

SSN: 2616 - 9916

Experimental Study of High Temperature Effect on Pb-Sn Eutectic Alloy During Creep Test

Hayder Bader Naser

Department of Karbala Sewerage, Karbala Province

haiderbader76@gmail.com

Received:	9/9/2021	Accepted:	15/11/2021	Published:	22/11/2021

Abstract

The paper deals with a creep behavior of Pb – Sn eutectic alloy is studied. The creep experiments were executed at constant tensile stress (4.2, 5.5 and 6.5 MPa) in range of temperature 360 - 430K. From traditional analysis of steady – state creep is acquired the creep data. The curves creep are showed temperature effect, the stress sensitive variable, and evident activation energy of creep. The evident activation energy and stress exponent for creep were delimited as approximately 57 KJ/mole and 4 respectively. The values of n and Q are delicate to the process controlling creep deformation. Optical microscope was used to test the microstructure of samples before and after creep deformation to find the mechanisms of creep deformation. The strain of Pb-61.7wt% Sn is increased during creep test and according to subjected stress value. The best value of strain at 360K and in stress 6.5MPa about 0.3, also can be get little strain values at (360, 370,380,390K) and at 4.2MPa are (0.1,0.15, 0.2, 0.3) respectively.

Key wards: Stress sensitivity, Pb-61.7wt%Sn eutectic alloy, Creep, Activation energy.

Introduction:

The operation hours and behavior of deformation for high temperature stainless steel can be affected by the previous elastic loading. This partially effect is due to generation of intergranular strains and anisotropic plastic deformations during loading. The research is to investigate of the plastic strains of intergranular during accumulate strain creep for stainless steel (316H). The experiment of synchrotron diffraction was performed at 550 C°, where the sample is loading incrementally for different values of plastic strain flowed by displacement is controlled by static stress at each stage [1, 2].

The brittle material and fluid nature is difficult to perform processing on it. The mechanical properties is critical for mechanical machining, therefore the deformation behavior properties for high plasticity alloy crystal unstudied very well. The strain rate effect on hardness deals with in this research, whereby was investigated mechanical polishing of plane for (KDP) crystal and it depend on nano indentation technology. The strain rate is increased from 0.001 to $0.1S^{-1}$. The hardness is increased from 1.67 to 2.07. The strain rate sensitivity is calculated as 0.053, specified volume of dislocation nucleation 169 A^{"3}. According to constant load the creep deformation is studied at deeps in room temperature. The strain rate sensitivity for steady state of creep flow was estimated while the mechanism of creep is discussed [3, 4, 5].

The plastic deformation at constant load in austenitic stainless steel is tested at temperature 4K. The creep at low temperature data is taken from studies and reports about austenitic stainless steel. The creep at frosting temperature for austenitic steel common such as AISI 304, 310, 316 and strength nitrogen steel such as 304HN and 3116LN. The analysis in study is logarithmic creep (strain creep depend on log time). The present study deals with austenitic stainless steel behavior in secondary creep stage. The creep slop

Journal of University of Babylon for Engineering Sciences by University of Babylon is licensed under a Creative Commons Attribution 4.0 International License.



quid log time depends on ratio of (stress / Yield strength). The law of cold work, strain of fault energy for creep behavior at low temperature is discussed [6, 7, 8].

The laminated composite of Cu-Pb-Sn and Q235 were processed by a horizontal semisolid rolling process. The racial structure, element distribution and attributes of the composite were investigated. The simulation by finite element was conducted to analyze the temperature field and solidification process during the semisolid rolling. A suitable semi-solid zone was noticed at a casting temperature of 1598K IN The simulation, which would dynamically kept fluidity and averted casting defects. The experimental results appeared that good interface between Cu-Pb-Sn alloy and Q235 steel was attained by the mooted process at 1598K, without casting defects or excessive deformation. The Cu and Fe alloy were bonded mainly by the pervasion of Fe into Cu matrix and a handful of microscopic Pb- rich layer[9, 10, 11]. The research aim is investigated of Pb-61.7wt%Sn alloy ability to resist creep stresses at low and high temperature.

