TOUGHNESS BEHAVIOUR OF CRYO-ECAP ALUMINUM 6063 BY CHARPY IMPACT TEST

By

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ABSTRACT

The severe plastic deformation technique that has attracted the material community in present days is Equal Channel Angular Pressing (ECAP). Ultra fine-grained microstructures can be produced by this technique without a significant change in geometry. The present work describes the study of cryo treatment effect post ECAP aging on impact strength of aluminium 6063 alloy using Charpy impact test. A channel having an angle of 108° is used for conducting ECAP process. Samples having dimensions of 100 mm X 9.5 mm X 9.5 mm were chosen and these samples solution were treated at 520 °C for 120 minutes. These samples were processed into two methods by dividing these solution treated samples into two groups. One set of samples has been dipped in liquid nitrogen for 20 minutes before the ECAP process has been carried out, obtaining Cryo-ECAP samples. The second set of solution-treated samples has been directly subjected to ECAP process, producing RT ECAP samples. The obtained samples dimensions were made suitable for Micro Vickers and Charpy Impact test and were given aging treatment at 180 °C for different timings. These two sets of samples are subjected to Micro Vickers hardness tests and Impact tests. The obtained results are correlated with macro and micro examinations.

Keywords: Charpy Impact Test, Aluminum 6063, Cryo-ECAP, Vickers Hardness, Impact Test.

INTRODUCTION

Severe plastic deformation reduces grain size in metallic alloys without changing dimensions (Hayoune, 2012; Valiev & Langdon, 2006; Varadala et al., 2018a). Equal Channel Angular Pressing (ECAP) is a process of pressing billet sample through closed angular channel, whose dimensions are same as billet specimen dimensions. For this purpose, a die and a punch are required, made of high carbon high chromium steel and heat treated for good strength. After closing this die, specimen is placed in the angular channel and pressed with the help of punch using UTM. The applied pressure on the punch causes the specimen to travel through the angular channel and comes outside from the lower side of the die. The specimen travels through this angular channel of the die without any dimensional change, so this process is called Equal Channel Angular Pressing. After this process, the specimen dimension remains unchanged, but the grains in the material gets deformed severely to nano-size. The reduction in grain size of the metals improves various mechanical properties like hardness, toughness, fatigue life etc.

Alloy 6063 is a commercial aluminum alloy, which can be hardened by aging heat treatment. Post ECAP aging influence on properties like microstructure, hardness and electrical conductivity were studied by Cerri and Leo (2005). Mechanical properties were highly improved according by pressing the billets using the dies having channel angle of 120° (Varadala et al., 2018b, 2019). This is done for aluminum 5083 with and without copper casing subjected to ECAP at room temperature.

Panigrahi et al. (2009) compared the mechanical properties for rolled Al 6063 alloys at cryogenic temperature and room temperature. Improvement in strength, ductility in severely deformed Al 6063 alloy can be observed by precipitation hardening and grain coarsening during aging. Grain dynamic recovery suppression happens in cryogenic treatment of samples before the ECAP process. This phenomena causes strength, ductility, and strain (Baldissera & Delprete, 2008; Bouzada et al., 2012; Kalia, 2010). Dynamic recovery suppression is likely due to preserving the high density defects and produces fine grain structures along with potential recrystallization sites. Cryogenic rolling followed by aging or annealing treatment shows improvement in the mechanical properties of some aluminum alloys like AA2219, and Aa6061 (Arnuri & Gurugubelli, 2020; Chen et al., 2013; Kalia, 2010; Shanmugasundaram et al., 2006). According to Cubides et al. (2020) "During ECAP, dynamic recrystallization (DRX) and strain-induced dynamic precipitation (SIDP) simultaneously occurs, resulting in a bimodal grain structure of original elongated coarse grains and newly formed equiaxed fine grains with a large volume fraction of β-$Mg_{17}Al_{12}$ precipitates".

The purpose of this study is to find T8 (Solution treatment + Cold work + Aging) heat treatment on toughness of Aluminum 6063, where cold work is carried out by CRYO and RT ECAP processes. Resistance towards a sudden load or impact can be determined by Charpy impact test. These prepared materials are tested to know the toughness behaviour by Charpy Impact test and the effect of aging phenomena on Cryogenic ECAP and RT ECAP is studied. The samples is subjected to macro and micro examination and results were analyzed.

