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Stress Relaxation Studies on Cryorolled AA 5083 Alloy

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Abstract

Cryorolling is one of the established severe plastic deformation (SPD) routes to obtain the ultra-fine grained (UFG) sheet metals. The final grain size obtained during the process is an important measure of the mechanical properties. Due to the intense rolling strain, it is practically difficult to measure the grain size by optical methods. Alternately, a transient mechanical test, stress relaxation is a proven method to verify the grain refinement. In this method, the transient data from stress relaxation is used to calculate the activation volume. Since the deformation mechanism changes with grain refinement, this test is widely adopted to verify the UFG structured metals and alloys. In the present work, cryorolling is performed using Al-Mg (AA 5083) alloy. Controlled uniaxial tests were performed on the as-received (solutionized) and cryorolled samples. The difference in activation volume of solution treated and cryorolled samples is used to explain the grain refinement mechanism.

Keywords: Severe Plastic Deformation, Cryorolling, Stress relaxation, Grain Refinement.

1. INTRODUCTION

Ultra-fine grained (UFG) materials with grain size in the range of 100nm-1μm have been receiving significant attention towards micro-fabrication and precision engineering due to their high mechanical homogeneity in the micron range and improved mechanical properties as compared to that of coarse-grained (CG) materials[1]. Recently, UFG materials are used in micro-forming which is defined as the production of metallic parts by forming with dimensions in the sub-millimeter range to produce micro metal products such as miniature screws, micro gears, connector pin etc. [2]. The application of UFG for precision applications such as micro-forming require homogenous grain size[3]. Therefore, it is important to achieve uniform grain size in UFG materials. Severe plastic deformation (SPD) is an effective way to produce UFG materials[4]. Equal channel angular pressing (ECAP)[5], High- pressure torsion (HPT)[6], Accumulative roll bonding (ARB)[7], Cryorolling (CYR)[8] and Constrained groove pressing (CGP)[9] are various SPD processes capable of producing sub-microcrystalline solids. Cryorolling constitutes thermo mechanical processing route to produce ultra-fine grained sheets of pure metals and alloys to enhance the mechanical strength[10]. Owing to certain advantages of cryorolling such as less plastic strain required to obtain UFG materials over other SPD processes and the ability to use the conventional rolling mill with pre-treated workpiece, it can be adapted for an inexpensive continuous process for industrial application[11].

According to Hall-Petch relationship, UFG materials exhibit higher yield strength with a large network of grain boundaries. The strength of UFG materials therefore depends upon the extent of grain refinement. Due to the intense plastic strain during cryorolling, traditional optical microscopy is not suitable to quantify the extent of refinement in UFG materials. Advanced electron microscopy methods such as scanning electron microscope (SEM), transmission electron microscope (TEM), and electron back scatter diffraction (EBSD) are suitable methods to evaluate UFG and nano structured materials. These techniques require tedious sample preparation and the inspection is confined to a tiny region of the macroscopic sample. An overall estimate of the degree of grain refinement imparted in the sample is challenging. Alternately, indirect estimates using transient mechanical tests such as stress relaxation can be used to verify the grain refinement in UFG materials. Moreover, macroscopic transient tests also help to understand the deformation mechanism in CG and UFG materials. It is essential to ensure uniform and consistent grain refinement when finalizing the process parameters of ultrafine grain refinement process. Using this method, it can be quickly estimated the average degree of grain refinement for a larger length scale. However, the present method does not focus on the local changes in the grain refinement and rather gives an average estimate, which is essential for practical applications.

1.1 Stress Relaxation and Related Phenomena

Stress relaxation is a reliable technique to investigate the flow behavior of metal, in particular, their time dependent deformation. In this test, deformation of the specimen during uniaxial tensile test is abruptly stopped without unloading. As shown in Fig.1, the applied stress drops continuously with time. Since kinetics of relaxation is governed by the deformation mechanism, this test can be used to understand the deformation mechanism and quantify the parameters like internal stress and activation volume[12].

