Sn-Pb eutectic solder [2 to 4]. The alloy containing over 90% Sn is known as "pewter". The creep behavior around the transformation temperatures of some important pewters Sn-0.5 and 9 wt.% Zn and Sn-5 wt.% Bi has been investigated [5,6], respectively. The addition of 1 wt.% Pb to 99 wt.% Sn is to reduce progressively the liquidus temperature of the melt [1] to obtain an alloy with melting point (230°C) which has industrial importance. Tin-lead pewter can be used instead of wooden and pottery items for tableware and other household purposes. It is widely used for utensils, such as tankards and goblets or decorative items like plates and candle-sticks or costume jewellery [1].

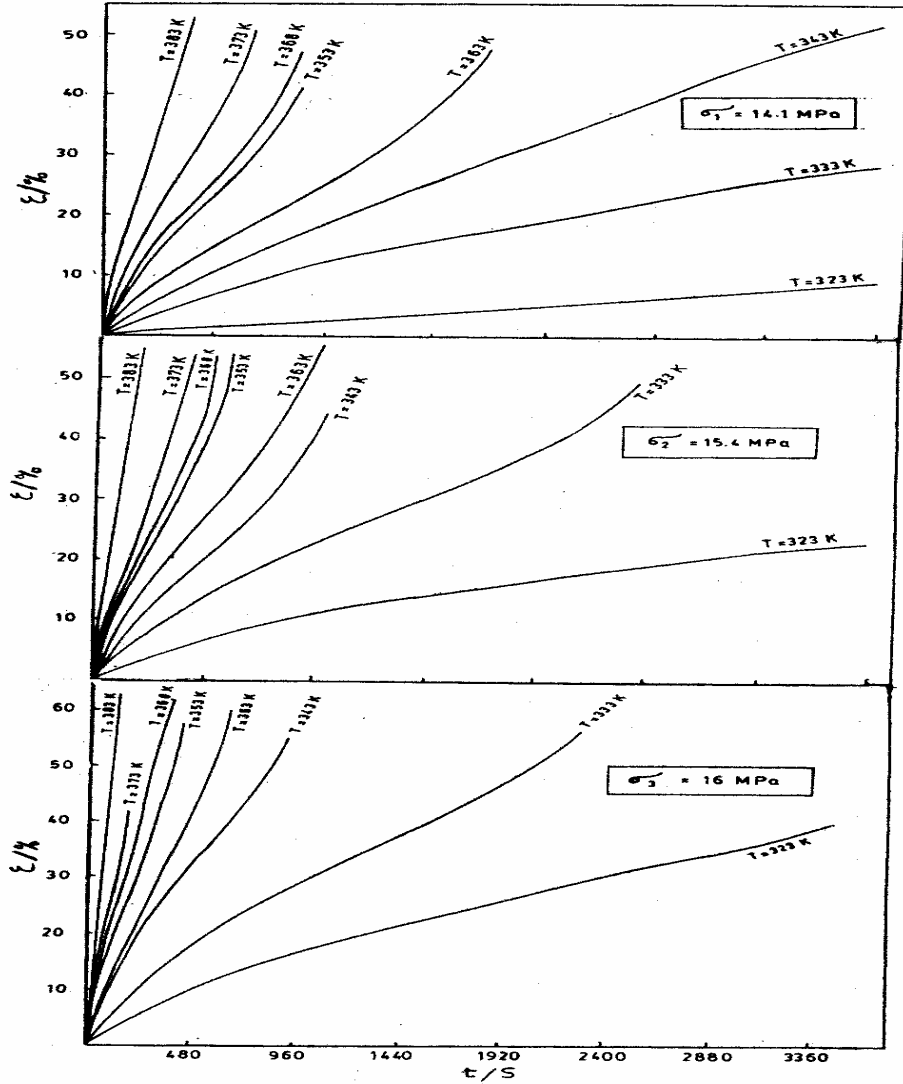
In spite of its importance, this alloy has not shown any interest in the study of its mechanical properties throughout the previous publications. So the aim of this work is to study the variations in the mechanical parameters of this alloy at different temperatures, and to investigate the effect of the microstructure on the creep parameters during transformation.

