Introduction

The transient creep distortion is specified by the motion of mobile dislocations and finish in their effective locking at obstacles or is originally believed as dislocation consuming source\(^1\). Tin-indium alloys are of individual importance, because the existence of indium appears to confer the special properties of wetting and bounding\(^2\) to glass or glazed surfaces, and gives an increase of the low hardness and the resultant low mechanical strength of Sn\(^3\). These alloys called Pewters (alloys of more than 90% Sn) are utilized especially as solders for packing and interconnection in the electrical, electronic, owing to their ease of fabrication into any needed form. Also, these alloys are used for utensils; they have been investigated\(^4,5\). Few publications (not many) dates in literature in the mechanical properties of Sn-In alloys, and Sn-Ag-In alloys have been done. The aim of the present work is destined to give some information about mechanical and structure properties of the present alloy Sn-Ag-X In.

Experimental

The transient creep experiments were inspected under several used stresses extended from 10 to 19.5 MPa for three ternary eutectic (Sn-3.5Ag-xIn) alloys, where x takes the values (x=0.5, 1 and 1.5 In). All tests at temperatures, ranging from 303 to 393 K. The transient creep is classified by \(\varepsilon_{tr} = \beta t^n\), Where \(\varepsilon_{tr}\) and \(t\) are the transient creep strain and time. The exponent \(n\) was found to have values extended from 0.646 to 1.59 for Sn-3.5Ag-0.5In, ranging from 0.82 to 1.64 for the second alloy Sn-3.5Ag-1In, and finally it ranging from 0.94 to 1.74 for Sn-3.5Ag-1.5In. The value of \(\beta\) was found to have values ranging between -14.38 to -7.2, -13.85 to -7, and -13.55 to -6.8 for the three alloys; the activation enthalpy shows that the operating mechanism controlling the tin process may be the grain bounding diffusion. Also, X-ray diffraction examination display the permanence of both \(\beta\)-Sn rich phase and the intermetallic compound Ag3Sn and very little particles or residue from the intermetallic composition \(\gamma\)-In Sn4.

Keywords: Binary alloy; intermetallic composition; transient creep; ternary alloys.
solution of 2% HCl, 3% HNO₃ and 95% (vol.%) ethyl alcohol was prepared and used to etch the samples. Phase identification of the used samples accomplished out by X-ray diffractometry (XRD) at 40 KV and 20 mA using Cu Kα radiation with diffraction angles (2θ) from 20.99° to 99.99° and a fixed scanning speed of 1°/min.

**Table1. Actual compositions of the experimental alloys, wt. %.**

| Experimental alloys | Sn  | In  | Ag  |
|---------------------|-----|-----|-----|
| Sn-3.5Ag-0.5In      | 96  | 0.5 | 3.5 |
| Sn-3.5Ag-1.0In      | 95.5| 1.0 | 3.5 |
| Sn-3.5Ag-1.5In      | 95  | 1.5 | 3.5 |

**Figures1. XRD pattern for tested alloys; they are mainly composed of two phase structure, that is, tetragonal β-Sn rich phase, and the intermetallic compounds as Ag₃Sn phase.**

**Results and Discussion**

The test of the X-ray diffraction model has been given in Fig.1; it displayed that Sn-3.5Ag-0.5In, Sn-Ag-1.0In, and Sn-3.5Ag-1.5In alloys shows only two phase structure, that is, β-Sn rich phase. In addendum to the intermetallic compounds as Ag₃Sn is the other phase.

The transient strain is given by the equation (1)\(^\text{(7)}\):

\[ \varepsilon_t = \beta t^n \]  \hspace{1cm} (1)

where \(\varepsilon_t\) and \(t\) are the transient creep strain, and time, \(\beta\) and \(n\) are constants relying on the empirical experience states.

**Figure2. illustrated the OM microstructure a) binary Sn-3.5Ag, b) Sn-3.5Ag-0.5In c) Sn-3.5Ag-1.0In**
Figure 3a. represented SEM images of the Sn-3.5Ag alloys, the microstructure composed of β-Sn areas, fine Ag3Sn precipitates, and eutectic area. In Fig.3b, EDS analysis of the Sn-3.5Ag alloys.