1. Materials and Method

The step-by-step procedure of the present work is shown in Figure 1. The Aluminum 6063 alloy is solution treated, quenched in water and placed in liquid nitrogen for 20 minutes and subjected to ECAP process. After ECAP process, aging heat treatment is given for different timings. In order to compare the results, another set of samples are subjected to ECAP without dipping in liquid nitrogen and followed by aging heat treatment. These two sets of samples were tested along with macro and micro examinations.

1.1 Material

Aluminum 6063 samples having cross sectional dimensions of 9.5 mm X 9.5 mm were chosen for present study. Initially, the material has been tested by spectrometer and the obtained chemical composition is shown in Table 1.

The prepared samples were cut into required dimensions, i.e., 9.5 mm X 9.5 mm X 100 mm as they are shown in Figure 2. This Figure 2 also shows the difference in appearance of the surfaces between liquid nitrogen immersed samples and not immersed samples.

1.2 Solution Treatment

Initially, all the samples were solution treated at 520 °C for 120 minutes. The samples were immediately quenched in water to get super saturated solid solution (SSSS). These samples were divided into two groups after this solution treatment.

1.3 CRYO ECAP and RT ECAP

In this experiment, the Equal Channel Angular Pressing (ECAP) die has a 130 mm channel length, 9.5 mm X 9.5 mm cross sectional area, and an angle (Φ) of 108° at the channel intersection and an outer curvature angle (Ψ) of 36° which is shown in Figure 3.

First group of samples, were named as CRYO ECAPed (Cryogenic Equal Channel Angular Pressed) samples, because these samples were dipped in liquid nitrogen (temperature –196 °C) for 20 minutes, before each ECAP operation. This process continued for up to four passes. Second group of samples was named as RT ECAPed (Room Temperature Equal Channel Angular Pressed) samples, because these samples were directly sent to ECAP operation at room temperature after solution treatment. For these group samples also, the process continues up to four passes.

In order to perform the ECAP operation, computer assisted universal testing machine (UTM) has been used for both groups of samples. This ECAP process using UTM can be seen in Figure 4. Molybdenum disulfide (MoS_2) has been used as