**Experimental Procedure:**

The Sn-Pb alloy containing 1 wt % Pb was locally prepared from high purity Sn (99.99%) and Pb (99.99%). The material was then swaged in the form of wires of length 10 mm and diameter 1 mm. Wire samples were annealed for one hour at 408K to eliminate the cold work introduced during swaging and were then slowly cooled to room temperature at a cooling rate  $\dot{T} = 6.11 \times 10^{-3}$  K/s. Such heat treatment usually allowed the double phases ( $\alpha$  and  $\beta$ ) to exist [7]. Tensile creep tests were then performed with constant stress conditions at temperatures ranging from 323 to 383K. The accuracy of temperature and elongation measurements are of the order of  $\pm 1$ K and  $\pm 2 \times 10^{-6}$  m, respectively. The grain diameter of the samples was determined before and after deformation using a metallurgical microscope. It was measured by the line intercept method [8]. The X-ray diffraction patterns of samples, rapidly quenched at room temperature (298K) after creep tests at all the working temperatures under a stress 15.4 MPa, were obtained. The lattice parameters were calculated using least-squares methods by a computer program.

**Experimental Results:**

In the present work, the creep curves of Sn-1wt % Pb alloy are obtained under different constant stresses ranging from 14.1 to 16 MPa near the transformation temperature, from 323 to 383K and are represented in Fig. 1. From this figure, it is observed that no transient regime could be resolved at 383K. Monotonic shift of straight lines due to temperature steps is observed in the relation between  $\ln \epsilon_{tr}$  and  $\ln t$ ,  $t$  is the transient time in [s], Fig. 2a,b,c. The transient creep parameters  $\beta$  and  $n$  are calculated from the relation:



**Fig. (1)** Creep curves at different constant stresses  $\sigma$  and different temperatures  $T$  for Sn-1wt% Pb alloy

$$\epsilon_{tr} = \beta t^n \tag{1}$$

where  $\epsilon_{tr}$  and  $\beta$  are in [%].

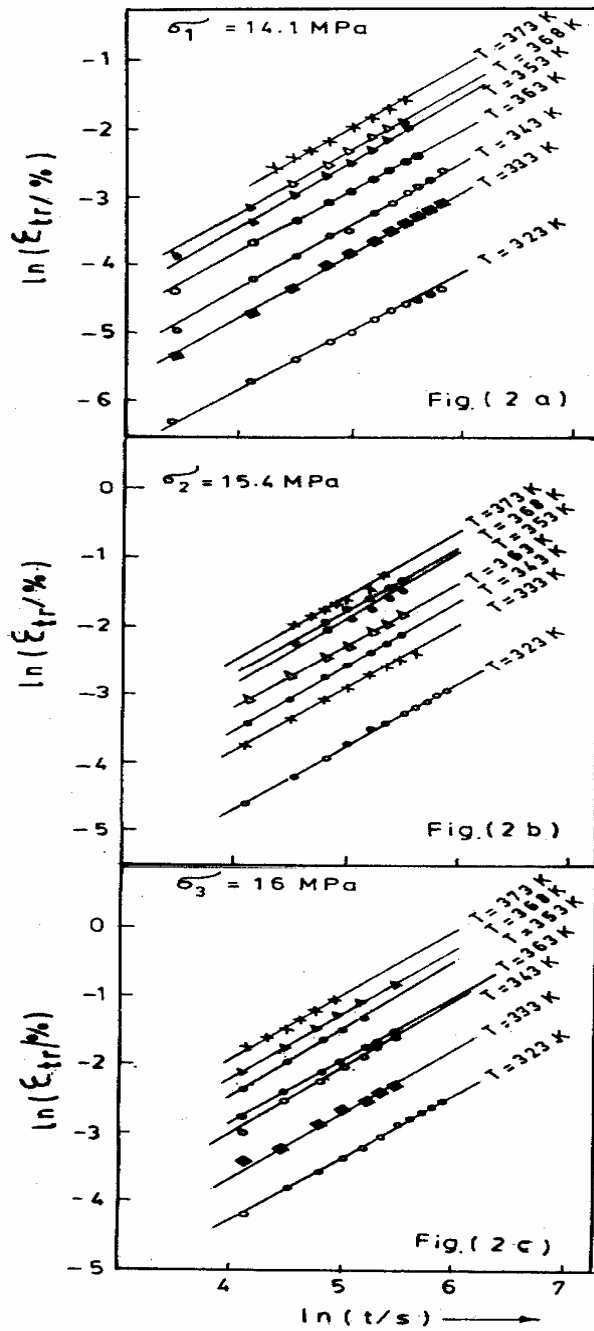


Fig. (2) Relation between  $\ln \epsilon_{tr}$  and  $\ln t$  for Sn-1wt% Pb alloy.

The parameter  $\beta$  is obtained from the intercept at  $\ln t=0$ , while  $n$  from the slope of the lines. Fig. 3 (a,b) represents the dependence of  $\beta$  and  $n$  of the alloy on the applied stress and the working temperature. The parameters  $\beta$  and  $n$  are found to exhibit values ranging from  $1.36 \times 10^{-4}$  to  $34 \times 10^{-4}$  and from 0.878 to 0.975, respectively. Peak values of both  $\beta$  and  $n$  are detected at the temperature 353K.