It can be observed in Fig.2a that the microstructure of the Sn-3.5Ag binary alloy consists of relatively fine Ag3Sn precipitates in the white β-Sn matrix. In Fig.2b, the microstructure of the Sn-3.5Ag-0.5In alloy showed a coarse γ-InSn4 in the β-Sn matrix. Fig.2c represented the microstructure of Sn-3.5Ag-1.0In alloy, where the volume fraction of γ-InSn4 is increased, in addition to, fine Ag3Sn, and β-Sn matrix. Fig.3a; represented SEM images of the Sn-3.5Ag alloys; the microstructure possessed β-Sn areas, fine Ag3Sn precipitates, and eutectic area. In Fig.3-b EDS analysis of the Sn-3.5Ag alloys.

Creep curves of Sn-3.5Ag-In alloys

Fig.4a-e displays typical creep curves of the three tested solder alloys expressed as creep rate versus time at constant test temperature in the range from 303 to 393K in 20K growing under applied stress level in the range of 10.5 to 19.5 MPa for all tested materials. Typical examples of creep curves of alloys are presented in this Fig.

Figure 4(a). Creep curves at 303 K and different applied stresses for Sn-3.5Ag-0.5In, Sn-3.5Ag-1In, and Sn-3.5Ag-1.5In ternary alloy.
Figure 4b. Creep curves at 323 K and different applied stresses for Sn-3.5Ag-0.5In, Sn-3.5Ag-1In, and Sn-3.5Ag-1.5In ternary alloy.

Figure 4c. Creep curves at 348 K and different applied stresses for Sn-3.5Ag-0.5In, Sn-3.5Ag-1In, and Sn-3.5Ag-1.5In ternary alloy.

Figure 4d. Creep curves at 373 K and different applied stresses for Sn-3.5Ag-0.5In, Sn-3.5Ag-1In, and Sn-3.5Ag-1.5In ternary alloy.

To compare the effect of small addition of In on Sn-3.5Zn lead-free solder alloys in the present study, Fig. 5 represented the creep curves of the three tested alloys at constant stresses = 14.37 MPa, it is obvious that the ternary Sn-3.5Zn-1.5In alloys is more superplastic than the Sn-3.5Zn-1In, and Sn-3.5Zn-0.5In. The relation between lnε and ln t gives straight lines as shown in Fig. 6a-e. This research denote that there is transition temperature under 50 °C.
Figure 4e. Creep curves at 393 K and different applied stresses for Sn-3.5Ag-0.5In, Sn-3.5Ag-1In, and Sn-3.5Ag-1.5In ternary alloy.

Figure 5. Comparison of isothermal creep curves at constant stress = 14.37 MPa and different temperature for the three tested ternary alloys.

Figure 6a. Relation between lnεtr and lnτ for Sn-3.5Ag-0.5In, Sn-3.5Ag-1In, and Sn-3.5Ag-1.5In ternary alloy, at 303K and different stresses.
Transient creep characteristics of Tin Base Alloy

**Figure 6b.** Relation between $\ln \epsilon_{tr}$ and $\ln t$ for Sn-3.5Ag-0.5In, Sn-3.5Ag-1In, and Sn-3.5Ag-1.5In ternary alloy, at 323K and different stresses.

**Figure 6c.** Relation between $\ln \epsilon_{tr}$ and $\ln t$ for Sn-3.5Ag-0.5In, Sn-3.5Ag-1In, and Sn-3.5Ag-1.5In ternary alloy, at 348K and different stresses.

**Figure 6d.** Relation between $\ln \epsilon_{tr}$ and $\ln t$ for Sn-3.5Ag-0.5In, Sn-3.5Ag-1In, and Sn-3.5Ag-1.5In ternary alloy, at 373K and different stresses.
Transient creep characteristics of Tin Base Alloy

Figure 6e. Relation between lnε<sub>tr</sub> and lnt for Sn-3.5Ag-0.5In, Sn-3.5Ag-1In, and Sn-3.5Ag-1.5In ternary alloy, at 393K and different stresses.

Figure 7. The temperature dependence of the parameters, n, at different applied stresses for Sn-3.5Ag-0.5In, Sn-3.5Ag-1In, and Sn-3.5Ag-1.5In ternary alloy.