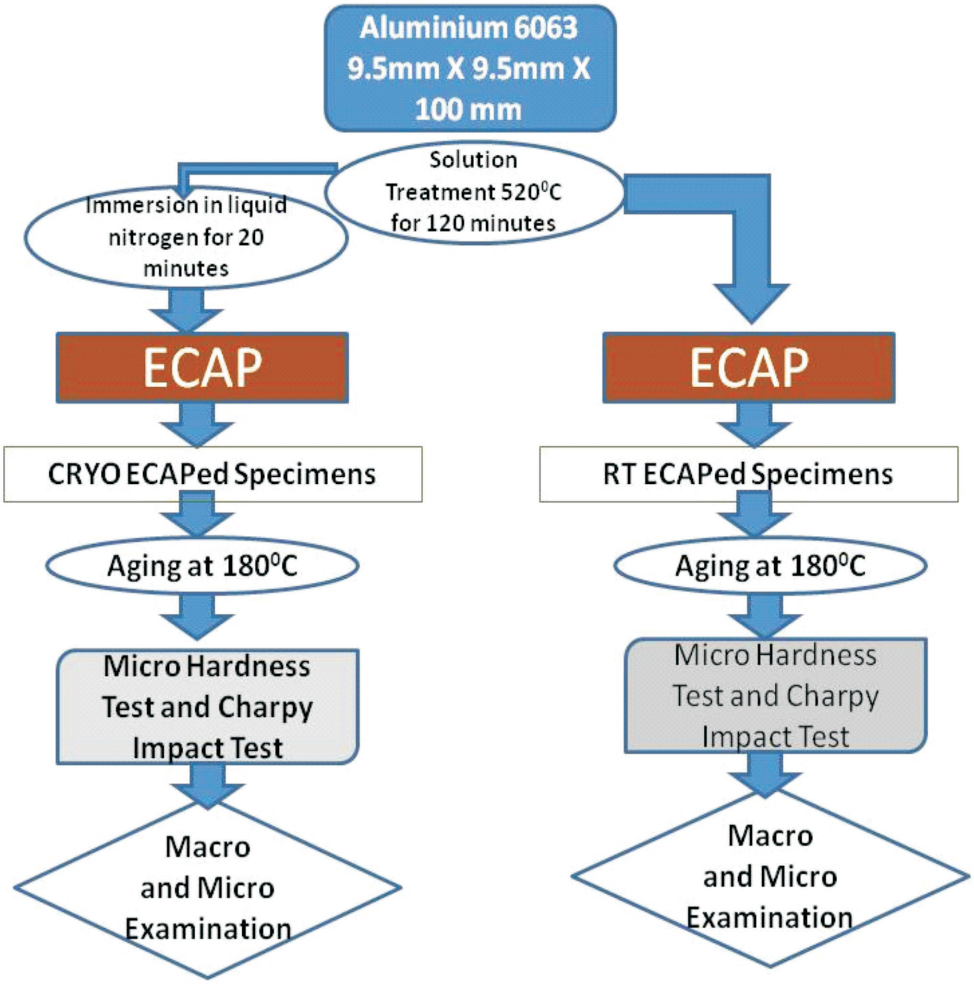


Figure 1. Process Diagram

Mg	Si	Fe	Cu	Zn	Mn	P	Al
0.63 %	0.43 %	0.15 %	0.049 %	0.03 %	0.054 %	0.005 %	98.6 %

Table 1. Composition of Aluminum 6063

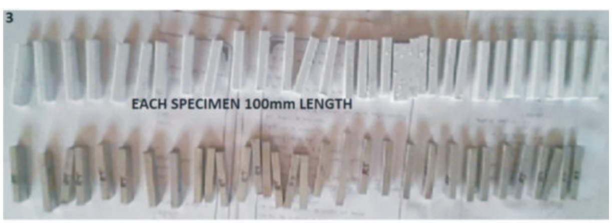


Figure 2. Specimens used for ECAP

Figure 3. ECAP Die Dimensions

lubricant for easy passage of billet specimen through the channel. The samples had a 1:1 aspect ratio after the ECAP process for both group of samples. All the samples were processed in route A (the exited sample placed into the die channel again without any rotation as it is) for four passes. Figure 5 shows the cryogenic treatment of solution treated samples before ECAP process. The deformed billet after ECAP process can be seen in Figure 6. While conducting

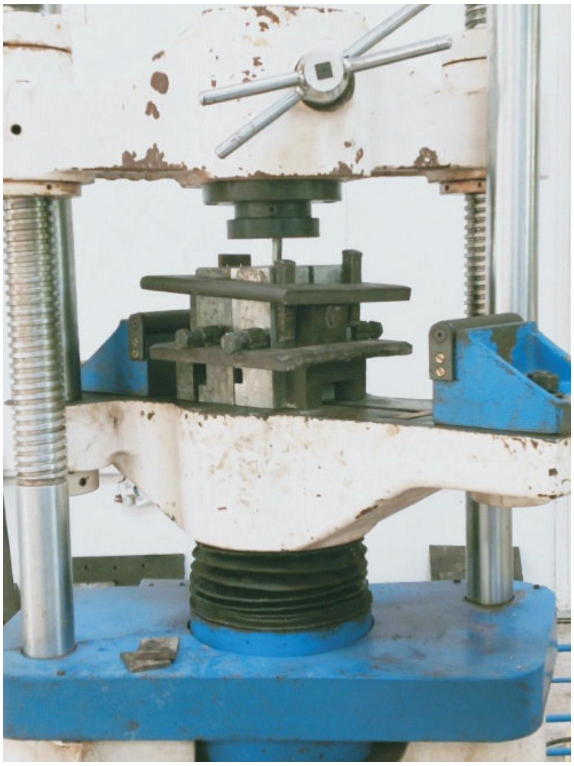


Figure 4. ECAP Process using UTM

Figure 5. Cryogenic Treatment of Solution Treated Samples before ECAP

ECAP operation, sample passing through the angular channel, can be seen in Figure 6. It is observed that more pressing force has been required for the pressing of CRYO ECAP samples in the UTM operation and shiny surfaces than the RT ECAP samples are obtained.

1.4 Aging

Two groups of samples, CRYO ECAPed and RT ECAPed processed samples were subjected to aging at the temperature of 180 °C, before going to testing. These two

Figure 6. Deformed Specimen in Die after ECAP

sets of samples were aged with 15 minutes of increment timings, till 500 minutes at 180 °C. These T8 heat treated (Solution treated + Cold worked (ECAP) + Aging) samples were subjected to Micro Vickers Hardness test and Charpy Impact Test.