The dependence of the steady state strain rate  $\dot{\epsilon}_{st}$  on the applied stress,  $\sigma$  is found to obey the relation [9]

$$\sigma = K \dot{\epsilon}_{st}^m \tag{2}$$

where  $K$  is a constant and the exponent  $m$  is dependent on both the applied stress and the working temperature. The steady state strain rate sensitivity parameter  $m$  ( $\partial \ln \sigma / \partial \ln \dot{\epsilon}_{st}$ ) is derived from the slope of straight lines relating  $\ln \sigma$  and  $\ln \dot{\epsilon}_{st}$  (see Fig.4) is found to exhibit values ranging from 0.102 to 0.127 (with an accuracy of  $\pm 0.002$ ) depending on the creep temperature. Figs. 5a and b are plots of the strain rate sensitivity parameter ( $m$ ) and the steady state creep rate  $\dot{\epsilon}_{st}$  versus the creep temperature, respectively. It can be seen that the peak in both  $m$  and  $\dot{\epsilon}_{st}$  is found at about 353K.

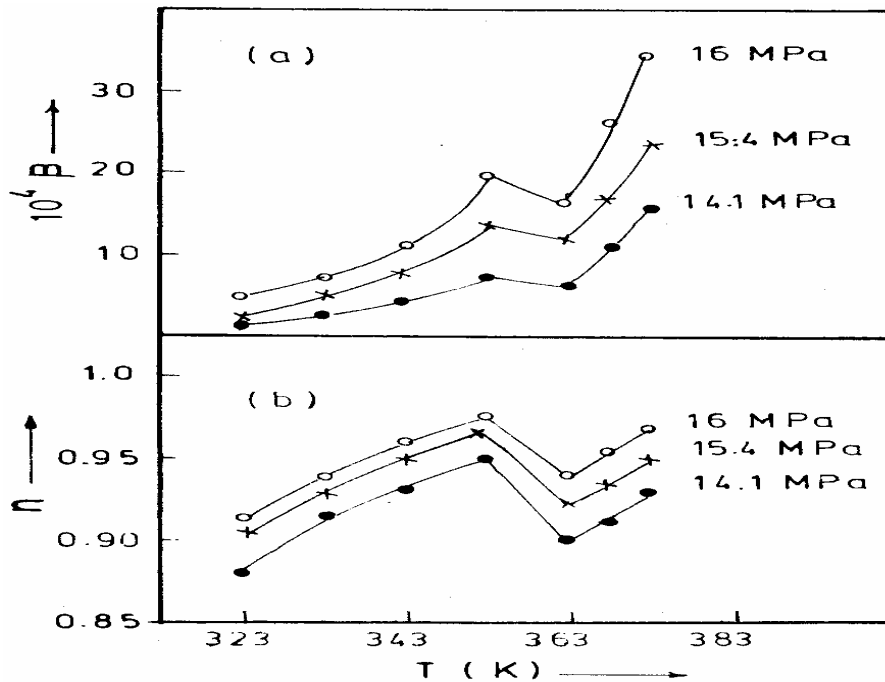


Fig. (3) (a) Relation between  $\beta$ , T at different stresses  
 (b) Relation between  $n$ , T at different stresses

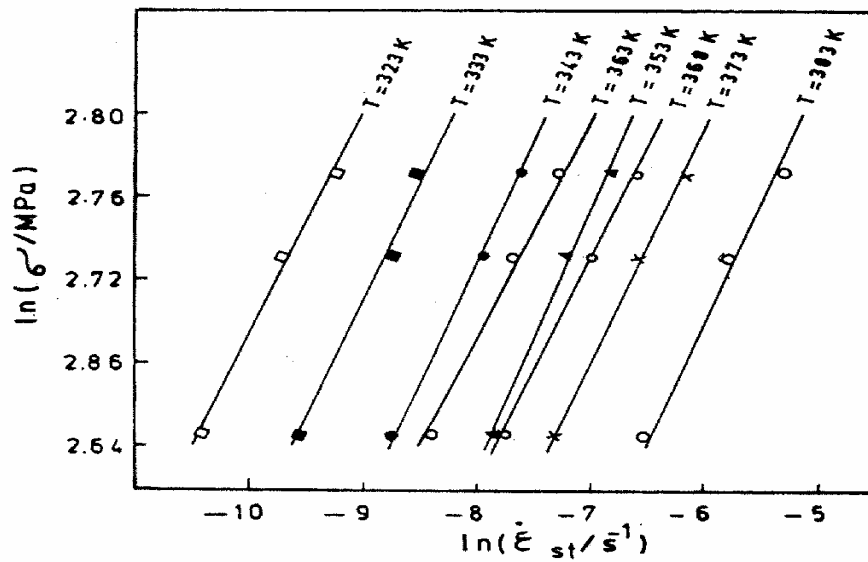


Fig. (4) A plot between  $\ln \sigma$  and  $\ln \dot{\epsilon}_{st}$  at different temperatures