On the other hand the exponent n was estimated from the slope of these lines and was found to have values extended from 0.646 to 1.59 for Sn-3.5Ag-0.5In, ranging from 0.82 to 1.64 for the second alloy Sn-3.5Ag-1In, and finally it ranging from 0.94 to 1.74 for Sn-3.5Ag-1.5In as represented in Fig.7, and Table 2.

Table 2. Comparison of the transient creep characteristics of the tested alloys.

| Experimental alloys   | Q (kJmol⁻¹) | γ   | β             | n             |
|-----------------------|-------------|-----|---------------|---------------|
| Sn-3.5Ag-0.5In        | 25 : 28.5   | 0.186 : 0.29 | -14.38 : -7.2 | 0.646 : 1.59  |
| Sn-3.5Ag-1In          | 20.16 : 25.3 | 0.10 : 0.21 | -13.85 : -7   | 0.82 : 1.64   |
| Sn-3.5Ag-1.5In        | 10.5 : 15.48 | 0.073 : 0.12 | -13.55 : -6.8 | 0.94 : 1.74   |

The intercepts of lnε<sub>tr</sub> and lnt at lnt = 0 gave the transient creep parameter β; it was calculated from Eq.(2) \(^{(7,8)}\).

\[
\ln \beta = (\ln t \varepsilon_{tr1} - \ln t \varepsilon_{tr2})/\ln t - \ln t
\]

The value of β was found to have values ranging between -14.38 to -7.2, -13.85 to -7, and -13.55 to -6.8 for the alloys as shown from Fig.8.
Finally the activation enthalpy of the transient creep $Q_{tr}$ was evaluated utilizing (4).

$$
\varepsilon_{tr} = \varepsilon_0 + t^n \exp\left(-\frac{Q_{tr}}{KT}\right)
$$

\(3\)

**Figures 8.** The temperature dependence of the parameters, $\beta$, at different applied stresses for Sn-3.5Ag-0.5In, Sn-3.5Ag-1In, and Sn-3.5Ag-1.5In ternary alloy.

The activation enthalpy of the transient creep $Q_{tr}$ have been found to be 25: 28.5 KJ/mol for the 1st alloy where (x=0.5In), 20.16: 25.5 KJ/mol for (x=1In) and 10.5 : 15.48 KJ/mol for the third alloy (x=1.5In) in the low and high temperatures regions respectively as represented in Figs.9,10, and Table 2.

**Figure 9.** Relation between ln$\varepsilon_{tr}$ and 1000/T at different applied stresses for Sn-3.5Ag-0.5In, Sn-3.5Ag-1In, and Sn-3.5Ag-1.5In ternary alloy, at low Temp.

**Figure 10.** Relation between ln$\varepsilon_{tr}$ and 1000/T at different applied stresses for Sn-3.5Ag-0.5In, Sn-3.5Ag-1In, and Sn-3.5Ag-1.5In ternary alloy, at high Temp.
Transient creep characteristics of Tin Base Alloy

Creep is a persistent operation, since transient and steady-state creep regions are joined to each other by the equation (3) \(^{(9,10)}\).

\[
\beta = \beta_0 \left( \varepsilon_{\text{st}} \right)^\gamma
\]  

(4)

where \(\beta_0\) is a fixed and \(\gamma\) is the steady-state creep exponent gauge the contribution of the transient technicality to the steady-state creep manner. The values of \(\beta\) and \(\varepsilon_{\text{st}}\) qualify us to plot a relation between \(\ln\beta\) and \(\ln\varepsilon_{\text{st}}\) as represented in Fig.11. A longitudinal reliance is obtained with intermediate value of \(\gamma\) for the three alloys. This relatively value of \(\gamma\) confirms that the mechanism dependable on the transient stage also operates in the steady-state stage. This average value of \(\gamma\) is identical with those acquired by Kenawy et al.\(^{(11)}\); and Mahmoud and Graiss\(^{(12)}\) in their study. The exponent \(\gamma\) was found to change from 0.186 to 0.29, 0.10 to 0.21, and 0.073 to 0.12 for the three alloys respectively; as shown in Table2.

Figure 11. Relation between \(\ln\beta\) and \(\ln\varepsilon_{\text{st}}\) at different applied stresses for Sn-3.5Ag-0.5In, Sn-3.5Ag-1.0In, and Sn-3.5Ag-1.5In ternary alloy, at high Temp.

