

IIAC 2014





Iranian Mines and Mineral Industries Development and Renovation Organization

## مرکز تحقیقات آلومینیوم ایران Iran Aluminium Research Centre

# This certificate is awarded for paper entitled as:

Enhanced Superplasticity in AA2024 Aluminium Alloy Processed by Multiple Compressions in a Channel Die

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### which was presented in

Iran International Aluminium Conference 2014 (25-26 May)

## Chairman, M.Soltanieh

Iran International Aluminium Conference 2014, Tehran.

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# Enhanced superplasticity in AA2024 aluminium alloy processed by multiple compressions in a channel die.

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**Abstract** : Superplasticity of AA2024 aluminium alloy processed by multiple compressions in a channel die was investigated by impression testing in the temperature range of 613-703 K. The values of strain rate sensitivity parameter of 0.49-0.56 and activation energy of 73 kJ/mol were obtained by impression creep technique. Based on the obtained results, it can be said that, the processed material exhibit superplastic deformation with the possible creep mechanism of grain boundary sliding. The studied material deformed superplastically in the strain rate range of  $10^{-3}$ - $10^{-2}$  s<sup>-1</sup> which is greater than ranges in which superplastic deformation usually occurs.

Keywords: multiple compressions in a channel die; superplasticity; strain rate sensitivity.

#### Introduction

Superplastic deformation occurs at typical forming rates of  $10^{-4}$ - $10^{-3}$  s<sup>-1</sup>. Because of these small deformation rates, the superplasticite forming industry is confined to a relatively small manufacturing niche associated primarily with the forming of high- value low-volume components for the aerospace and other similar industries [1]. Thus, increasing the rate of superplastic forming, can expand the utilization of this forming technique into the fabrication of high- volume parts for the automobile and consumer product industries.

In recent years, bulk ultra fine grain materials produced by severe plastic deformation (SPD) methods drew considerable interest among materials scientists and engineers. Using SPD techniques, materials can be deformed to very large strains and hereby several of the physical and mechanical properties can be improved [2]. Processing through the imposition of sever plastic deformation provides the potential for achieving grain refinement to sub micron or even the nanometre level. Furthermore, if the ultra fine grains are reasonably stable at elevated temperatures, typically at temperatures above 0.5 Tm, where Tm is the absolute melting temperature, the processed materials are capable of exhibiting excellent superplastic deformation behaviour [3]. Superplasticity of ECAP processed copper [4], aluminium alloys [5,6] and magnesium alloys [7,8] has been studied extensively. Recently, some researches have been carried out for investigating the application of ECAP process to achieve superplastic forming capability in soft Sn-based alloys [9-11]. Moreover, occurrence of superplastic deformation behaviour in materials subjected to high pressure torsion has been also reported [12,13].

superplasticity, impression techniques are of special interest.

This is because they can be particular advantages when the material is only available as small test pieces [14]. The superplasticity of Sn-37% pb eutectic [15], Sn-40% pb-2.5% sb peritectic [16], Sn-5% sb [10] and Zn-22% Al eutectoid [17] alloys, studied by the impression method, has shown that the parameters commonly considered as characteristics of superplasticity, like strain rate sensitivity index, m, can be obtained by this simple test. The short review presented here, shows that despite investigations about superplastic deformation behaviour of materials subjected to ECAP and HPT, no study of the superplasticity of multiple compressions in a channel die by either conventional testing or impression method has been reported. The aim of this work is thus to study the potential of multiple compressions in a channel die for developing superplasticty in AA2024aluminium alloy using impression creep method.

#### Experimental Procedure Material and processing

The material used was a commercial wrought Al-2024 alloy, the chemical composition of which is shown in table 1. From this material specimens with dimensions 15 mm  $\times$ 14.8 mm  $\times$  7.5 mm were cut and used for subsequent processing. The specimens were fully annealed at 673K for 1 horse followed by slow cooling to room temperature. The appearance of the annealed microstructure is shown in fig 1. This figure shows the annealed specimens contain small particles dispersed

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more or less uniformly. These particles were identified as  $CuMgAl_2$  [18].