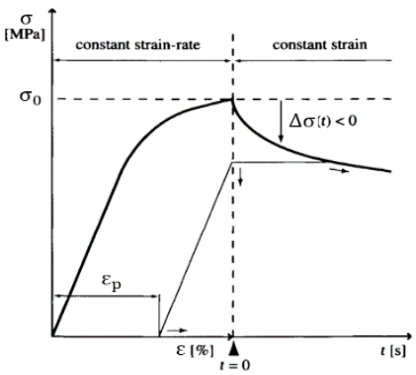

Fig.1. Schematic representation of stress relaxation test [13]

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The stress vs. time during relaxation can be modeled using a logarithmic law[13] given by

$$\Delta\sigma = A\, ln(1 + Bt) \quad (1)$$

where, $\Delta\sigma = \sigma_t - \sigma_0$, σ_t is the flow stress at time t and σ_0 is the stress at beginning of relaxation time (t=0), A and B are constants:

$$A = \frac{K\,T}{V^*},\ B = \frac{-E\,\varphi\,\rho_m\,b\,v_o V^*}{K\,T}\exp\left(-\frac{\Delta G - \sigma^* V^*}{K\,T}\right)$$

where K is the boltzmann constant, T is the absolute temperature (^{0}K), V^* is the apparent activation volume, E is the elastic modulus, φ is the geometric coefficient, ρ_m is the mobile dislocation density, b is the burgers vector, v_o is the dislocation velocity, ΔG is the activation energy barrier, σ^* is the effective stress component.
Equation (1) can be rewritten in the following form

$$\Delta\sigma = -\frac{K\,T}{V^*}\, ln(1 + \frac{t}{C_r}) \quad (2)$$

where V^* is the apparent activation volume and C_r is the time constant.Stress relaxation test has been successfully used to estimate the activation volume for several FCC (Al and Cu) and BCC (Fe) materials[14-16]. Activation volume represents the average volume of dislocations involved in the rate controlling deformation mechanism. For instance, activation volume of 10^2 to 10^3 b^3 is estimated for slip dominated mechanisms in FCC materials[14]. Conrad and Narayan[17] have studied the effect of grain size on flow stress in Zn and they distinguished three grain size regimes (depicted in Fig. 1 and 2 of [17]) which identify corresponding activation volume and deformation behavior. Regime I corresponds to grain size 1mm to 1μm which have apparent activation volume varying from 10^2 to 10^3 b^3. This is the regime of conventional deformation of polycrystalline metals[18]. Grain size ranging from 1μm to 10 nm is related to regime II and corresponding activation volumes are $20b^3$ (grain size= 10–100 nm), $70b^3$ (grain size= 180 nm) and $100–300b^3$ (grain size= 6–40 μm). Regime III corresponds to grain size below 10 nm. Lee et al.[19] found the activation volume of 10 b^3 for UFG Mn TRIP steel and indicated that plastic deformation behavior is mainly controlled by the dislocation bow out mechanism originating from grain boundary sources. Mohebbi et al.[15] obtained the apparent activation volume of UFG AA 1050 in the range of 50-70 b^3 suggesting grain boundary sliding contribution in relaxation. The ultrafine grained structure is known to induce additional deformation mechanisms such as grain boundary sliding or coble creep- diffusion controllable plastic deformation (especially for nanostructured materials) that can change the activation volume. Therefore, the stress relaxation test can serve as an indirect mechanical test that can quickly indicate the grain refinement. The objective of the present study is to produce the UFG materials through cryorolling and compare the activation volume of coarse grained and fine grained materials from stress relaxation experiment. AA 5083 was used in the present study which is a non-heat treatable Al-Mg alloy (5xxx series), known for its excellent corrosion resistance, capability to withstand extremely low temperature without brittleness and lower cost compared to 2xxx and 7xxx alloys[20].

1. EXPERIMENTAL PROCEDURE

The chemical composition (as determined by spark emission spectroscopy (ASTM E 1251-2011)) of 6.5 mm thick AA 5083 alloy from Hindalco India Ltd. is shown in Table 1.

Table 1: Chemical Composition of AA 5083 alloy (Wt %)

