International Refereed Journal of Engineering and Science (IRJES)
ISSN (Online) 2319-183X, (Print) 2319-1821
Volume 6, Issue 7 (July 2017), PP.59-65

Annealing Response of Aluminum Alloy AA6014 Processed By Severe Plastic Deformation

*Sanjeev Sharma[1], M.Sheik Hassan[2], Brejesh Kumar[3]
[1]*Department of Mechanical Engineering Amity University Gurgaon Haryana, India,*
[2]*Department of Mechanical Engineering Amity University Gurgaon Haryana, India,*
[3]*Center of Nano Technology Amity University Gurgaon Haryana, India,*
*Corresponding author: *Sanjeev Sharma*

Abstract: In this paper the study of micro structural stability during annealing with respect to time of conventionally grains (CG) and ultrafine-grained (UFG) of Aluminum AA6014 i s carried out. It has been observed that, the effect of the second phase magnesium-silicon particles in the CG and UFG AA6014 samples leads to a rapid hardness which increases from 40HV10 to 70HV10 within 7 days. Artificial aging shows that the material hardness even increased after 20 hours of annealing at 180°C. In total 30 hours of annealing, the hardness arrives at its maximum and then reduces due to the formation of Mg2Si precipitates, which rise in size and change their coherency. The precipitates cannot efficiently pin the dislocations and act as barriers to the dislocation motion which indicate an overall decrease in the hardness. It also has been found that the ultrafine-grained AA6014 alloy loses its thermal stability at approximately 200°C and recrystallized at 300°C. Thermal stability is strongly dependent on the material purity, second phase particles and/or oxide particles which may break up during rolling and lead to some dispersion strengthening.

Keywords: Annealing, Ultra-Fine Grained (UFG), Nano- Structured Material, Severe Plastic Deformation, Accumulative

I. INTRODUCTION

Severe Plastic Deformation (SPD) techniques [1] such as Equal Channel Angular Pressing (ECAP) [2], High Pressure Torsion (HTP) [3], Cyclic Extrusion Compression (CEC) [4], Repetitive Corrugation and Straightening of sheet metals [5] Continuous Confined Strip Shearing and Mechanical Milling process [6] are have s o m e drawbacks. Firstly, the above pointed out processes are found to be improper to produce bulk materials. Secondly, they required forming with dies, which are quite expensive and also required very high forces. As compared to the above processes, the ARB has no such inadequacies. In order to produce ultrafine grained and/or nano-structured bulk aluminum sheets, the ARB process was at first suggested by Saito et al. [7] then it was modified successfully by Tsuji et al. [8] by producing UFG bulk sheet of interstitial free (IF) steel. The four fundamental controlling factors of the ARB process were annealed temperature, inlet temperature, rolling speed and the percentage of reduction. Among the above four factors, annealed temperature was found to be the most important factor influencing accumulative roll bonding. Saito et al. [9] have carried out the same research with AA5083, IF steel and AA1100.

Table i. Chemical Composition Aluminium Alloys Aa6014 % By Weight

Si	Cu	Fe	Mn	Mg	Cr	Zn	Ti	Others	Al
1.5	0.52	0.5	0.2	0.6	0.1	0.2	0.15	0.15	Bal-ance

From the material selection point of view Aluminium alloy AA6014 derives its strength primarily from the precipitation strengthening mechanism of the second phase particles Mg2Si. Another advantage of this allow is hat it does not build any strain marks upon sheet metal forming and it is for this reason usually used for the outer body automobile panels. The chemical composition aluminium alloys AA6014 % by weight can be seen in table 1 [10].

II. EXPERIMENTAL PROCEDURES

This section addressed the influence of annealing temperature and time on the micro structural evolution, precipitation kinetics, mechanical properties and thermal stability.

Therefore the experimental procedure is divided into three major areas:

1) The influence of precipitates in CG aluminium alloy AA6014:

Three initial material conditions are compared with as-received T4 conditions, solutionised conditions, and solutionised and aged conditions for 20h at 180°C.

2) Thermal stability measurements of CG and UFG materials:
In order to find out the thermal stability of the ARB processed materials, all the samples were annealed at 180°C up to 65 hours in furnace. In the ARB process, the AA6014-N2 and AA6014-N4 samples were annealed for a total time of 11 min. and 18 min. respectively.
3) Thermal stability of CG and UFG samples was investigated by heating the samples in a furnace for 1 hour at different temperatures of 100oC, 200oC, 300oC, 400oC, 450oC, and subsequently quench them in water. Finally, the Vickers hardness of the samples was measured using a Vickers Hardness Tester V-100 from Leco in the normal plane. The thermal stability of the CG samples was measured for comparison purposes. Differential scanning calorimeter, DSC 204 F1 Phoenix from Netzsch (Fig. 1) [11], measures the difference in heat flow between the test sample and the reference sample while both simultaneously heat in a chamber according to a given temperature program, where the relevant parameters such as the rate of temperature increase and the maximum allowable temperature can be specified. From the DSC curves, it is possible to recognize if processes such as transformation changes, precipitation development or recovery and recrystallization are taking place. Generally, any internal microstructural change, whichoccurs during heating, will cause a peak in the DSC curves, indicating that the sample requires more (or less) heat than the reference sample in order to maintain the constant temperature.

