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Transient and steady-state creep characteristics of Transformations in Al-Zn Binary Alloys

M. Y. Salem

Physics Department, Faculty of Science at New Valley, Assuit University, 72511, El-Kharga, Egypt

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Abstract: Transient and steady - state creep tests were studied under different applied stresses ranging from 12.236 to 14.275 MPa from 523 K to 633 K for Binary (Zn -28.5 Al based) Alloys. The transient creep is described by $\varepsilon_{tr} = \beta t^n$, where ε_{tr} and t are the transient creep strain and time. The parameters β and n were calculated and found to change from - 3.73 to -1.791 and 0.227 to 0.718 respectively. The alloy showed increase in the creep parameters β and n. The exponent γ have values 0.53 - 0.9, the activation energy of transient creep are 63.8 KJ/ mole and 66.8 KJ/ mole in Low and high temperature range respectively. Also, the steady state creep was investigated in the same temperature range under same stresses. The results of creep characteristics show two deformation regions (below and above 543 K). The strain rate sensitivity parameter (m) has been found to increase by increasing the working temperature up to about 0.53 for the alloy. The activation energies of steady state creep of the alloy have been found to be 76.7 and 79.72 KJ/mol, in the low and high temperature regions respectively (below and above 543 K), characterizing grain boundary diffusion. Metallographic observations and X-ray analysis have been confirmed that grain boundary sliding (GBS) assimilation by dislocation motion is the aim mode of deformation.

Keywords: Activation energies, Grain boundary, Strain, Transient creep.

1 Introduction

Creep may be known as the continued distortion of a material with time when undergo to a stationary stress or load [1]. Its properties are known to depend on deformation temperature, internal microstructure of the tested material [2,3]. The creep of solid solution alloys perhaps because of as for dislocation mounting or viscous slipping in which dislocations pull dissolved atom atmospheres are controlling [4,5].

The present paper, aims to describe the creep behaviour of Zn-28.5Al. Using Scanning electron microscope (SEM), and Energy Dispersive X-Ray Spectroscopy (EDX), and X-ray diffraction (XRD) the influence Zn-addendum to Al on the creep disposal have been investigated.

Al and its alloys are important metallic materials for many applications in different domain, because of its minimum cost, elevation particular strength, high thermal conductivity, and good resistance to corrosion, Al alloy has been extremely used for automotive heat exchangers to replace extra classical accessories such as stainless steels and Cu alloys [6-8]. Zn-Al alloys are technologically remarkable and necessary. They have excellent mechanical properties, high strength, simple finishing, power to bond totally with the shell, and, convenient[9-11]. Many Al-Zn alloys are also utilized in structural purpose and its alloys is a great issue because of their numerous employment in industries [12], where resistance creep becomes a significant characteristic. One of the evident ways to upgrade ductility is by adding alloying elements, and many studies have been completed, resulting in persistent perfection alloys [13,14]. For this aim, it is requisite to recognize how the creep parameters change with alloying additions, reiterated thermal cycles and applied stresses, thus, the future employment of these samples highly depends on the success of beneficent their accuracy with respect to the structural employment. The elegant studies to explain the features of creep in Zn-Al samples have been done by Y.N. Wang and, J.C. Huang [15], Mahmoud S. Soliman, Farghalli A. Mohamed[16], and Hidetoshi Somekawa et al.[17]

The present paper target to study the properties of the tested alloys. There is a variation in m and activation

^{*}Correspondingauthore-mail:mahmoud salem569@yahoo.com



energy at about 0.8 T_m . However, the enormous discrepancy in activation energy for creep through these studies submit that the rate dominant creep process differ at the transition temperature, Temperature domain for creep can be divided to 3 part: (1) high temperature creep (T > 0.6T_m), (2) moderate temperature creep ($0.3T_m < T < 0.6T_m$), and (3) minimum temperature creep ($T < 0.3T_m$). Usually, creep studies examine altitude temperature deformation [18].