1.5 Micro Vickers Hardness test and Charpy Impact Test

Micro Vickers Hardness tester has been used for Hardness measurements and the inputs were: applied load - 300 grams and dwell time - 10 seconds. For obtaining one Micro Vickers Hardness point at each aging point, 10 readings were taken and the average value has been calculated. This process is repeated for both groups of samples. For comparison purpose, undeformed conventional alloy of Aluminum 6063 has been taken for same heat treatment process and aging at 180^{0}C has been done and at different time intervals Micro Vickers Hardness values were taken. By using these values, age hardening curves were drawn.

To limit the difficulty of the process and to cover all the regions of under aging, peak aging and over aging, certain points were chosen on these age hardening curves obtained by Micro Vickers Hardness test. These aging time points were selected for both Cryo-ECAPed and RT ECAPed aging curves. The samples obtained from ECAP process were subjected to aging for these selected timings to conduct Charpy Imapct test. They were cut carefully into dimensions of 9.5 mm x 9.5 mm x 55 mm and made notch at exact dimension according to ASTM specifications for Charpy Impact test. At each aging point five specimens were chosen for obtaining average value. By using conventional Charpy Impact testing machine, Charpy Impact tests were conducted and the impact testing machine used for testing can be seen Figure 7.

In Charpy impact test, the specimen is in cantilever position (Horizontally). The sample is placed in such a way that, the notch face and hammer direction are in the same direction as shown in Figure 8. Figure 9 shows some V-notched RT ECAPed specimens followed by aging at different time intervals. The pointer is set up to its maximum value before releasing the hammer from the initial height towards the sample. The values of toughness energy observed are tabulated. The same process was repeated for all the other samples in the same manner. For obtaining one Impact test value at each aging point, five specimens

Figure 7. Impact Test Machine

Figure 8. Specimen Placed on Parallel Jaws

Figure 9. V-notched Charpy Impact Test Samples (ECAPed)

were tested and average value of toughness has been calculated.

1.6 Macro and Micro Examinations

The specimens were subjected to macro and micro examinations after the Charpy Impact test. For micro examination, the samples were subjected to Scanning Electron Microscope. SEM Examination requires sample preparation and proper care had been taken while cutting and metallographic preparation of the samples has been done to reveal the features of Cryogenic ECAPed and RT ECAPed samples followed by aging. In order to correlate the results obtained by Micro Vickers and Charpy Impact test results, all these examinations were discussed.

2. Results and Discussions

ECAP process causes grain refinement and strain hardening, thereby improves the strength of the material. The dislocation tangles formed by the dislocation density, increase during the process of severe plastic deformation. This results in further strengthening of the alloy. Behaviour of the alloy after age hardening and Charpy Impact Test after four passes of ECAP at Cryogenic (CRYO) and Room Temperature (RT) are evaluated. Micro Vickers Hardness and Charpy Impact Test results of this alloy are correlated with the microstructures obtained by SEM.

2.1 Age Hardening

The hardness curves for undeformed, room temperature ECAPed and cryogenic ECAPed alloys are shown in Figure 10. These curves are drawn for T8 heat treated (solution treating + cold working (here ECAP) + Aging) Aluminum 6063 alloy. The peak hardness at 90 minutes of aging has been shown by the samples which were solution treated and cryogenic and room temperature ECAPed.

The peak hardness value has been 116 HV for the Cryo-ECAP age hardening curve and for the RT ECAP age hardening curve it has been 95 HV. The formation of ultra-

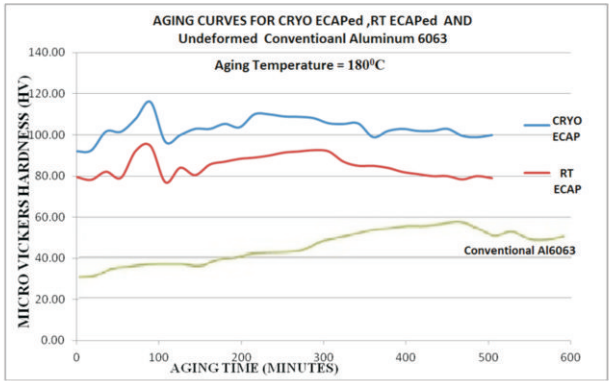


Figure 10. Age Hardening Curves for CRYO ECAP, RT ECAP Al6063 Samples at 180 °C

fine grain structures during ECAP resulted in these higher hardness values for cryogenic ECAPed alloy.