Methodology

اب ل العاروم الهندسية هم جلبة جمداهعة بمصاب للعام وم الهندسية هم جلبة جماهعة بمصاب ل العا

1-Speciemens preparation:

The specimens are done in high purity 99.9% and mixing in suitable weights from binary alloy elements and the mixture is melting with $CaCl_2$ to resist oxidation in graphite crucible. The fuse is casted in molded made of steel in shape of rode. The length of rode is 15cm and in diameter of 1cm. The casting alloy is heat treated at temperature 450K for interval time 48 hours, and then is suddenly cooled to room temperature to get of accurate grains. The alloy is drew to coils specimens in length of 50mm to conducting tensile test on it. The drawing specimens are treated at temperature 440K for interval time 4 hours and then is slowly cooled to room temperature. The cooling rate is $2*10^{-2}$ K/sec, because of allowed of grains stability. The specimens are left for interval one week before conducting the tests on it, because the grains were more stable

2-Mechanical tests:

The creep test is done by especial equipment. The one of specimen ends loading by weights. These weights are limited to generate constant tensile stress, when the specimen is connected to equipment. These specimens are put inside electric furnace. The furnace has thermal dual to control the temperature. The temperature is increased to the required temperature before start of loading. The specimen is fitted between two decoder, one is fixed and other is mover and then is subjected slowly loading. The loading begins from zero to the required stress. Figure 1 illustrates tensile mechanism used. The specimens are tested with varying of stress and temperature for each specimen. The testing is done with function of time. The curves are drew in different temperature and constant stress. The experiments of strain-time isothermal (equally temperature) under constant applied stresses (4.2, 5.5, 6.5MPa) at different temperature which located in field from 360K to 450K by changing value 10K. The elongation of coils specimens is measured by circular elongation scale (dial gage) in sensitive 10^{-5} m. The piloted error for scale in $\pm 0.5\%$. The temperature control is done of ± 1 K by using thermo computer equipment type K (Kelvin thermo computer MOD: TOUCH K). The equipment is connected with thermal sensor type K is located near of study specimen.



JOURNAL OF UNIVERSITY OF BABYLON

For ENGINEERING SCIENCES (JUBES)





Info@journalofbabylon.com | Journal.eng@uobabylon.edu.iq

Fig. 1, The equipment of Creep Test.IB-Creep30 Max. Capacity 30KN Europe(Spain).

3-Results and Discussion:

ـابـل للعلــــوم الهندسية مـــجلـة جــــاهعة بــــابـل العلـــوم الهندسية مـــجلـة جـــامعة بــــابـل العا

Figure 2, shows phases equilibrium curve double alloy Pb-Sn. The eutectic point is located at minimum of melting temperature in thermal equilibrium curve, therefore the alloy is conducted alone, composite behavior from side of melting temperature, also in nature of internal microstructure. The ascendant plasticity alloy is consist at eutectic point, and the conditions of ascendant plasticity is materialized according to references [12, 13]. The microstructure equilibrium of eutectic alloy Pb-60wt% Sn from eutectic mixture that is rich phase by lead (Pb) and rich phase by Tin (Sn). The continuous strain is depended at specified stress level during creep process on: test specimen grains size, deformation temperature, separation interaction lattice defects with melted atoms.

اب ل

ـاب ل للعلــــوم الهندسية مـــمحلـة جــــاهعة بــــاب ل للعلـــوم الهندسية مـــمحلـة جـــامعة بــــ

For ENGINEERING SCIENCES (JUBES)



Fig.2, Phases equilibrium curve for double alloy Pb-Sn. The high plasticity alloy is formed at eutectic point (Vertical line).

In metallic material there are microstructure defects which appeared with increasing the strain. This is led to stop the slipping of grains boundaries and clambering, and versa the plasticity deformation rate appeared at application of stress on material in low and high temperature. The rate of deformation can be used as a parameter to measure force of tensile stress of material. During creep test the grains boundaries shared to increase or decrease strength of material, and this depends on temperature value of deformation and applied stress and faults concentration. These factors worked separately while the final behavior is produce from previous factors.