2 Experimental

Al, Zn of purity 99.99% were used to prepare the binary Zn-28.5 Al based alloy by vacuum melting. The alloys were ready by melting individually in elevation purity pure graphite crucible. The sample was rolled to wires. In this study, the samples were annealed at 813 K for 3 hours and then quietly calm down to room temperature. After this heat treatment, the samples were annealed at room temperature for 168hour prior testing. This process allowed a small contain of grain growth and grain stabilization to take place [19]. Creep deformations were performed on annealed wire samples of 0.8 mm diameter and 50 mm gauge length. The slowly cooled samples were crept under constant applied stresses ranging from 12.236 to 14.275 MPa at temperatures ranging from 523 to 633 K using a traditional type creep machine [20]. The accuracy of temperature measurement is of the range \pm 1K. Strain measurements were done with an accuracy of $\pm 1 \times 10^{-5}$.

2.1. Transient stage

With regard to the transient interval of the creep curve, the transient creep strain is commonly touted by the familiar Friadel's equation [21-25]:

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

Where ε_{tr} and t are the transient creep strain, and time, β and n are (transient creep parameters) constants relying on the experimental test conditions. In the present paper the relation between $\ln \varepsilon_{tr}$ and $\ln t$ gives straight lines.

The exponent n was determined from the slope relate $ln\epsilon_{tr}$ and lnt, while the coefficient β was calculated from the intercepts at lnt = 0:

$$\ln\beta = (\ln t_2 \varepsilon_{tr1} - \ln t_1 \varepsilon_{tr2}) / \ln t_2 - \ln t_1$$
(2)

It was found that n increases with increasing deformation temperature. The coefficient β was rising with rising temperature and rising the applied stress.

The relation between coefficient β and strain rate of the steady state creep (ϵ 'st) written in the equation [14].

$$\beta = \beta_0 \, (\epsilon_{st})^{\gamma} \tag{3}$$

Where, β_0 and γ are constants depend on the experimental conditions.

2.2. Steady-state creep stage

Steady state creep menstruations were carried out especially on (Zn-28.5Al based) Alloys. The samples were inspected in the temperature domain 523 to 633 K in steps of 10 K under stationary applied stresses.

The steady-state strain rate ε_{st} of the examined samples is calculated from the slopes of the linear parts of the acquired creep curves. It increases with increasing both the T and σ (stress).

Strain rate sensitivity parameter $m = \partial \ln \sigma / \partial \ln \varepsilon$ st was calculated from the inclination of the straight lines of $ln\sigma$ and $ln\dot{\varepsilon}_{st}$. The activation energy of steady creep at stable load was studied utilize the following formula [3].

$$Q = R(\partial \ln \varepsilon_{st} / \partial (1/T))_{(4)}$$

where T and R are temperature (Kelvin), and the gas constant, respectively.

Moreover, the acquired results verify the equation of steady state creep [26,32]

$$\varepsilon \cdot_{st} = c \left(\frac{\sigma}{d}\right)^{1/m} \exp\left(\frac{Q}{kT}\right)$$
(5)

where m = 0.5 for dislocation ascent over grain boundaries [33,34] therefore, it is considered here that the higher elongation is attributed to the dislocation motion results in *GBS* and contained it during the deformation.

3 Results

Isothermal creep curves for the tested alloy were studied using three applied stresses at twelve working temperatures ranging from 523 to 633 K in steps of 10 K (see Fig.1). The gradation of creep curves with regard to the temperature display an acute rising in behavior particularly at high temperatures. Fig.(2) shows the creep behaviour as a plot of $\ln \varepsilon_{tr}$ against $\ln t$. The exponent *n* was found to have values ranging from 0.227 to 0.718. The parameter β was mention to have ranging from -3.73 to -1.791. Fig. 3 exemplify the temperature dependence of *n* and β on the experimental conditions sight up.

In order to correlate between transient and steady state creep the linear relation between $\ln\beta$ and $\ln\varepsilon_{st}$ for different constant stresses through phase transformation, was plotted (see Fig.4). Eq.(3)

connecting β and ε_{st} was found to be valid with the exponent γ ranging from 0.53 - 0.9. The activation enthalpy of the transient creep Q_{tr} was calculated using the following equation [35,36]:

$$\varepsilon_{tr} = \varepsilon_0 + t^n \exp\{-Q_{tr}/KT\}$$
(6)

Where ε_0 is the instantaneous strain.