2.2 Charpy Impact Test

The toughness behaviour of the material is determined by the amount of energy absorbed during the impact test. Toughness and impact strength are higher, if the amount of energy absorbed is higher. In this present study, it has been proposed that the enhancement in properties of Aluminum 6063 can be seen by cryogenic treatment before ECAP process. Both CRYO ECAPed and RT ECAPed specimens were given aging treatment with an incremental time of 30 minutes and then tested by Charpy Impact Testing equipment. The results obtained were tabulated in Table 2. The graphs obtained from these values can be seen in Figure 11.

Throughout all the aging timings, Cryo-ECAPed specimens showed more toughness strength values than RT ECAPed. More brittle sound has been heard during breaking of Cryo-ECAPed specimens.

2.3 Macro Examination

A more brittle nature of Cryo-ECAPed samples than RT ECAPed samples at every aging time has been seen by macroscopic examination of impact tested specimens. RT

Charpy Impact Test Values (Joules)										
Aging Time (Minutes)	0	30	60	90	120	150	180	240	360	480
CRYO ECAP	128	142	148	174	182	150	152	158	120	108
RT ECAP	120	136	132	168	150	125	120	124	110	90

Table 2. Charpy Impact Test Values

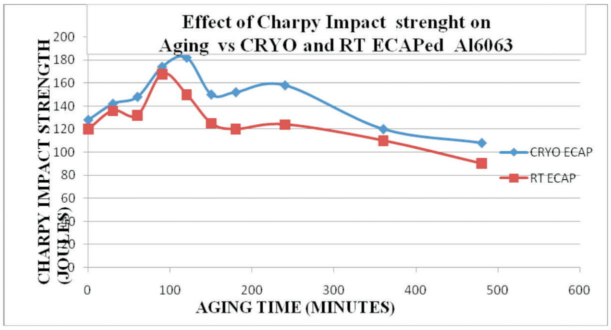
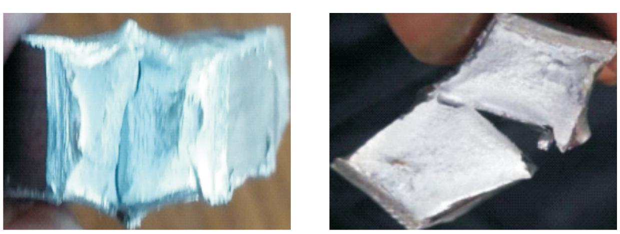


Figure 11. Effect of Charpy Test Strength for CRYO and RT ECAPed Al6063 vs. Aging Time (180 °C)

ECAP samples aged for 0 minutes, 60 minutes, 120 minutes and 150 minutes are shown in Figure 12(a), Figure 13(a), Figure 14(a) and Figure 15(a). More brittleness can be seen at 120 minutes, which is nearer to peak aging time compared to other aging times. Cryo-ECAP samples aged for 0 minutes, 60 minutes, 120 minutes and 150 minutes are shown in Figure 12(b), Figure 13(b), Figure 14(b) and Figure 15(b). At 120 minutes, the Charpy Impact test value showed the peak value with a more brittle and shiny surface than all other samples. While breaking, more flat surfaces were observed for Cryo-ECAP samples, whereas cup-like fractured surfaces were observed for RT ECAP samples. Sound also differs at the time of impact for Cryo-

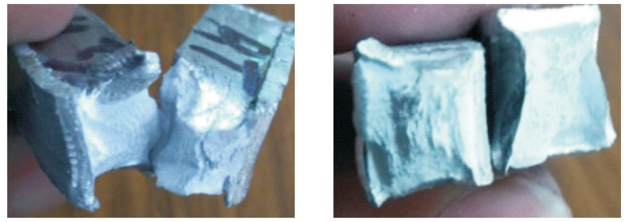
(a) (b)

Figure 12. 0 Minutes Aging (a) RT (b) Cryo



(a) (b)

Figure 13. 60 Minutes Aging (a) RT (b) Cryo



(a) (b)

Figure 14. 120 Minutes Aging (a) RT (b) Cryo

(a) (b)

Figure 15. 150 Minutes Aging (a) RT (b) Cryo

ECAPed and RT ECAPed samples, where more brittle sound can be for Cryo-ECAPed specimens than RT ECAPed specimens.