1-The curves creep characteristics:

Figure 3 illustrates creep curves of alloy Pb-61.7wt %Sn at different deformation temperature and subjected to different applied stress. Figure 3 shows the characteristics of secondary creep after loading with primary creep is very small. The triplex creep shows clearly. This refers to that the alloy hardness return directly and stable at secondary deformation. The different of creep rate (ε) shows that the creep behavior of alloy Pb-61.7 wt%Sn (eutectic) is effected by deformation temperature in high, fast and applied stress. There is low translate for high strains by increasing stress or temperature. Hence, the creep is increased by increasing of applied stress level and applied temperature. The rate of creep strain is limited at any time by strain creep derivative for time. The low value of rate ($\delta\varepsilon$ / δ t) as rate of creep strain for constant stage (ε _{st}). Figure 4 illustrates the relationship between calculate values for (ε _{st}) by function of creep temperature at constant applied stress. There is increasing in (ε _{st}) by the effect of creep temperature and constant applied stress during three stages of temperature.



م جلة جـاهعة بـــابـال للعلـــوم الهندسية مـــجلـة جـــاهعة بــــابـال للعلـــوم الهندسية مـــجلـة جـــاهعة بــــابـال للعلـــوم الهندسية

JOURNAL OF UNIVERSITY OF BABYLON

For ENGINEERING SCIENCES (JUBES)

Vol.29, No.3. | 2021



ISSN: 2616 - 9916





0.0022 0.0020 4.2MPa a 0.0018 5.5MPa 6.5MPa 0.0016 0.0014 0.0012 Strain rate 0.0010 0.0008 0.0006 0.0004 0.0002 0.0000 -0.0002 350 360 370 380 390 400 410 420 430 440 Temperature(K)



3-2, Constant state creep rate on stress:

It is possible to get the rate of creep strain as a constant state (ε_{st}) in constant temperature and applied stress according to the law [14]:

$$\varepsilon_{\text{st.}} = B. \sigma^n \dots (2)$$



ISSN: 2616 - 9916

www.journalofbabylon.com

info@journalofbabylon.com | Journal.eng@uobabylon.edu.iq

لم ل العلــــوم الهندسية مـــمحلة جــــامعة بــــابـل العلـــوم الهندسية مـــجلـة جـــامعة بــــابـل العلـــ

strain rate of constant state $(\varepsilon_{st.} = \frac{\partial \varepsilon}{\partial t})$

-6.0

-6.5

-7.0

-7.5

B : constant, n : stress exponential $\left(\frac{\partial ln\varepsilon_{st.}}{\partial ln\sigma}\right)$ The creep curve in figure 3 gives creep

From linear part inclined for strain curve with time. Stress exponential (n) can be get as shown in figure (5) by using squares methods and take the line best for suitable points as function in temperature as illustrated in figure (6). The value (n) depend on deformation temperature, and from figure (6) the value of (n) decreases with the increase of deformation temperature. The present study shows that the applied stress at different temperature increases with the increase of strain rate for constant state (ε_{st}). The applied stress

mainly depends on reactions between establish separations from continues deformation. The centers are not let separation motion in specimens of solid solution. Figure (6) shows that the decrease of (n) with temperature due to the increase of strain rate sensitive (m), also figure (6) refers to get arranging because of

thermal acerbating at high temperature. Therefore, the separation motion is easy at high temperature and low values of stress exponential at this range of temperature. The creep deformation is limited by (n) value, hence, the values of (n) between (3-8) represented type of creep technique separation [15, 16]. The values

of (n) 2.8 to 4.3 that the separation climbing is control technique of creep deformation.





.

88

Fig. 5, the relationship between Lne^{-st} and Lno for PD-01.7-wt%Sn alloy at different temperature test.

Journal of University of Babylon for Engineering Sciences by University of Babylon is licensed under a <u>Creative</u> <u>Commons Attribution 4.0 International License</u>.

For ENGINEERING SCIENCES (JUBES)







Fig. 6, The relationship between exponential stress (n) with temperatures.

In general, the high plasticity when (m) is equal or bigger than 0.3, where m is calculate from the following equation [17]:

$$\sigma = C * \varepsilon_{\text{st.}} \dots (3)$$

Where, C: constant, m: strain rate sensitive.