Table 1: Chemical composition of the Al 2024 alloy (wt%)

Cu	Mg	Si	Mn	Fe	Al
4.05	1.43	0.43	0.38	0.32	Balance

The annealed specimens were compressed several times in a channel die. The pressing were carried out at room temperature using a ram speed of 25 mms-<sup>1</sup> and mineral oil used as lubricant. A specimen of height 15 mm and  $14.8 \times 7.5 \text{ mm}^2$  section was placed in the die in such a way that 14.8 mm side just slides into the 15 mm wide channel. Fig 2. Shows the deformation scheme during multiple compressions in a channel die. The sides of the specimen are designated as A, B and C. The first pressing was carried out on face A to half of the height of the sample.

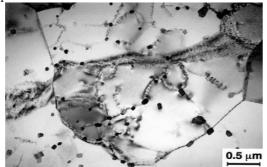


Figure 1: Microstructure of the Al-2024 alloy in the fully annealed condition [18].

This makes the side which was previously 7.5 mm to be about 14.8 mm. The specimen was then rotated such that the second pressing was carried out on face B. The 14.8 mm side slides into 15 mm wide channel and becomes the height for the next pressing. After the second pressing, the specimen was rotated such that the next pressing was carried out on face C. After each pressing, the specimen was rotated and reinserted into channel such that the specimen dimension equal to channel width becomes the height for the next pressing. This sequence ensures that the sample is pressed in all three directions.

It is worth noting that after each pressing pass, the grinding carried out on the bulged portion of the sample to preserve its initial dimensions. The above mentioned stages were repeated up to 7 passes ( $\varepsilon = 5.6$ ). The multiple compressions in a channel die sequence has been illustrated in fig 2, schematically.

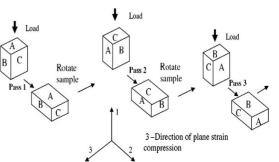


Figure 2: Schematic representation of the sequence of multi-Axial forging process.

#### **Microstructural observation**

The microstructural observation by transmission electron microscopy (TEM) was done for the multiple compressed in a channel die specimens. To prepare TEM thin foils, specimens were ground to a thickness of about 100µm and then twin-jet electropolished using a 33% HNO3 and 66% CH3OH solution at -30<sup>®</sup>C and 20V. EM 208 F transmission electron microscope operated at 100 kV was used for Microstructural examination.

#### **Impression creep tests**

The rectangular samples with dimensions of 12 mm ×7 mm were cut from the multiple compressed specimens in a channel die for impression testing. Constant-load impression creep tests were carried out by a SANTAM universal tensile testing machine equipped with a threezone split furnace. A flat-ended cylindrical punch of 2mm diameter was mounted in a special holder located in the center of the vertical loading bar. The 6mm thick sample were laid on an anvil below the loading bar, and this assembly inserted into the split furnace. Impression creep tests performed under punching stresses in the range 8-72 MPa and in the temperature range 613-703K for dwell times up to 180 min. The impression depth was measured automatically as a function of time and the data were recorded by the computer. The accuracy of load, impression depth and testing temperature measurements were,  $\pm 0.1N$ ,  $\pm 0.001$  mm, and  $\pm 1K$ , respectively.

#### **Results and Discussions**

Typical impression creep curves; representing penetration depth of the punch as a function of dwell time under different punching stress levels tested at 613K for the multiple compressed specimens after 4 and 7 passes are shown in Fig 3, a and b, respectively. As it can be seen although each of the individual impression curves does not always exhibit a pronounced primary creep stage, they all eventually show a steady state region where depth increases linearly with time. The steady state impression velocity can be determined by plotting dh/dt against time.

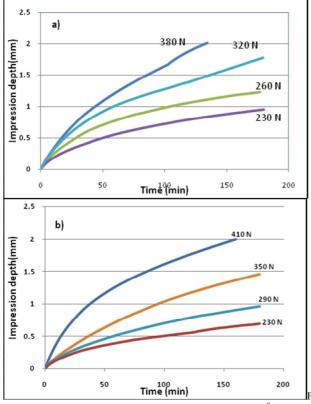
It is generally accepted that the mechanical behaviour of metallic materials at homologous temperatures higher than 0.5 can be fairly expressed by the power law creep in a wide range of strain rate. Thus for steady state creep,



the high temperature creep rate, z, is described by the following formula:

$$\dot{\varepsilon} = A(\sigma)^{1/m} exp\left(-\frac{Q}{RT}\right) \tag{1}$$

where A is a material parameter,  $\sigma$  is the applied stress, m denotes the strain rate sensitivity index , Q is the activation energy, T is the temperature, and R is the universal gas constant.



igure 3: Impression creep curves of the material tested at  $340^{\circ}$ C. a) pass 4, b) pass 7.