Fig.5 illustrates the relation between $\ln \varepsilon_{tr}$ and 1000/T. These results yield activation enthalpies of 63.8 and 68.8 KJ/mole in low and high temperature regions, respectively as shown in table (1).

Materials	n	β	γ	Q (kJmot ⁻¹)
Zn-28.5A1	0.227 : 0.718	-3.73 : -1.791	0.53 : 0.9	63.8 : 66.8

Table (1): Comparison of the transient creep characteristics of the tested alloys.

3.1. Steady-state creep stage

Also, steady state creep magnitudes were achieved especially on (Zn -28.5Al based) alloys. The alloys were inspected in the temperature domain 523 K to 633 K in level of 10 K with fixed used load. As shown in Fig.1. The creep distortion average is considerable effective at elevation temperatures (above 543 K).

Strain rate sensitivity parameter $m = \partial \ln \sigma / \partial \ln \varepsilon$ st [37–39], was estimated from

the gradient of the upright lines of $\ln \sigma$ and $\ln \hat{\mathcal{E}}_{st}$. The value of m estimate from the information in Fig.6 as the steady strain rate $\dot{\mathcal{E}}_{st}$ are drawn with temperature in Fig.7. The steady-state strain rate ε'_{st} of the examined alloys is counted from the gradient of the regular parts of the acquired creep curves Fig.(1). It increases with increasing the temperature and stress as presented in Fig.(7). ϵ'_{st} was found to have values ranging from $5.9*10^{-6}$ to $1.5*10^{-5}$. It is obvious that m exhibits low value (0.28 \pm 0.04) up to about 543 K. After that m increases with temperature until 633 K reaching the value of 0.53, illustrating that the sensitivity parameter m be temperature attached. The activation energy of steady creep at fixed loads were estimate using relation (4). The activation energies have been found to be 76.7and 79.72 kJ/mol in the low and high temperature regions (below and above 543 K), respectively (see

Fig.8), as	written	in	Tab	le.	(2)).
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Materials	(m)	٤´st	Q (kJmol ⁻¹)
Zn-28.5Al	0.28 : 0.53	5.9*10 ⁻⁶ : 1.5*10 ⁻⁵	76.7 : 79.72

Table (2): Comparison of the steady state creep characteristics of the tested alloys.

SEM micrograph of the Fig.(9): Showed deformed samples crept at 523 K at stress 12.236, 13.255, and 14.275 MPa, it is possible observe Zn precipitates in the grain boundaries in the form of dendrites and Al-rich light regions. Fig.(10): Showed SEM micrograph of the deformed samples crept at 543 K at 13.255, and 14.275 MPa, in this micrograph there are some thinner Zn precipitates in the grain boundaries even without dissolving in the matrix. Fig.(11): Showed SEM micrograph of the deformed samples crept at 623 K at 13.255, and 14.275 MPa, this micrograph lets to observe completely scattered precipitates in the matrix without the existence of grain boundaries obviously realized. This point out that the heat treatment conditions followed in this sample simplify the diffusion of Zn from the grain boundaries into the matrix.

Energy-dispersive X-ray spectroscopy (EDX), is an analytical technique used for the elemental analysis or chemical characterization of a sample. It relies on an interaction of some source of Xray excitation and a sample. EDX pattern for the (Zn-28.5Al based) alloy is shown in Fig.(12). Using EDX analysis, the percentage of Zn found here was about 71.5 wt.% and 28.5 wt. % Al.

X-ray diffraction (XRD) scans of the deformed (Zn-28.5 Al based) Alloys samples are shown in Fig.(13). XRD analysis approved that the Aluminum and Zinc exhibit effective samples of Zn after the solidified operation.

Fig.(14) showed the variation of d-spacing [Å], with 20, the value of the angle varies from 350 to 1000 for 533 K, 553 K, and 633 K crept under constant applied stresses equal 14.275 MPa. Fig.(15): Showed the variation of Rel. Int.[%], with 20 for 533 K, 553 K, and 633 K crept under constant applied stresses equal 14.275 MPa. Fig.(16): Showed the variation of FWHM with 20 for 533 K, 553 K, and 633 K crept under constant applied stresses equal 14.275 MPa. Fig.(16): Showed the variation of FWHM with 20 for 533 K, 553 K, and 633 K crept under constant applied stresses equal 14.275 MPa.