ـابـل للعلــــوم الهندسيـة مـــجلـة جــــاهعة بــــابـل العلـــوم الهندسيـة مـــجلـة جـــاهعة بــــابـل العا

The value of m for Pb-61.7 wt%Sn is less than 0.3 at low temperature in range (360-390K), therefore the high plasticity is not occur in this range. The value of m for Pb-61.7 wt%Sn is increased on 0.3 at high temperature in range (400-430K). The m is located in range (0.3-0.35), and this refers that the high plasticity occurred in this range. The values of m for this alloy is increased with homogenous temperature, and this refers to that the narrow strength of alloy Pb-61.7wt%Sn increased with increasing the temperature.

3-3, Evaluate of creep activation energy:

To evaluate activation energy (Q_C) of creep process from Arenose equation (log ε_{st} =f(1/T)) as shown in figure 6, which gives straight lines in order to different applied stresses at different temperature. The activation energy calculation for constant state creep depends on applied stress. Greatly, the value of experimental activation energy and stress exponential are used to evaluate control technique in deformation process. The deformation occurred for crystals multiple materials at temperature is greater than (0.5 T_m) by different deformation technique that is connected by value of stress exponential and activation energies. The slipping viscous separation is characterized in stress exponential value (3) at high temperature, and in (5) value at low temperature. The suitable activation energies is equal of spread for melted and separation spread,

Journal of University of Babylon for Engineering Sciences by University of Babylon is licensed under a Creative Commons Attribution 4.0 International License.



ISSN: 2616 - 9916

www.journalofbabylon.com

Info@journalofbabylon.com | Journal.eng@uobabylon.edu.iq

where separation climbing is control technique in pure metal at high temperature with value of (n) in rang (4-6) and activation energy is equal to self-spread activation through mesh.

The present alloys have different creep technique is showed in high and low range of temperature. The mean of creep activation energy of constant state at low temperature is in range (360-390K) in value (49 KJ/mole), while the mean of creep activation energy of constant state at high temperature in range (400-430K) in value (51 KJ/mole). The mean grain dimensions measured by scanning microscope in mechanical laboratory of Almusaib institute as shown in figure7. This is suitable with activation energy of other alloys as shown in table 1. This refers to that the weight concentration is very important in grains grow, therefore these alloys are different [18].



Fig.7, The scanning microscope.

Table 1, Characteristics comparative of constant state creep for alloy with other alloys.

Alloy	Melting Temperature K	Applied Stress (MPa)	Elongation %	Sensitive rate	Activation energy Q(KJ/mole)	Grain dimension mean(µm)	References	
Pb- 61.7wt%Sn(eutectic)	455	4.2-6.5	185	0.25-0.37	50-57	3.5-5.5	Present work	
Pb-10Sn(binary)	588	12.85-23.2	18	0.18-0.45	47.2-89.2	71	[15, 16]	
Pb-Sn(eutectic)	456	7.89- 12.92	72	0.56-0.9	43-64	5.5-9.5		

Journal of University of Babylon for Engineering Sciences by University of Babylon is licensed under a Creative Commons Attribution 4.0 International License.

وه الهندسية

_____ابل

عاب ل العانوم الهندسية من جلبة جنهمة ب

لب ل العام وم الهندسية م دلة ج مهعة ب

ولتخ والمتحتج ب

JOURNAL OF UNIVERSITY OF BABYLON

For ENGINEERING SCIENCES (JUBES)







Fig.8a,b, The relationship between lnɛ⁻st and inverse of temperature (1000/T) at different applied stress for Pb-61.7wt%Sn alloy: (a)at low temperature (b)at high temperature.

3-4 the microstructure:

The microstructure of alloy for creep specimens at different temperature and applied stress can be shown by using photosynthesis microscope. Figure 9 illustrates specimens microstructure at constant stress (6.5MPa) and at temperature (360, 420, 430K) respectively.

It shows the changing of microstructure deformation with temperature change.

Journal of University of Babylon for Engineering Sciences by University of Babylon is licensed under a <u>Creative</u> <u>Commons Attribution 4.0 International License</u>.



Vol.29, No.3. | 2021

b-360K, 1200X



d-430K, 1200X c-420K, 1200X Fig.9, The specimens microstructure of Pb-61.7wt%Sn alloy at lissome 430K for interval 4.5hours (a) Befor creeping. (b,c,d) After creeping at constant stress 6.5MPa.