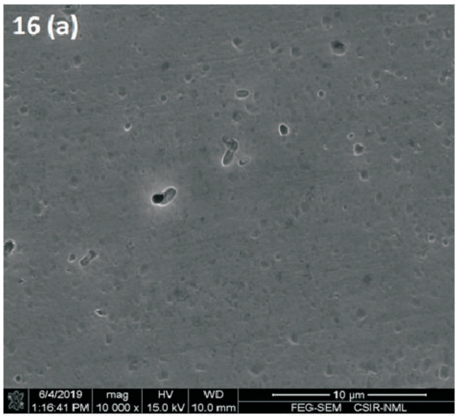
2.4 SEM Analysis of the Alloy during Age Hardening

To perform structural analysis on the precipitates formed in Cryo-ECAPed and RT-ECAPed Al6063 specimens who were aged at 180^{0}C, the Scanning Electron Microscope (SEM) is used. At 0 minutes, 90 minutes, and 360 minutes of aging time for both types, the SEM images were obtained.

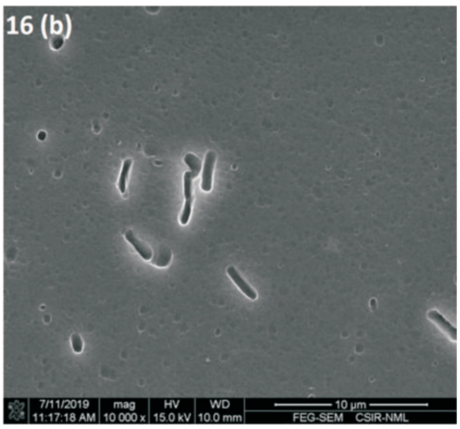
SEM images for 0 minutes of aging for RT and Cryo-ECAPed specimens are shown in Figure 16(a) and Figure 16(b). Dynamic precipitation takes place during ECAP process of Cryo-ECAPed samples, which results in more precipitation in Cryo-ECAPed specimens.

SEM images for 90 minutes of aging for RT and Cryo-ECAPed specimens are shown in Figure 17(a) and Figure 17(b). During the process of aging, precipitates are formed and the coherency of precipitates is more here. This is in agreement with the higher hardness values and Charpy impact strength values obtained for the Cryo-ECAPed alloy than RT ECAPed specimens.

SEM images of 360 minutes of aging for RT and Cryo-ECAPed specimens are shown in Figure 18(a) and Figure 18(b). More destruction has been observed in the regions where precipitates are present. The precipitates were grown and their coherency is also deviated in both the

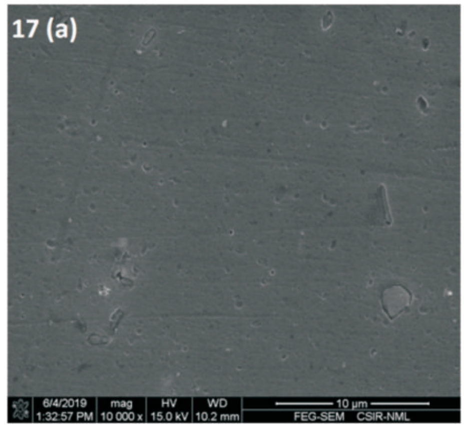


(a)

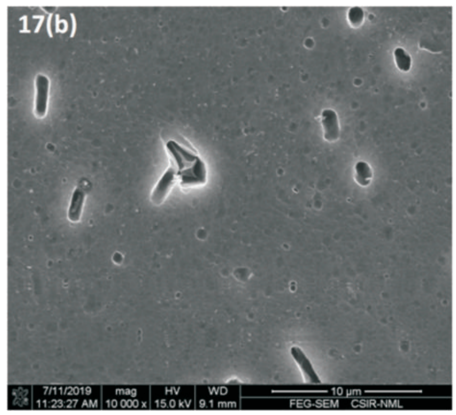


(b)

Figure 16. SEM Images of Al6063 at 0 Minutes of Aging (a) RT ECAPed Alloy (b) Cryo-ECAPed Alloy