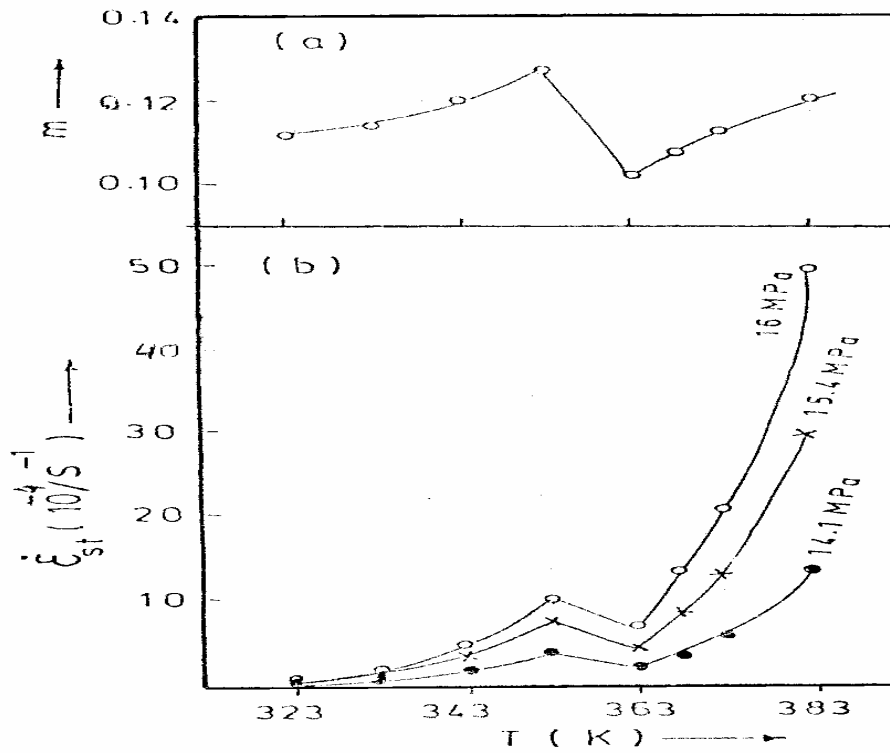


Fig. (5) (a) Relation between strain rate sensitivity parameter  $m$  and creep temperature  
 (b) Relation between  $\dot{\epsilon}_{st}$  and  $T$  at different stresses