Conclusions:

*جل*ة جـــامعة بــــابـل للعلـــوم الهندسية مـــجلـة جــــامعة بــــابـل للعلـــوم الهندسية مـــجلـة جـــامعة بــــابـل للعاـــ

Based on the experimental work,

a-Before Creep,1200x

- 1-The strain rate sensitive for constant state (m) and activation energy (Q) are increased with increasing of deformation temperature while decreased of stress exponential (n).
- 2-The strain rate sensitive for constant state is changed from 0.23 to 0.27 in rang of experimental temperature, and called separation climbing technique.
- 3-The value of creep activation energy for constant state above of transformation temperature 56 KJ/mole.
- 4-The value of (n) is convergence with creep experimental value for same alloy. The range of stress exponential 4.6 - 3.7 for Pb-61.7wt%Sn is occurred in it. The creep deformation technique is appeared due to separation climbing.

Journal of University of Babylon for Engineering Sciences by University of Babylon is licensed under a Creative Commons Attribution 4.0 International License.



SSN: 2616 - 9916

www.journalofbabylon.com

nfo@journalofbabylon.com | <u>Journal.eng@uobabylon.edu.iq</u>

- [1] Al Mamun A.1, Reinhard C., Mostafavi M., EFFECT OF PLASTICITY ON CREEP DEFORMATION IN TYPE 316H STAINLESS STEEL, Proceedings of the ASME 2019 Pressure Vessels & Piping Conference PVP2019
- [2] Eyiroshe, T. O., et al , influence of prior cyclic plasticity on creep deformation using crystal plasticity modeling, IJSS, ISSN:139-140, p. 129-137.3-
- [3] Mao J. 1, 3Liu W. 1, Li D.1, Zhang Ch. 1 and Ma Y. 2,* The Strain Rate Sensitivity and Creep Behavior for the Tripler Plane of Potassium Dihydrogen Phosphate Crystal by Nanoindentation, Micromachines 2021, 12, 369. <u>https://doi.org/10.3390/mi12040369</u>.
- [4] Borc, J.; Sangwal, K.; Pritula, I.; Dolzhenkova, E. Investigation of pop-in events and indentation size effect on the (001) and (100) faces of KDP crystals by nanoindentation deformation. Mater. Sci. Eng. A 2017, 708, 1–10.
- [5] Bach, J.; Stoiber, M.; Schindler, L.; Höppel, H.; Göken, M. Deformation mechanisms and strain rate sensitivity of bimodal and ultrafine-grained copper. Acta Mater. 2020, 186, 363–3.
- [6] R. P. Reed1 and R.P. Walsh2 Low-temperature creep of austenitic stainless steels, IOP Conf. Series: Journal of Physics: Conf. Series 897 (2017) 012002.
- [7] Jing, H.; Guo, H.;Wang, L.;Wei, J.; Xu, L.; Han, Y. Influence of Ag-modified graphene nanosheets addition into Sn–Ag–Cu solders on the formation and growth of intermetallic compound layers. J. Alloys Compd. 2017, 702, 669–678
- [8] Wu, J.; Xue, S.; Wang, J.; Wu, M. Coupling effects of rare-earth Pr and Al2O3 nanoparticles on the microstructure and properties of Sn-0.3Ag-0.7Cu low-Ag solder. J. Alloys Compd. 2019, 784, 471–487.
- [9] Yubo Zhang 1,2,*, Jiaming Liu 1, Ying Fu 3, Jinchuan Jie 1, Yiping Lu 1, Qingtao Guo 2, Tongmin Wang 1 and Tingju Li 1,*, Microstructure and Fabrication of Cu-Pb-Sn/Q235 Laminated Composite by Semi-Solid Rolling, Metals 2018, 8, 722; doi:10.3390/met8090722.
- [10] Azimi, M.; Toroghinejad, M.R.; Shamanian, M.; Kestens, L. The Effect of strain on the formation of an intermetallic layer in an Al-Ni laminated composite. Metals 2017, 7, 445.
- [11] Zhang, J.; Liu, S.; Lu, Y.; Jiang, L.; Zhang, Y.; Li, T. Semisolid-rolling and annealing process of woven carbon fibers reinforced Al-matrix composites. J. Mater. Sci. Technol. 2017, 33, 623–629.
- [12] Dronskowski R., First-Principles Approaches to Metals, Alloys, and Metallic Compounds, Metals 2018, 8, 705; doi:10.3390/met8090705.
- [13] William Harrison, Mark Whittaker and Veronica Gray, Advanced Methods for Creep in Engineering Design, http://dx.doi.org/10.5772/intechopen.72319
- [14] Sevsek, S.; Bleck, W. Ab Initio-Based Modelling of the Yield Strength in High-Manganese Steels. Metals 2018,8,24.
- [15] El-Daly, A. A. Abdel-Daiem, A. M. Abdel-Rahman, A. N. Mohammed, S. M. 2004. Effect of Znaddition and structural transformation on the creep behaviour of Pb–10 wt.% Sn alloy. Materials Chemistry and Physics 85, pp.163-170.