(a)

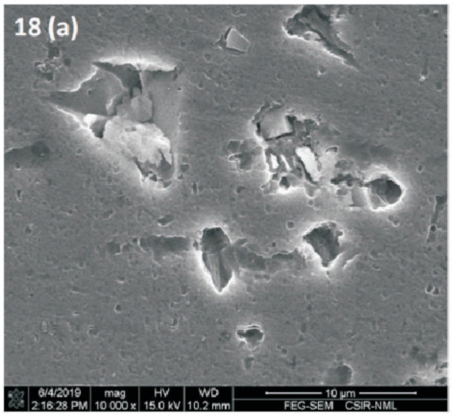


(b)

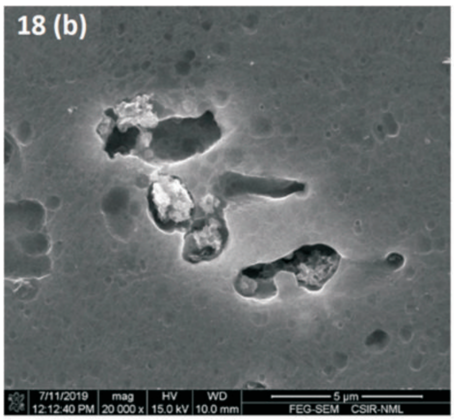
Figure 17. SEM Images of Al6063 at 90 Minutes of Aging (a) RT ECAPed Alloy (b) Cryo-ECAPed Alloy

cases and the deviation is more for the RT ECAPed alloy due to over aging. Further, after 360 minutes of aging, a tremendous growth of the precipitates in RT-ECAPed alloy has been observed and hence the hardness is not maintained consistent.

Two micro structural phenomena including precipitation of Mg_2Si and restoration is caused by the Post ECAP aging. Precipitates formed will pin dislocations during the aging process. The glide motion of dislocations is obstructed by this pin dislocation. Recovery and recrystallization will take place due to restoration of dislocations, which were introduced during the ECAP process. Dislocation free grains nucleation and growth occurs during recrystallization process within the deformed structure. The T8 (Solution treated, ECAPed and followed by aging) Micro Vickers hardness of the ECAPed alloy is more compared to that of the undeformed conventional alloy due to this reason. The peak hardness occurs much earlier than that of the conventional alloy, because of the two reasons, and they are, i) increase in dislocation density and ii) enhancement of the diffusion rate of Mg_2Si through dislocation pipes. Finely dispersed phases like precipitates of β'' and β' in the microstructure are formed by the dislocation sites which act as nucleation sites. At 90 minutes, peak aging values observed for both Cryo-ECAPed and RT ECAPed specimens due to this phenomenon. The hardness and Charpy Impact strength of Cryo-ECAPed alloy were observed as 120 HV and 174 Joules respectively. The hardness and Charpy Impact strength of RT ECAPed alloy were observed as 112 HV and 168 Joules respectively.



(a)



(b)

Figure 18. SEM Images of Al6063 at 360 min of Aging (a) RT ECAPed Alloy (b) Cryo-ECAPed Alloy

Conclusion

The effect of T8 heat treatment (Solution treatment + ECAP + Aging) on Vickers Micro Hardness and Charpy Impact test along with microstructure of 6063 Aluminum alloy is studied and the Charpy Impact strength is correlated with microstructures. The given conclusions are drawn from the present experimental investigations.

- Significant reduction in peak hardening time has been observed for both Cryo-ECAPed and RT ECAPed alloys over the conventional alloy of Aluminum 6063. Both Cryo-ECAPed and RT ECAPed samples have obtained peak hardness at 90 minutes, where for the commercial alloy, it has been 480 minutes.
- The higher peak hardness measurement of 116 HV has been obtained for Cryo-ECAPed Al 6063 due to more dynamic precipitation obtained in Cryo-ECAPed alloy than RT ECAPed Al 6063, which has 95 HV.
- The toughness value obtained by Charpy Impact test is peak at 182 J for Cryo-ECAPed alloy and for RT ECAPed alloy it is 168 J. Due to Cryogenic treatment 120 minutes has been taken for getting peak aging point, whereas it is only 90 minutes for RT ECAPed alloy. The higher toughness of the CRYO ECAPed alloy is due to its more refined grain structure.
- Excellent agreement between the Micro Vickers Hardness, Charpy Impact test behaviour and microstructures have been observed from the present investigations.