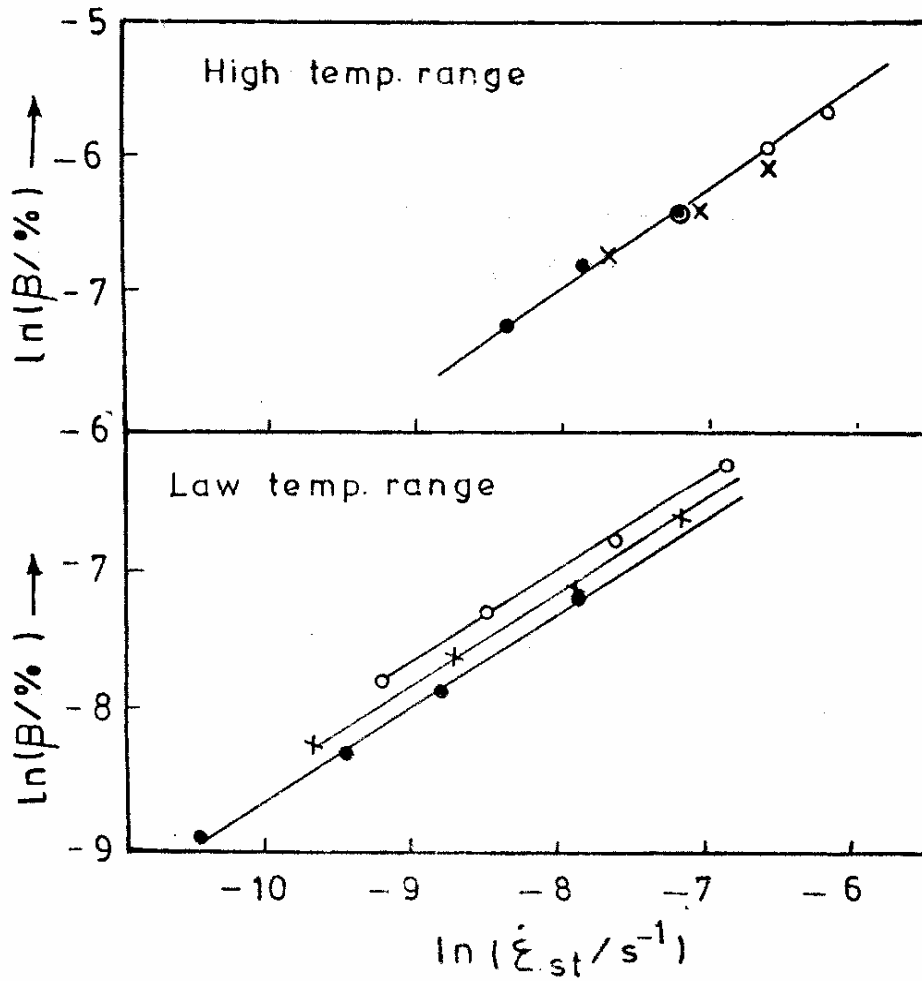


Fig. (6) Relation between  $\dot{\epsilon}_{st}$  and  $\ln \beta$  for different stresses  
 • 14.1, x 15.4, o 16.0 MPa

In order to correlate between transient and steady state creep, the relation [10,11]

$$\beta = \beta_0 (\dot{\epsilon}_{st})^\gamma \tag{3}$$

is found to be valid through the transformation regions as shown in Fig. 6.  $\gamma$  has values of 0.67 and 0.75 in the temperature ranges (323-353K) and (353-383K), respectively.

The activation energies of the transient and the steady state creep,  $Q_{tr}$  and  $Q_{st}$ , calculated from the slopes of the straight lines relating both  $(\ln \beta)$  and  $(\ln \dot{\epsilon}_{st})$  against  $(1000/T)$  are fitted by the least squares method and represented in Fig.7.

The activation energy of the steady state creep,  $Q_{st}$  in [J/mol] is calculated according to the equation [12]

$$\dot{\epsilon}_{st} = A \exp (-Q_{st}/RT) \quad (4)$$

where A is a constant, R is the gas constant in [ J / mol ] and T is the absolute temperature in [ K ].

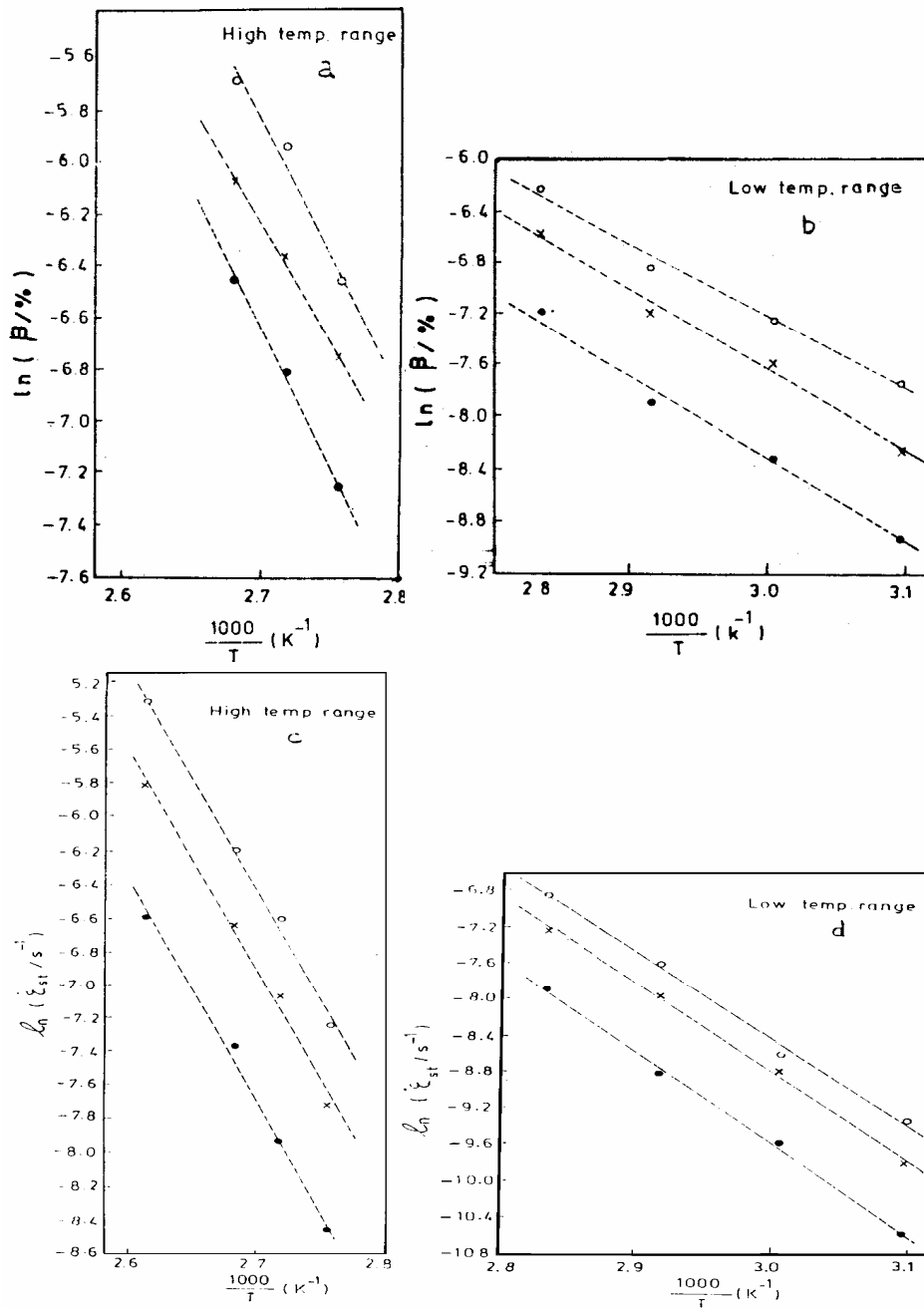
The values of both  $Q_{tr}$  and  $Q_{st}$  are found to be slightly affected by the applied stress. In the low temperature range  $Q_{tr}$  has values of  $(52 \pm 4)$ ,  $(52 \pm 4)$  and  $(47 \pm 3)$  kJ/mol for the applied stresses 14.1, 15.4 and 16 MPa, respectively. In the high temperature range  $Q_{tr}$  has the values of 90, 82 and 86 kJ/mol. (with an accuracy of  $\pm 7$  kJ/mol) according to the same sequence of the applied stresses. These  $Q_{tr}$  values give average values of  $(50 \pm 4)$  and  $(86 \pm 7)$  kJ/mol for the three applied stresses in the two temperature ranges, respectively.