Mg	Mn	Cr	Cu	Fe	Si	Ti	Al
4.80	0.630	0.064	0.033	0.293	0.087	0.013	Balance

The samples were cut to dimensions of 100 mm x 200 mm and solution treated at 530^{o}C for 2 h in muffle furnace followed by water quenching (as per ASTM B918M). Cryorolling was performed by immersing the sample in liquid nitrogen bath for 30 minutes. For every successive pass, the material was dipped for sufficient time in liquid nitrogen to attain stable temperature. The conventional rolling mill in a four high configuration with the roll diameter of 142 mm was used to perform the cryorolling experiments. A constant rolling velocity 14 m/min was maintained during the process. The degree of reduction per pass (4-5%) was varied to minimize the temperature rise during rolling and to achieve a more uniform distribution of strain. Since high reduction in thickness is essential for achieving ultra-fine grain structure, samples were rolled up to ~ 85 % thickness reduction (i.e. from 6.5 mm to 1 mm thickness). Vicker hardness test was employed to measure the hardness post cryorolling. Hardness was measured along the rolling direction by applying a load of 10 kg for 15 s. All the samples were polished using 1000# grid emery paper before hardness measurement. Hardness measurement was repeated five times for each sample to ensure statistical significance. Both the solutionized (SL) and cryorolled (CYR) samples (prepared as per ASTM E8 standard) were tested in INSTRON 5582 machine. Stress relaxation tests were conducted at an initial strain rate of 5 x 10^{-3} s^{-1} with intermittent pausing of the uniaxial tensile test at a predefined load in the uniform elongation zone for a given interval of time. A constant relaxation time of 60 s was chosen for the present study. The predefined load can be chosen by 90 % of maximum load to get more decrease in the stress value. The experimental data was used to fit equation (1) by least square method and apparent activation volume was calculated from equation (2)

3. RESULTS AND DISCUSSION

3.1 Mechanical Properties

Engineering stress-strain curve of SL and CYR AA 5083 alloy is shown in Fig 2. The yield strength and ultimate tensile strength of CYR material are higher (460MPa and 509MPa respectively) compared to SL condition in which the values are 129MPa and 300MPa respectively. The strength increase is reflected in the hardness, with 161 Hv for CYR which is approximately 80 % higher than SL sample (89 Hv). The tensile properties and hardness of SL and CYR materials are shown in Table 2. During grain refinement, the dynamic recovery associated with climb and cross slip of dislocations is suppressed leading to high dislocation density. This reduces the mean free path of the dislocations. Therefore the strength of CYR is higher than that of SL, whereas the ductility and work hardening exponent (n) are less due to lower work hardening

rate and inability to accumulate dislocations during subsequent plastic deformation.

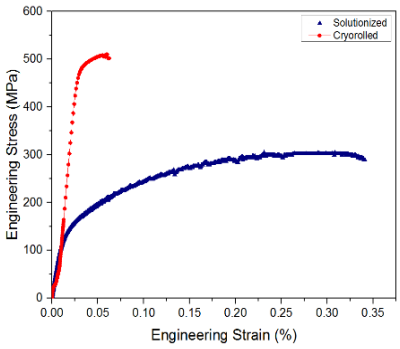

Fig.2. Engineering stress-strain curves of solutionized and cryorolled samples

Table 2: Tensile Properties of Solutionized and Cryorolled AA 5083 alloy

Material Conditions	Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Elongation (%)	Hardness (Hv)
Solutionized	129	300	30	89
Cryorolled	460	509	6.2	161

3.2 Stress Relaxation

Experimental stress relaxation curves of engineering stress vs. time for solutionized and cryorolled materials are shown in Fig.3 (a,b). Stress relaxation data is recorded and used to obtain constants (apparent activation volume and time constant) of the logarithmic model by curve fitting (Table 3). The activation volume in SL condition is 134 b^3 which is much larger than in CYR (64 b^3) material. It is also observed from the stress relaxation curves that the stress drop is higher in CYR material than in SL material due to higher tensile strength achieved by CYR material.

3.2.1 Grain Refinement

From Table 3, it can be seen that the activation volume in solutionized and cryorolled conditions corresponds to regime I and regime II[17] respectively. It mention earlier that the corresponding grain size ranges are 1µm-1000µm and 100-180nm respectively. Singh et al.[21], and Lee et al.[22] studied the microstructure of SL and CYR AA 5083 materials and found the grain size to be around 85 µm and 0.15 µm respectively. The activation volume decreases with increasing strain and decreasing grain size[16]. The low value of activation volume for UFG material is attributed to the fact that dislocation segment involved in the thermal activation is limited by grain size. From the above observation, it can be concluded that severe grain refinement has taken place during cryorolling in the present work leading to UFG structured. These results are also consistent with findings of Krishna et al.[23].

3.2.2 Deformation Mechanism

It is believed that the rate controlling mechanisms in the plastic deformation of coarse grained FCC materials are an intersection of forest dislocations and cross slip. However, it is not true for very fine or ultra-fine grained materials. According to Conrad and Narayan[17], dislocation glide and an intersection of dislocations within the grains is the suggested deformation mechanism corresponding to activation volume of 10^2 to 10^3 b^3 (Regime I). Activation volume of the present CYR material is far smaller than the SL material and also falls within the range of activation volume reported by Hayes et al.[24].