Fig.(17): Showed the variation of Area with 2θ for 533 K, 553 K, and 633 K crept under constant applied stresses equal 14.275 MPa. Fig.(18): Showed the variation of Backgr.[cts] with 2θ for 533 K, 553 K, and 633 K crept under constant applied stresses equal 14.275 MPa.

Fig.(19): Showed the variation of Height [cts] with 2θ for 533 K, 553 K, and 633 K crept under



constant applied stresses equal 14.275 MPa. Phase graph of Al-Zn is shown in Fig. (20).

4 Discussion

The monotonic move across higher strains and lower fracture with increasing temperature was stopped at 543 K. Such reduction observed may be due mainly to some sort of change in the state of the tested material which start at this temperature and dominates for higher temperature free of the tested load [40]. Creep is believed as a thermally stimulate process to make up energy losses which may happened as a result of the presence of definite defects and their interaction with the stress-induced non-resonant bowing of dislocation segments [41]. This consideration is consistent with the increasing value of the total creep strain (ε_{tot}) and the corresponding activation energy Q obtained at higher working temperature and stress see Eq.(7)

 $\varepsilon_{\text{tot}} = A \sigma^{m} \exp\{-Q/RT\}$ (7)

With ε_{tot} the total strain rate (= total creep strain ε_{tot} / fracture time t_f).

In the present paper the kind of transient creep is presence to be pronounced subject on the working temperature. The temperature reliance of the estimated values of n and β show a sole transition temperature at 543 K. The enhanced creep of the alloys above 543 K as observed from the variation of the parameters n and β below and above this temperature might be attributed to the increased mutual solubility of the Ingredients phases above 543 K. The spotted increase in n and β , could be because of the variations in the microstructure of the phase due to the changes in their quantitative proportions [42].

According to the Al-Zn binary phase scheme Fig.(16), the solubility of Zn in Al is 28.5 wt% (this tested alloy) at 543 K, and so small at ambient temperatures. The atomic radius of Aluminum atoms compensates to 1.43 Å, while the one of Zinc is around 1.34 Å. Therefore, Zn atoms do not need to form intermetallic phases with Al atoms, which denote that the Al–Zn interactions are partly small. Small refrigeration the Al–Zn samples from an elevation temperature gives the alloys a two-phase structure above the accurate solubility [14]. The two phases are the α -phase (Al-rich phase) and the β -phase (Zn-rich phase). Heating this alloy, the b-phase dissolves in the a-phase and completely vanishes at the transformation temperature [15]

Also, the marked excess of parameters because of coarsening of the β -phase particles [16], and, hence, reduced their pinning behavior for the moving

dislocations. The abnormal increase in the parameters at the temperature of 543 K can be referred to the dissolution of the β -phase, thus making free Zn atoms which thus move to the created moving dislocations and pinning them. Above 543 K an enhanced raise of parameters with increasing the deformation temperature, this behaviour can be point out the further heating which fully dissolves the β -phase [17]. The temperature effect on the work-hardening parameters becomes dominant after the preponderance of Zinc rich phase has resolved. Through, heating increases the excess of dislocations at the top of the aggregate at grain boundaries. This process accompanied with dislocation demolition by the excessed thermal agitation [15]. The activation energy for transient is 63.8 KJ/ mole in Low temperature range, and 66.8 KJ/ mole for high temperature Range display the temperature dependence of the overall creep strain for alloys crept under various stresses [40]. The activation enthalpies (see Fig.5) point that the transient creep in the down temperature zone is controlled by dislocation intersection, while at elevation temperatures; creep is controlled by grain boundary sliding [42].

It is commonly well recognized that the inner structure of an alloy domination and modifies the nature and mode of interaction of the existing disorder and the alloy composition. So, the extreme significant conclusion of the creep tests is the enormous reliance of the creep parameters on the micro-structural changes as well as the mix element [25].

The enhanced dependence of γ ranging from 0.53-0.9 appear to be happened by the existence of Znrich phase which increase the transient and steady state creep stages. The creep distortion is set up on the mechanical properties of the constitutive phases [16]. On the basis of this statement, the difference in the strengths between the constituent phases can cause simultaneous change in the internal stress.