The elevate of parameter \(\beta\) with elevation experiment temperature as presented in Fig.6 is established on the fact that \(\beta\) represents the precipitation reliance of \(\varepsilon_{\text{tr}}\) on the precipitation temperature \(^{(13,14)}\). So it could be pronounced that a rise of the working temperature helps dislocations to overcome the precipitates that act as barrier \(^{(15)}\). The driving force for both reconfiguration of these dislocation provenances and consuming of the opposite ones is quickened by the utilized stress which in addition to the effective energy oscillatory stresses facilitates the movement of dislocations parallel to the utilized stress orientation\(^{(16,17)}\). Also; the distinguish elevate in parameter \(\beta\) could be due to the increase in the motion of groups of piled-up dislocations\(^{(18)}\) while the number of A Guinier-Preston zone (GP zones) is still fixed. Dislocations of increased mobility existing among a stable number of zones result in less of a pinning effect and, accordingly, dislocations become free and their movement must be easier. This guide to an excess in \(\beta\). The observed development of \(\beta\) can be because of the outward flexible energy provided thermally (through testing temperatures). This energy simplified the motion of existing dislocations between the courses of creep and shortens the duration of the creep process\(^{(19,20)}\).

Fig.12 showed the variation of \(d\)-spacing [Å], with 2\(\theta\), the value of the angle varies from 22 to 99 for Sn-3.5Ag ternary Sn-3.5Ag-1In, and Sn-3.5Ag-1.5In alloys. Fig.13: Showed the variation of Rel. Int.[%], with 2\(\theta\) for same tested alloys. Fig.14; showed the variation of FWHM with 2\(\theta\) for the same tested alloys.

It is obvious that activation energies for binary alloys are more than that of ternary Sn-3.5Ag-1In, and Sn-3.5Ag -1.5In alloys because of ternary alloys are more refine in grain size and superplastic than binary alloys. The activation enthalpies, point that the transient creep in the low temperature area is controlled by dislocation intersection, while at altitude temperatures, the dominant technique is grain boundary sliding \(^{(21)}\).
Figure 12. Showed the variation d-spacing [Å], with 2θ for Sn-3.5Ag-0.5In, Sn-3.5Ag-1In, and Sn-3.5Ag-1.5In ternary alloy.

Figure 13. Showed the variation of Rel. Int.[%], 2θ for Sn-3.5Ag-0.5In, Sn-3.5Ag-1In, and Sn-3.5Ag-1.5In ternary alloy.

Figure 14. Showed the variation of FWHM with 2θ for Sn-3.5Ag-0.5In, Sn-3.5Ag-1In, and Sn-3.5Ag-1.5In ternary alloy.
Transient creep characteristics of Tin Base Alloy

It can be seen that, although each of single curves do not permanently showed a sharp primary creep stage they all eventually show a relatively short secondary region in which the load propagates longitudinal with time. Necking and fracture of all samples does not occur; there should not be a third stage as in the ordinary tensile creep test. Also, the tested alloy of tin base alloy under low working temperature obeys the logarithmic creep which obeys the equation \( \varepsilon = \sigma \log t \). The samples are so stable in texture (ternary) with precise grain size. The intermetallic compounds \( \text{Ag}_3\text{Sn} \) as will be obtain by X-ray diffraction will precipitate within grain size and makes pining through the grains. The influence of indium content on the creep resistance of the tested Sn-3.5Ag-0.5In alloys can be inferred from their respective microstructure.

Conclusion

This paper has inspected the effects of small amount of In on microstructure and creep properties of Sn-3.5Ag based lead-free solder alloys; the Conclusions are.

1-For transient creep stage, for all three samples, the activation energies estimated indicate that value of 0.5In is more than that of 1In and 1.5In, therefore the first samples is lower elongation than the two other.

2- The values of \( \gamma \) submit a high reliance of steady-state creep on the transient creep stage.

3-The transient creep parameter \( n \) and \( \beta \) rises with rising the distortion temperature and loads.

4- Addition of In to the Sn matrix led to; the structure fine \( \text{Ag}_3\text{Sn} \) precipitates in the white \( \beta\)-Sn matrix, in addition to eutectic areas.

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Transient creep characteristics of Tin Base Alloy

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