Journal of University of Babylon for Engineering Sciences by University of Babylon is licensed under a <u>Creative</u> <u>Commons Attribution 4.0 International License</u>.



[16] Heba Y. Zahran 1,2, Ashraf S. Mahmoud 2 and Alaa F. Abd El-Rehim, Effect of Bi Content on the



ISSN: 2616 - 9916

www.journalofbabylon.com

Info@journalofbabylon.com | Journal.eng@uobabylon.edu.ig

Microstructure and Mechanical Performance of Sn-1Ag-0.5Cu Solder Alloy, Crystals 2021, 11, 314. https://doi.org/10.3390/cryst11030314.
[17] WhittakerMT,HarrisonW,Hurley PJ,Williams S. Modelling the behaviour of titanium alloys at high temperature for gas turbine applications. Materials Science and Engineering: A.2010;527(16):4365-4372

[18] Gray V, Whittaker M. Development and assessment of a new empirical model for predicting full creep curves. Materials. 2015;8(7):4582-4592.





دراسة تجريبية لتأثير درجة الحرارة العالية على سبائك الرصاص زنك اليوتكتك خلال اختبار
الزحف
حیدر بدر ناصر
دائرة مجاري كربلاء، محافظة كربلاء
haiderbader76@gmail.com

الخلاصة

البحث تناول دراسة سلوك الزحف لسبيكة الرصاص – القصدير (اليوتيكتيك). وأجريت تجارب الزحف عند إجهادات شد ثابتة (٢,٢، ٥,٥، ٥,٢ ميكا باسكال) في نطاق درجات الحرارة (٢٠٩ – ٣٦٠ كلفن). تم تحليل تقليدي لعمليات الزحف المستقرة والحصول على المعلومات المطلوبة. أظهرت منحنيات الزحف تأثير درجات الحرارة، متغير تحسس الإجهاد، وتوضيح طاقة تنشيط الزحف. أن طاقة التنشيط الواضحة وأس الإجهاد للزحف تحدد بالقيم التقريبية ٥٧ كيلو جول /مول و٤ بالتناوب. أ، قيم n وp تم التحسس بها بعملية السيطرة على نشوه الزحف. المجهر البصري تم استخدامه لفحص البنية البلورية للنماذج قبل وبعد نشوه الزحف ومتغيراتها. أن أنفعال مسبيكة Pb-61.7% Sn تزداد خلال فحص الزحف اعتمادا على قيمة الإجهاد المسلط. أن أفضل قيمة لانفعال السبيكة عند درجة حرارة عند أجهاد وإجهاد SMP حرارة (٥.3, 380,390K) وإخد المتعال واطئة عند درجات حرارة (٥.3, 380,390K) وإجهاد موات حرارة (٥.3, 30, 300, 50.)

deals

الكلمات الدالة: - حساسية الاجهاد، سبائك اليوتكتك، الزحف، طاقة التشغيل.

Journal of University of Babylon for Engineering Sciences by University of Babylon is licensed under a <u>Creative</u> Commons Attribution 4.0 International License. SSN: 2616 - 9916

nfo@journalofbabylon.com | Journal.eng@uobabylon.edu.iq | www.journalofbabylon.com