$Q_{st}$  values are found to be 81, 79 and 78 kJ/mol (with an accuracy of  $\pm 5$  kJ/mol) and has values of 106, 107 and 108 kJ/mol (with an accuracy of  $\pm 9$  kJ/mol) for the previous applied stresses in the two temperature ranges, respectively.

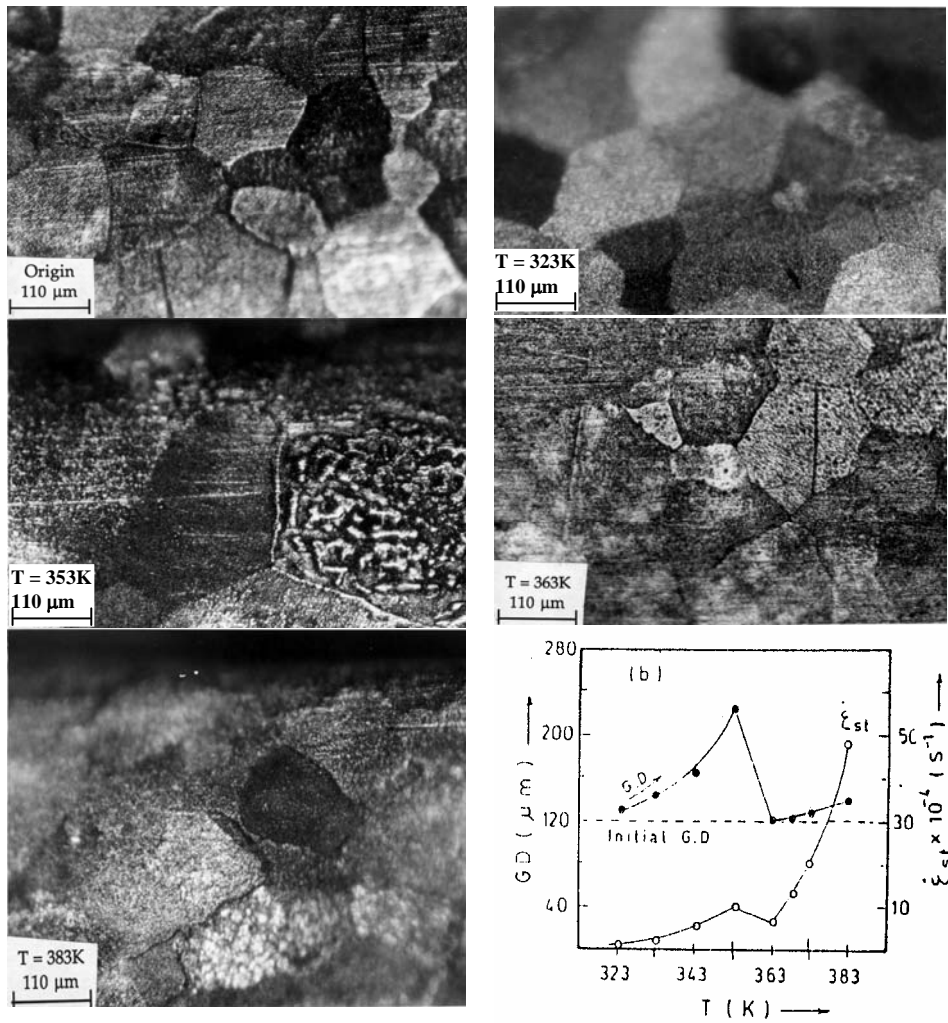
The metallographs of some crept samples under a stress of 16MPa and temperature 323, 353, 363 and 383K together with the uncrept sample are shown in Fig. 8. The average grain diameters GD versus temperature of the Sn-1wt % Pb alloy are illustrated in Fig. 8b. It is found that, they are larger than those of the original sample.