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References

[1]. Arnuri, S., & Gurugubelli, S. N. (2020). NDT evaluation of age hardening kinetics of RT and Cryo–ECAPed Aluminum 6063 alloy from attenuation coefficient and longitudinal velocity measurements obtained by ultrasonic testing. *Materials Today: Proceedings.* https://doi.org/10.1016/j.matpr.2020.05.706

[2]. Baldissera, P., & Delprete, C. (2008). Deep cryogenic treatment: A bibliographic review. *The Open Mechanical Engineering Journal,* 2(1), 1-11. https://doi.org/10.2174/1874155X00802010001

[3]. Bouzada, F., Cabeza, M., Merino, P., & Trillo, S. (2012). Effect of deep cryogenic treatment on the microstructure of an aerospace aluminum alloy. In *Advanced Materials Research* (Vol. 445, pp. 965-970). Trans Tech Publications Ltd. https://doi.org/10.4028/www.scientific.net/AMR.445.965

[4]. Cerri, E., & Leo, P. (2005). Influence of severe plastic deformation on aging of Al–Mg–Si alloys. *Materials Science and Engineering: A,* 410, 226-229. https://doi.

org/10.1016/j.msea.2005.08.135

[5]. Chen, Y., Li, Y., He, L., Lu, C., Ding, H., & Li, Q. (2008). The influence of cryoECAP on microstructure and property of commercial pure aluminum. *Materials Letters,* 62(17-18), 2821-2824. https://doi.org/10.1016/j.matlet.2008.01.091

[6]. Cubides, Y., Zhao, D., Nash, L., Yadav, D., Xie, K., Karaman, I., & Castaneda, H. (2020). Effects of dynamic recrystallization and strain-induced dynamic precipitation on the corrosion behavior of partially recrystallized Mg–9Al–1Zn alloys. *Journal of Magnesium and Alloys,* 8(4), 1016-1037. https://doi.org/10.1016/j.jma.2020.09.005

[7]. Hayoune, A. (2012). Thermal analysis of the impact of RT storage time on the strengthening of an Al-Mg-Si alloy. *Materials Sciences and Applications,* 3(7), 460-466. https://doi.org/10.4236/msa.2012.37065

[8]. Kalia, S. (2010). Cryogenic processing: A study of materials at low temperatures. *Journal of Low Temperature Physics,* 158(5), 934-945. https://doi.org/10.1007/s10909-009-0058-x

[9]. Panigrahi, S. K., Jayaganthan, R., & Pancholi, V. (2009). Effect of plastic deformation conditions on microstructural characteristics and mechanical properties of Al 6063 alloy. *Materials & Design,* 30(6), 1894-1901. https://doi.org/10.1016/j.matdes.2008.09.022

[10]. Shanmugasundaram, T., Murty, B. S., & Sarma, V. S. (2006). Development of ultrafine grained high strength Al–Cu alloy by cryorolling. *Scripta Materialia,* 54(12), 2013-2017. https://doi.org/10.1016/j.scriptamat.2006.03.012

[11]. Valiev, R. Z., & Langdon, T. G. (2006). Principles of equal-channel angular pressing as a processing tool for grain refinement. *Progress in Materials Science,* 51(7), 881-981. https://doi.org/10.1016/j.pmatsci.2006.02.003

[12]. Varadala, A. B., Gurugubelli, S. N., & Bandaru, S. (2018). Equal channel angular extrusion of semicircular AA 5083 covered with copper casing. *Emerging Materials Research,* 7(3), 160-163. https://doi.org/10.1680/jemmr.18.00026

[13]. Varadala, A. B., Gurugubelli, S. N., & Bandaru, S. (2018). Equal-channel angular extrusion of Al 5083 alloy with copper shielding. *Emerging Materials Research,* 7(4), 227-232. https://doi.org/10.1680/jemmr.18.00043

[14]. Varadala, A. B., Gurugubelli, S. N., & Bandaru, S. (2019). Enhancement of structural and mechanical behavior of Al-Mg alloy processed by ECAE. *Materials Today: Proceedings,* 18, 2147-2151. https://doi.org/10.1016/j.matpr.2019.06.654

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