To correlate impression and tensile creep data, equivalent stress and strain rate can be evaluated from the impression velocity, dh/dt and the pressure under the punch, ( $I = 4F/\pi d^2$ ) at a given load, F, and punch diameter, d, as:

$$\sigma = c_1 P$$
 and  $\varepsilon = c_2 \frac{dh/dt}{d}$  (2)

Where  $c_1 \approx 1/3$  and  $c_2 \approx 1$  are constants for a wide range of materials [19]. It has been experimentally shown that both steress and temperature dependencies of the steady state impression velocity agree with the corresponding dependencies of the creep rate in conventional creep tests [20,21]. Combining Eqs(1) and (2), the strain rate sensitivity index, m, can be determined by :

$$m = \left[\frac{\partial \ln \sigma}{\partial \ln(\frac{dh}{dt})}\right]_{T}$$
(3)

Equation 3, shows that, m, can be considered as the slope of the plot of stress against impression rate on a log-log scale. Using Eqs 1 and 2 after some mathematical calculations, the activation energy, Q, can be expressed as:

$$Q = -K \left[ \frac{\partial \ln(\frac{dh}{dt})}{\partial (\frac{1}{T})} \right]_{g,m}$$
<sup>(4)</sup>

Thus, a semi logarithmic plot of the steady-state impression rate (dh/dt) versus (1/T) has a slope of the activation energy, Q.

The punching stress was plotted as a function the steadystate impression velocity on a double logarithmic scale, and shown in Fig 4 for the fully annealed sample and multiple compressed specimens after 2,4 and 7 passes at the test temperature of 613K. It can be seen that the slope of the lines increases by increasing the number of pressing passes.Based on Eq. 3 the slope of each of the lines yields the corresponding strain rate sensitivity parameter. The m values obtained by finding the slop of the lines have been summarized in table 2.

Since the specimens subjected to 7 passes of multiple compressions in a channel die exhibit a high m value, around 0.5, at the test temperature of 613K, the m value temperature depency of which was investigated. The plot of punching stress versus steady state impression velocity on a log-log scale is shown in fig 5, it is clear that all lines in the figure are almost parallel.

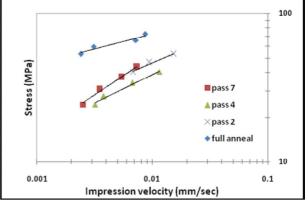


Figure 4: The flow stress as a function of steady-state impression rate at 613K for the 7, 5, 2 pass and full annealed Material.

Table 2: Strain rate sensitivity from impression creep experiments as a function of number of pressing passes.

state	Strain rate sensitivity index, <i>m</i>	
Full anneal	0.20	
Pass 2	0.34	
Pass 4	0.38	
Pass 7	0.52	



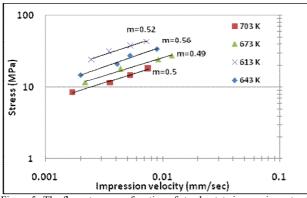


Figure 5: The flow stress as a function of steady-state impression rate at various temperatures.