For steady state the current examination shows that creep tests are extra sensitive to the divergence of the samples structure, this may reflect the influence of Zn-atom on the behaviour of the samples (see Fig.1).

At low temperature the fully precipitated α -and β phases are formed. Above the transition temperature 543K, the precipitated (α - β) phase transform into the double phase (α + $\dot{\alpha}$) with the zinc- rich $\dot{\alpha}$ - phase or the conjugate solid solution of the Al-Zn system which is produced from the metastable rhombohedral R-phase when it losses some coherency. Above 623 K this double phase (α + $\dot{\alpha}$) transformation into the f.c.c. α solid solution phase. The spotted excess of the creep strain rate with growing temperature may therefore be the effect of disconnected β -phase in the α - matrix. The drain of creep spotted at transition temperature is fundamentally due to the structural change happen in the proximity of this temperature. Below the transformation temperature the anomaly might be refer to the coarsening of α - and β - grains related of Zinc and Aluminum which happen to accept the present equilibrium component. Over the transformation temperature β -phase initiate to convert into the structure of solid solution $\dot{\alpha}$ -phase.

The excess of strain rate sensitivity parameter with temperature is in accordance with past result of Al-Zn binary alloys. The amount of m show that the bossy mechanism in the low temperature region is dislocation glide as in Fig.(7), while in the high temperature region the dominating mechanism is dislocation climb [43].

Moreover, the obtained results verify the equation (5) of steady state creep: It is clear that the value of ε_{st} is increased with temperature and the increase $\ln \varepsilon_{st}$ was greater for higher degree as shown in Fig (7a). It is probable that this increase introduced screw dislocation. Because this kind of dislocations simplify dislocations cross slip during phase transformation an excess in *m* is observed as shown in Fig(7b).

The activation energy of steady state was ranged from 76.7 and 79.72 kJ/mol [18,24]. Moreover, they are regular with the activation energy for the grain boundary of Zinc and Aluminum as in Fig (8).

According to Bialobrzeski [44], the most beneficial elements with respect to corrosively are those with high solubility to the matrix as shown in Fig (9) and elements with the identical gauge galvanic potential as the Zn-Al has a few negative effects on the corrosion resistance (contrast to copper), and it depends on the fabrication and heat treatment [45], some grains are moved as a whole at the triple line.

Fig.(10) exhibit the increase of the deposition and the existance of particles at the grain boundary during different ageing temperatures. Also, indicates that numerous particles spread inside the grains of the alloy and have a spheroid shape. The typical microstructures of the studied alloys are also, shown in Fig.(11). X-ray diffraction is a useful instrument for describe the microstructure of crystalline materials as shown in Fig.(12). The preliminary results of EDAX indicate that these second phases vary with Al/Zn ratio [46]. For Aluminum-based alloys in which alloying atoms can resolve in the Aluminum-rich phase (α phase), two kinds of influence share in to the variation of the Al-rich phase lattice parameter from the lattice parameter of fine Aluminum the transfer due to resolve atoms and the moving owing to misfitting embodiment [18]. The X-ray diffraction (XRD) patterns of the tested (Zn -28.5 wt % Al based) samples are seen in Fig.(13). According to the XRD and EDS data, the continued precipitates phase $\alpha - \theta$, and Zn as light zones.

The value of d-spacing with 2θ , given in

Fig.14(a–c) for 533 K, 553 K, and 633 K crept under constant applied stresses equal 14.275 MPa. could be refer to the rest of internal strains and stresses through the diffusion of Zinc atoms in the Aluminum-rich phase which doing as indiscriminate scattering centers for X-ray integral density.