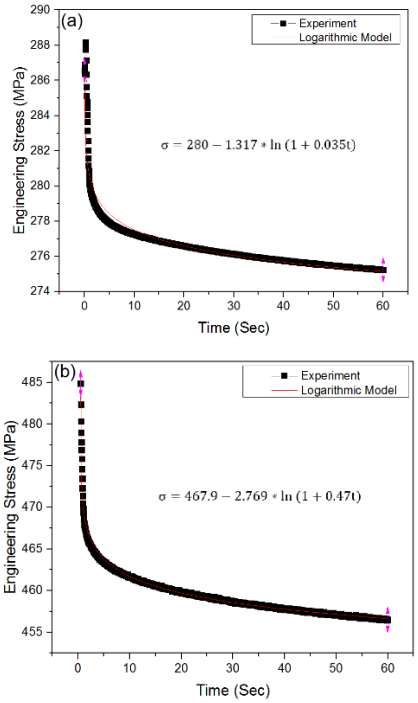

Fig. 3. Stress relaxation curves of AA 5083 alloy in (a) solutionized and (b) cryorolled conditions

Table 3: Constants of the Logarithmic Equation (1)

Processing Condition	Constants	
	Apparent Activation Volume (b^3)	Time Constant (Cr)
Solutionized	134	28
Cryorolled	64	2

It is necessary to investigate possible phenomena that occur for very fine grain size with low activation volume. Due to very small grain size and low dislocation density inside the grains in UFG materials, dislocation pile up at grain boundaries. Due to large amount of dislocations available at grain boundaries, diffusion occurs at very low strain rate during stress relaxation. Consequently, grain boundary sliding is a possible phenomenon that occurs in UFG materials. Kapoor et al. [25] also suggested the possibility of grain boundary deformation mechanism for UFG material at low activation energy and at low activation volume. The process involves the nucleation of dislocations at grain boundaries, glide through small grains and absorbed at the opposite grain boundaries. Moreover, dynamic recovery also possibly occurs during stress relaxation at low strain rate in UFG materials. High stored energy available in very fine grained materials act as driving force to promote the recovery in the dislocations during stress relaxation. It is also believed that dynamic recovery is more favorable than work hardening during relaxation due to continuously decreasing of plastic strain rate. So, it is concluded that it is possible to predict grain refinement and understand the possibilities of deformation mechanism by calculating the activation volume through simple stress relaxation experiments. However, it is required to compare the performance of precision components under different degree of grain refinement and evolve a standard for the input material quality for UFG based on their degree of grain refinement estimated from stress relaxation.

4. CONCLUSIONS

Cryorolling is a potential route to produce the ultrafine grained (UFG) materials. The mechanical properties were evaluated

through the monatomic tensile test of solutionized and cryorolled material. To understand the deformation mechanism of UFG materials, the activation volume was calculated from stress relaxation data. Following conclusions are made based on the present study.

1. The YS and UTS of cryorolled sample are approximately 256% and 70% greater than solutionized sample. Increase in the strength of cryorolled material attributed to suppression of dynamic recovery leading to higher dislocation density. However, the ductility of cryorolled material is low which is due to low work hardening rate and inability to accumulate dislocations during deformation.
2. The activation volume of cryorolled material is smaller than solutionized material. It shows that small amount of dislocations available in grain interior and most of the dislocations are close to the grain boundaries.
3. Dislocation glide and interaction of dislocations are the common mechanisms for large grain size (solutionized) material, while grain boundary sliding occurs in UFG materials due to small grain size and large grain boundary network.

References

[1]. Progress of Precision Engineering and Nano Technology. *Trans Tech Publications Ltd*, 2005.

[2]. J. Xu, J. Li, X. Zhu, G. Fan, D. Shan, and B. Guo, Microstructural Evolution at Micro / Meso-Scale in an Ultrafine-Grained Pure Aluminum Processed by Equal-Channel Angular Pressing with Subsequent Annealing Treatment, **8** (2015) 7447–7460.

[3]. J. Xu, X. Zhu, D. Shan, B. Guo, and T. G. Langdon, Effect of grain size and specimen dimensions on micro-forming of high purity aluminum, *Material Science and Engineeirng A*, **646** (2015) 207–217.

[4]. M. S. Mohebbi, A. Akbarzadeh, B. H. Kim, and S. K. Kim, Analysis of Strain Rate Sensitivity of Ultrafine-Grained AA1050 by Stress Relaxation Test, *Metallurgical and Material Transaction*, **45** (2014) 5442–5450.

[5]. R. Z. Valiev and T. G. Langdon, Principles of equal-channel angular pressing as a processing tool for grain refinement, *Progresses in Material Science*, **51** (2006) 881–981.