Based on line slopes the m values of the studied specimens were found to be in range of 0.49-0.52. The steady state impression rate were plotted against reciprocal of the absolute temperature on a semi logarithmic scale, for the specimen subjected to 7 pressing passes of multiple compressions in a channel die, as shown in fig 6, to obtain the activation energy values. It is clear that the curves exhibit a single linear behaviour. therefore, it can be said that the activation energy of the examined specimen is stress independent. The activation energy values of the tested samples were found to be in the range of 71-74 kJ/mol, as indicated by the curve slopes. These values are comparable to the activation energy of 77.4 kJ/mol for grain boundary diffusion in aluminium [24]. The obtained activation energy together with the strain rate sensitivity index imply that the multiple compressed specimens after 7 pressing passes deform superplastically with the dominant creep mechanism of grain boundary sliding at 613-703K temperature interval and in the strain rate range of 10<sup>-3</sup>-10<sup>-2</sup>s<sup>-1</sup>

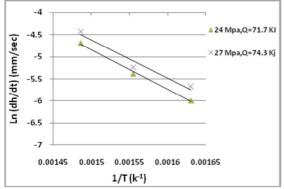


Figure 6: Temperature dependence of the strain rate.

Occurrence of superplasticity in AA2024 aluminium alloy subjected to thermomechanical processing has been reported in the strain rate rang of  $5 \times 10^{-4}$ -**1.5 × 10<sup>-2</sup>s<sup>-1</sup>** and at temperatures of 723-785K. The strain rate sensitivity parameter of 0.35-0.4 obtained for this material under the conditions mentioned above [24,25]. It is worth noting that the superplastic properties of the thermomechanically processed specimens gathered by uniaxial tensile tests with a constant initial strain rate.

Comparing the superplasticity of multiple compressed specimens after 7 pressing passes with that of thermomechanically treated samples, it can be said that the former deforms superplastically at lower temperature and in higher strain rate than those of the latter. The thermomechanically processed samples average grain size was reported in the range of 5-8  $\mu$ m [25] while that of the multiple compressed specimens after 7 pressing passes was in the range of 0.35-0.5  $\mu$ m, as indicated in fig7.The observed difference in superplastic deformation behaviour of the two differently processed samples can be attributed to the corresponding microstructure.

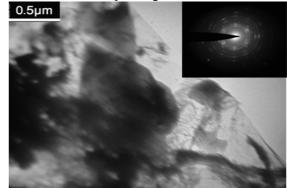


Figure 7: Microstructure and SAED pattern of the Al-2024 alloy multiple compressed in a channel die after 7 pressing passes tested at 673K for 180 min.

#### Conclusion

1. Multiple compressions in a channel die can be used for developing superplasticity in AA2024 aluminium alloy. It is experimentally shown that 7 pressing passes is required for achieving superplasticity.

2. The strain rate sensitivity calculated in this study for multiple compressed specimens after 7 pressing passes is greater that of thermomechanically treated samples.

3. The multiple compressed specimens after 7 pressing passes deform superplastically in the strain rate range of  $10^{-3}$ - $10^{-2}$ s<sup>-1</sup> (greater than the range in which superplasticity occurs in the thermomechanically treated samples) and at 613-703K temperature interval (less than the interval at which the samples subjected to thermomechanical processing, deform superplastically.

4. Based on the calculated m and Q values it can be said that grain boundary sliding is the dominant mechanism for the deformation of multiple compressed samples after 7 pressing passes.

#### References

[1] A. Barnes. "Superplastic aluminum forming expanding its techno-economic niche" in Materials science forum. 1999: Trans Tech Publ.



[2] I. Charit and R.S. Mishra, "High strain rate superplasticity[4] K. Neishi, Z. Horita and T.G. Langdon, "Achieving in a commercial 2024 Al alloy via friction stir processing" superplasticity in ultrafine-grained copper: influence of Zn *Materials Science and Engineering: A. 359* (2003) 290-296. and Zr additions" *Materials Science and Engineering: A.* 352 [3] S.V. Dobatkin, E.N. Bastarache, G. Sakai, T. Fujita, Z.(2003) 129-135.

Horita and T.G. Langdon, "Grain refinement and superplastic[5] M. Ferry and N. Burhan, "Microstructural evolution in a flow in an aluminum alloy processed by high-pressurefine-grained Al–0.3 wt.% Sc alloy produced by severe plastic torsion" *Materials Science and Engineering: A. 408* (2005)deformation" *Scripta materialia. 56* (2007) 525-528.
141-146. [6] J. Gubicza, N. Chinh, T. Csanadi, T. Langdon and T.

fcc metals" *Materials Science and Engineering: A.* 462 (2007) 86-90.