The X-ray diffraction patterns of all crept samples under a stress 15.4 MPa after rapidly quenched at room temperature (298 K) are obtained. X-ray integral intensity (I) of the diffraction lines (301) and (200), and the ratio (c/a) for the Sn-rich phase are calculated as a function of the creep temperature. They show minima at 353K as indicated in Fig. 9.





**Fig. (7)** Relation between (a,b)  $\ln \beta$  and (c,d)  $\ln \dot{\epsilon}_{st}$  against  $1000/T$  at different applied stresses :  
 • 14.1, x 15.4, o 16.0 MPa,(---) calculated values from the best fitting equation



**Fig. (8)** (a) Micrographs of crept samples under 16 MPa  
 (b) The dependence of grain diameter GD on the mechanical processes operated at the given temperatures for samples crept under 16 MPa

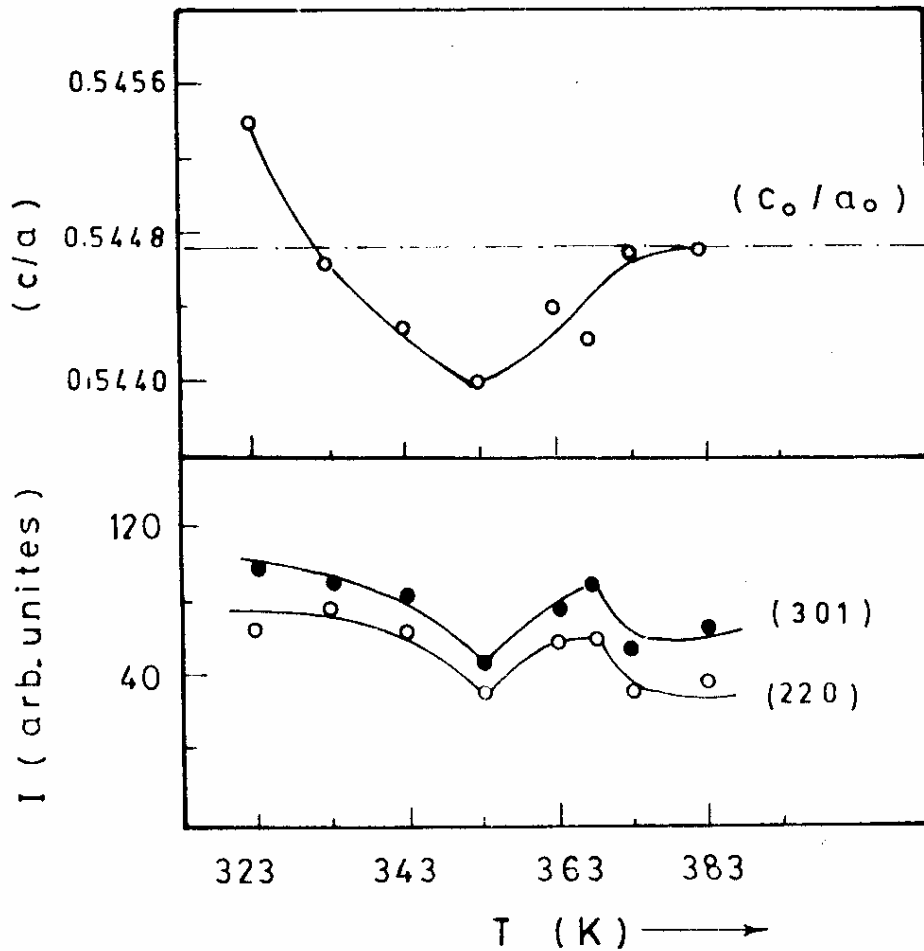


Fig. (9): The dependence of the integral X-ray intensity  $I$  and the ratio  $c/a$ , for the Sn-rich phase on the creep temperature of Sn-1wt% Pb alloy samples crept under 15.4 MPa.