[6]. K. Edalati and Z. Horita, A review on high-pressure torsion (HPT) from 1935 to 1988, *Material Science and Engineeirng A*, 652 (2016) 325–352.

[7]. M. Reza Toroghinejad, F. Ashrafizadeh, and R. Jamaati, On the use of accumulative roll bonding process to develop nanostructured aluminum alloy 5083, *Material Science and Engineeirng A*, **561** (2013) 145–151.

[8]. S. K. Panigrahi, R. Jayaganthan, and V. Pancholi, Effect of plastic deformation conditions on microstructural characteristics and mechanical properties of Al 6063 alloy, *Materials and Design*, **30** (2009),1894–1901.

[9]. A.K. Gupta, T.S. Maddukuri, and S.K. Singh, Constrained groove pressing for sheet metal processing *Progress in Materials Science*, **84** (2016) 403–462.

[10]. D. R. Satish, F. Feyissa, and D. R. Kumar, "Cryorolling and Warm Forming of AA6061 Aluminum Alloy Sheets," *Materials and Manufacturing Processes*, 32 (2017) 1-8

[11]. H. L. Yu, C. Lu, A. K. Tieu, H. J. Li, A. Godbole, and S. H. Zhang, Special rolling techniques for improvement of mechanical properties of ultrafine-grained metal sheets: A review, *Advanced. Engineernig Materials*, 18 (2016) 754–769.

[12]. V. I. Dotsenko, Stress Relaxation in Crystals, *Physica Status Solidi*, 93 (1979) 11–43.

[13]. Thermally Activated Mechanisms in Crystal Plasticity, *Pergamon materials series; 8*, 2003

[14]. L. Xiao and J. Bai, Stress relaxation properties and microscopic deformation structure of H68 and QSn6.5–0.1 copper alloys at 353 K, *Material Science and Engineeirng A*, **244** (1998) 250–256.

[15]. M. S. Mohebbi, A. Akbarzadeh, Y. O. Yoon, and S. K. Kim, Stress relaxation and flow behavior of ultrafine grained AA 1050, *Mechanics of Materials*, **89** (2015) 23–34.

[16]. Q. Wei, S. Cheng, K. T. Ramesh, and E. Ma, Effect of nanocrystalline and ultrafine grain sizes on the strain rate sensitivity and activation volume: Fcc versus bcc metals, *Material Science and Engineeirng A*, **381** (2004) 71–79.

[17]. H. Conrad and J. Narayan, Mechanisms for grain size hardening and softening in Zn, *Acta Materiallia*, 50 (2002) 5067–5078.

[18]. S. Cheng, J. A. Spencer, and W. W. Milligan, Strength and tension/compression asymmetry in nanostructured and ultrafine-grain metals, *Acta Materiallia*, **51** (2003) 4505–4518.

[19]. S. Lee, S. J. Lee, and B. C. De Cooman, Work hardening behavior of ultrafine-grained Mn transformation-induced plasticity steel, *Acta Materiallia*, **59** (2011) 7546–7553.

[20]. X.-Y. Liu and J. B. Adams, Grain-boundary segregation in Al–10%Mg alloys at hot working temperatures, *Acta Materiallia*, **46** (1998) 3467–3476.

[21]. D. Singh, P. Nageswara Rao, and R. Jayaganthan, Microstructures and impact toughness behavior of Al 5083 alloy processed by cryorolling and afterwards annealing, *International Journal of Minerals Metallurgical and Materials*, **20** *(*2013) 759-769.

[22]. Y. B. Lee, D. H. Shin, K.-T. Park, and W. J. Nam, Effect of annealing temperature on microstructures and mechanical properties of a 5083 Al alloy deformed at cryogenic temperature, *Scripta Materiallia*, **51** (2004) 355–359.

[23]. K. S. V. B. R. Krishna, K. Chandra Sekhar, R. Tejas, N. Naga Krishna, K. Sivaprasad, R. Narayanasamy, and K. Venkateswarlu, Effect of cryorolling on the mechanical properties of AA5083 alloy and the Portevin–Le Chatelier phenomenon, *Materials and Design*, **67** (2015) 107–117.

[24]. R. W. Hayes, D. Witkin, F. Zhou, and E. J. Lavernia, Deformation and activation volumes of cryomilled ultrafine-grained aluminum, *Acta Materiallia*, **52** (2004) 4259–4271.

[25]. R. Kapoor and J. K. Chakravartty, Deformation behavior of an ultrafine-grained Al-Mg alloy produced by equal-channel angular pressing, *Acta Materiallia*, **55** (2007) 5408–5418.