[7] R.B. Figueiredo and T.G. Langdon, "The development of superplastic ductilities and microstructural homogeneity in a magnesium ZK60 alloy processed by ECAP" *Materials Science and Engineering: A. 430* (2006) 151-156.

[8] Y. Miyahara, Z. Horita and T.G. Langdon, "Exceptional superplasticity in an AZ61 magnesium alloy processed by extrusion and ECAP" *Materials Science and Engineering: A. 420* (2006) 240-244.

[9] S. Lee, "Superplastic properties of Pb-62% Sn eutectic alloy after equal channel angular pressing (ECAP)" *Journal of Materials Processing Technology. 201* (2008) 441-444.

[10] R. Mahmudi, H. Mahjoubi, P. Mehram, "superplastic indentation creep of fine-grained Sn-1%Bi alloy" Proceedings of the 1<sup>st</sup>, international conference of ultrafine grained and nano structured material,(2007).

[11] R. Mahmudi, R. Alizadeh and A. Geranmayeh, "Enhanced superplasticity in equal-channel angularly pressed Sn–5Sb alloy" *Scripta materialia*. *64* (2011) 521-524.

[12] R.S. Mishra, R. Valiev, S. McFadden, R. Islamgaliev and A. Mukherjee, "High-strain-rate superplasticity from nanocrystalline Al alloy 1420 at low temperatures" *Philosophical Magazine A.* 81 (2001) 37-48.

[13] G. Sakai, Z. Horita and T.G. Langdon, "Grain refinement and superplasticity in an aluminum alloy processed by high-pressure torsion" *Materials Science and Engineering: A. 393* (2005) 344-351.

[14] R.Mahmudi, R.Roumina and B. Raeisinia, "Investigation of stress exponent in the power-law creep of Pb–Sb alloys" *Materials Science and Engineering: A. 382* (2004) 15-22.

[15] G. Cseh, N. Chinh and A. Juhasz, "Indentation curves and viscosity measurements on glasses" *Journal of materials science letters*. *17* (1998) 1207-1209. [6] J. Gubicza, N. Chinh, T. Csanadi, T. Langdon and T. Ungar, "Microstructure and strength of severely deformed [16] R. Mahmudi and A. Rezaee-Bazzaz, "Superplastic power-law creep of Sn-40% Pb-2.5% Sb peritectic alloy" *Journal of materials science.* 42 (2007) 4051-4059.

[17] R. Hooper and C. Brookes, "Incubation periods and indentation creep in lead" *Journal of materials science*. *19* (1984) 4057-4060.

[18] S. Lee, M. Furukawa, Z. Horita and T. Langdon, "Developing a superplastic forming capability in a commercial aluminum alloy without scandium or zirconium additions" *Materials Science and Engineering: A. 342* (2003) 294-301.

[19] A. Juhasz, P. Tasnadi, P. Szaszvari and I. Kovacs, "Investigation of the superplasticity of tin-lead eutectic by impression creep tests" *Journal of materials science.* 21 (1986) 3287-3291.

[20] F. Yang and J. Li, "Impression test of 63Sn 37Pb eutectic alloy" *Materials Science and Engineering: A. 201* (1995) 40-49.

[21] S. Chu and J. Li, "Impression creep; a new creep test" *Journal of materials science*. *12* (1977) 2200-2208.

[22] A. Rezaee-Bazzaz and R. Mahmudi, "Impression creep of Sn-40Pb-2· 5Sb peritectic solder alloy" *Materials science and technology. 21* (2005) 861-866.

[23] N. Kumar, K. Raman, D. Sastry and E. Little, "Investigation of superplasticity by the impression creep technique" *Journal of materials science*. *25* (1990) 753-755.

[24] W. Zheng and Z. Baoliang, "Superplasticity in the 2024AI alloy" *Journal of materials science letters*. *13* (1994) 1806-1808.

[25] L. Rus, M. Tintelecan, M. Sas-boca, D. Noveau and I. Marian, "Superplastic behavior of 2024 aluminum alloy sheet subjected to termomechanical processing" Annals of the University Dunarea de Jos of Galati: Fascicle IX, Metallurgy & Materials Science. 29 (2011).