### Discussion:

The phase diagram of Sn-Pb system [7] shows that, for Sn-1wt% Pb alloy both the Pb-rich phase ( $\alpha$ -phase) and Sn-rich phase ( $\beta$ -phase) exist in an equilibrium state in the low temperature region (323-353K). By increasing the working temperature, the solubility of Pb in Sn increases, i.e. Pb will go from the precipitates into the solution and disappears completely at the transition temperature (353K) [7].

The creep curves of Sn-1wt %Pb alloy show a monotonic shift towards higher strains and lower fracture time with increasing the temperature. This shift is interrupted and enhanced at the transformation temperature (353K) due to the release of internal stresses caused by the dissolution of the f.c.c.  $\alpha$ -solid solution phase in the tetragonal  $\beta$ -matrix (Sn-rich phase).

Some of the creep curves show the three stages of the ideal creep curve [13]. Each stage has its own controlling mechanisms. During the first creep stage (transient creep), the rate controlling mechanism is determined by the transient creep parameters  $\beta$  and  $n$  given by equation (1). An increase in the values of both  $\beta$  and  $n$  (Fig.3) is noticed with increasing the applied stress and the working temperature in the temperature range before their peak values. This increase is thought to be due to the redistribution and rearrangement of dislocation in the network at transformation. It seems trivial that  $\beta$  increases with stress and with temperature as transient strain is produced by thermally activated dislocation glide. The increase of  $n$  with stress is not such trivial and may reflect a dislocation multiplication. As shown in Fig.3, the behavior of all the curves is the same for the different applied stresses, so the controlling mechanism is stress independent.

The peaks of both  $\beta$  and  $n$  at 353 K represent a higher strain associating phase transformation due to the release of stored deformation energy by the dissolution of  $\alpha$ -phase. The formation of the softer  $\beta$ -phase by decreasing the internal stresses set up at this temperature enhanced the transient strain ( $\epsilon_{tr}$ ). The observed decrease in  $\beta$  and  $n$  values after the peak may be attributed to the coarsening of Sn-rich phase grains which inhibit or stop the effect of grain boundary migration and to rearrange the atoms in the direction of homogenization of the single Sn-rich phase.

The activation energy  $Q_{tr}$  has values of  $(50\pm 4)$  and  $(86\pm 7)$  kJ/mol which indicates that the dominant mechanisms may be dislocation [14] glide and slipping in the low and high temperature ranges, respectively.

In the second creep stage (steady state), the low strain rate ( $\dot{\epsilon}_{st} = 10^{-4}$ ) is presumed to be in the range of the grain boundary sliding mechanism [2] which was concluded to be the dominant deformation process. The strain rate sensitivity parameter  $m$  has the smallest values between 0.102 and 0.127 (Fig.5a) suggested by creep theories based on a dislocation glide along the grain boundaries.

The values of  $Q_{st}$  around the transformation temperature are found to be (79) and 106 kJ/mol. They identify the mechanisms of Coble creep (74.5 kJ/mol

[15]) and viscous grain boundary sliding [16], respectively. The grain boundary sliding caused an increase in the grain diameter of the crept samples (Fig.8a and b). The pronounced decrease of the grain size after transformation may be due to dynamic recrystallization.

From the X-ray diffraction analysis, the ratio ( $c/a$ ) and the integral intensity ( $I$ ) are found to change with the creep temperature. They have relatively high values at 323K which may be due to the existence of the very fine Pb precipitated particles which causes a small effect on ( $I$ ). The decrease in their values to minima at the transformation temperature (353K) may be due to the dissolution of Pb precipitate where the atomic radius of Pb ( $1.75\text{\AA}$ ) is greater than that of Sn ( $1.58\text{\AA}$ ). So that the dissolution of Pb-phase in the Sn-phase may cause an increase in the lattice parameter  $a$  and therefore decreases the  $c/a$  ratio of the tetragonal Sn-rich phase. Above (353K) the rate of the recovery process due to heating is thought to be larger than the strain hardening because of the observed small strain rate, and this is accompanied by the increase of ( $c/a$ ) with temperature. Their nearly stable values at the high temperature range (373-383K) may be due to both the homogenization in the solid solution phase.

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