The value of Rel. Int., with 2θ given in Fig.15(a-c) for 533 K, 553 K, and 633 K crept under constant applied stresses equal 14.275 MPa. The results mean that the elevation crystallinity of the tested films. The magnitude of full-width at half-maximum-FWHM, with 20 given in Fig.16 (a-c) for 533 K, 553 K, and 633 K crept under fixed applied stresses equal 14.275 MPa it is appearing that the value of FWHM lowering with growing tested transformation temperature. Fig.17(a-c) showed the value of Area, with 20 for 533 K, 553 K, and 633 K crept under constant applied stresses equal 14.275 MPa. The value of Backgr, with 2θ given in Fig.18(a-c) for 533 K, 553 K, and 633 K crept under constant applied stresses equal 14.275 MPa it is shown that the value of Backgr increases with increasing transformation temperature. Fig.19(a-c) showed the value of Height, with 2θ for 533 K, 553 K, and 633 K crept under constant applied stresses equal 14.275 MPa it is shown that the value of Height increases with increasing transformation temperature. Fig.(20) showed Phase diagram of Al-Zn System.







Fig.(1): Isothermal Creep Curves at different applied stresses and different temperature for Zn-28.5 Al.



Fig.(2): Relation between $ln(\epsilon_{tr})$ and lnt for Zn-28.5 Al at different stresses temperatures.







Fig (3): The dependence of the parameters n, β on the working temperature at different applied stresses Zn-28.5 Al.

γ = 0.53**-** 0.9

12.0

Ln ε_{st}

hηβ

12.6



for different applied Stress.



Fig.(6): The variation of $ln\sigma$ with $ln\epsilon'_{st}$ for different applied stresses for Zn-28.5 Al.



Fig (4): The relation of ln β with ln ϵ_{st}^{\cdot} at applied Stress for tested alloy.

σ₁=12.236 MPa σ₂=13.255 MPa

=14.275 MPa

-11.4







Fig.(7): Steady-state strain rate ε_{st} and strain rate sensitivity parameter *m* as a function of creep temperature for Zn-28.5Al.









Fig.(9): SEM images of the deformed (Zn- 28.5 Al) alloys samples crept at 523 K at stress 12.236, 13.255, and 14.275 MPa, Zn precipitates in the grain boundaries in the form of dendrites and Al–rich light regions.

Fig.(8):Relation between lne[•]_{st} and 1000/T at different applied stresses for Zn-28.5Al.





(a)



(b)

Fig.(10): SEM images of the deformed (Zn-28.5 Al) alloys samples crept at 543 K at a fixed stress 13.255, and 14.275 MPa ,In this micrograph there are some thinner Zn precipitates in the grain boundaries even without dissolving in the matrix.



(a)



Fig.(11): SEM images of the deformed (Zn-28.5 Al) alloys samples crept at 623 K at a fixed at stress 13.255, and 14.275 MPa . This micrograph lets to observe completely dispersed precipitates in the matrix without the presence of grain boundaries obviously defined. This indicates that the heat treatment conditions followed in this sample simplify the diffusion of Zn from the grain boundaries into the matrix, thus ameliorative the similarity of the structure of the phases in the alloy.



Fig.(12): EDX pattern for the (Zn-28.5 Al) alloy.





Fig.(13) X-ray diffraction patterns of the tested samples crept at 533 K, 553 K, and 633 K crept at 14.246 MPa.





Fig.(14) : The variation of d-spacing [Å], with 2θ, for 533 K, 553 K, and 633 K crept under constant applied stresses equal 14.275 MPa..

















Fig.(16): The variation of FWHM with 2θ for 533K, 553 K, and 633 K crept under constant applied stresses equal 14.275 MPa.

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Fig.(17): The variation of Area.[cts] with 2θ for 533 K, 553 K, and 633 K crept under constant applied stresses equal 14.275 MPa.







Fig.(18): Showed the variation of Backgr [cts] with 2θ for 533 K, 553 K, and 633 K crept under constant applied stresses equal 14.275 MPa.













Fig.(20): Phase diagram of Al-Zn diagram

4 Conclusions

The main conclusions to be drawn from this work may be summarized as follows:

1.The transient creep parameters n and β have a strong temperature dependence exemplified by an activation energy of 63.8, 66.8 KJ/ mole for low and high temperature range respectively in confirming dislocation intersection and grain boundary sliding.

2. The strain rate sensitivity values, m, were calculated to be 0.26 at lower temperatures and 0.53 at higher temperatures, respectively, the activation energies of steady state creep of the alloy have been found to be 76.7 and 79.72 kJ/mol, in the low and high temperature regions respectively (below and above 543 K), characterizing grain boundary diffusion.

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