The sample was weighed and heated in the chamber at the rate of 10 K/min. The chosen temperature profile was repeated twice. During the first run, it was possible to observe the internal processes taking place within the sample and the second run were used as a reference. The sample was weighed and heated in the chamber at the rate of 10 K/min. The chosen temperature profile was repeated twice. During the first run, it was possible to observe the internal processes taking place within the sample and the second run were used as a reference.

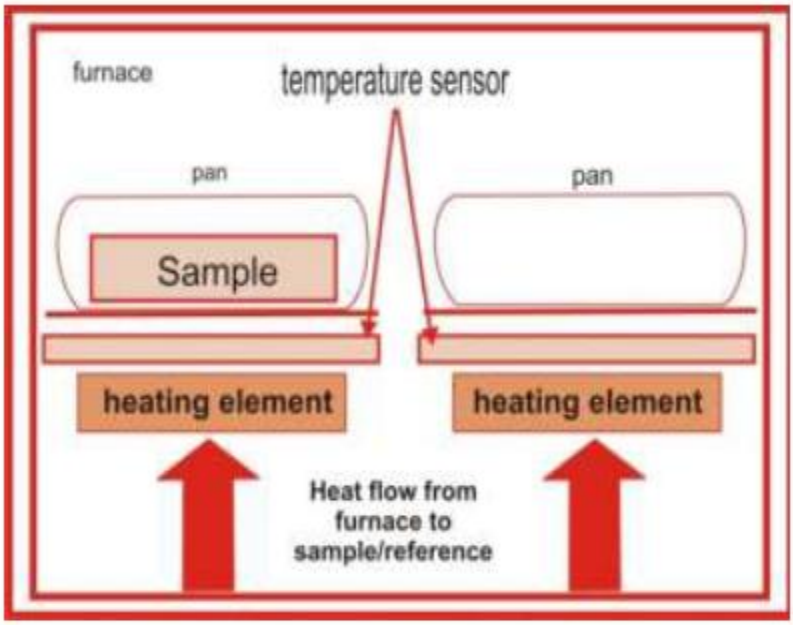

Fig.1. Schematic drawing of a differential scanning calorimeter

The all test samples were prepared by Accumulative Roll Bonding (ARB) Process belongs to a group of severe plastic deformation (SPD) techniques used to develop ultrafine-grained and/or nano-structured sheets by plying repeated rolling, which leads to high levels of shear strain throughout the sheet thickness. The surface of one millimeter thick and 50-100 mm wide metal strips was preliminary wire brushed in order to remove oxide layer for better interlamellar bonding of sheets, and subsequently folded or stacked on the top of each other and rolled together without lubrication using a two-high rolling mill (Vaid Engineering Industries, New Delhi, roller diameter: Ø = 135 mm) or a four-high rolling mill (Vaid Engineering Industries, New Delhi; roller diameter: Ø = 30 mm). The process was repeated a number of times. The roll diameter and the peripheral roll speed of the four-high rolling mill average 30 mm and 80 revs /min, respectively. The initial state of the material and the ARB parameters are listed in table 2.

Table Ii. Initial states of material prior to rolling and the corresponding arb process parameters

Material	Thick-ness reduc-tion	State before Initial Preheati ng	ARB cycles	Sheet Thickne ss	Operati ng Temp.
AA6014	50%	Solutioni sed (520^0 C/1 h) water quenche d	8-10	1mm	180^{0}C

III. RESULT AND DISCUSSION

1) Annealing Response of CG Aluminium Alloy

AA6014: It clearly show in Fig.2 that the hardness of the asreceived T4 conditions and solutionised conditions differ by around 30 HV10 without any artificial aging i.e. at room temperature (arrows 1 and 2). During artificial aging at 180°C, the hardness of these two states as- received and solutionised increases due to precipitation hardening.

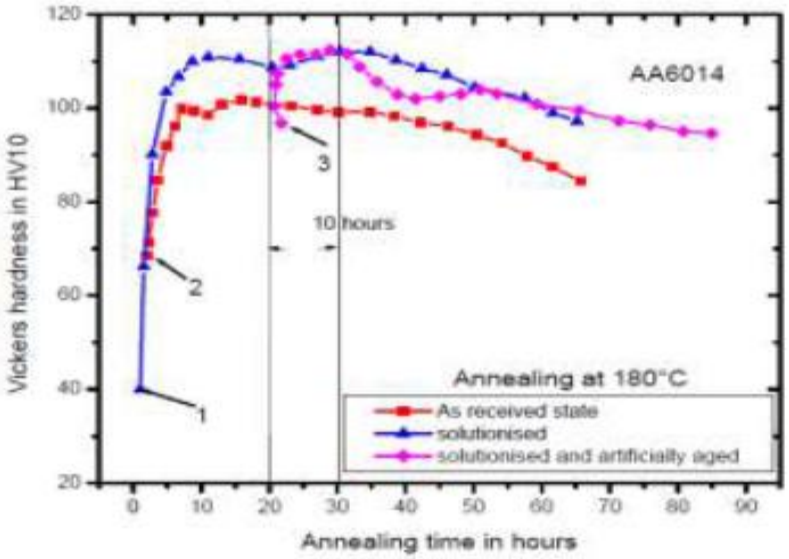

Fig. 2. Hardness of aluminium alloy AA6014 after annealing at 180°C in three different conditions

However, still there is a minor difference in hardness of the as-received and solutionised state during annealing. It shows that a higher hardness level can be achieved by

Material Thickness Reduction State Before Initial Preheating ARB Cycles Sheet Thickne Ss Operatingtemp.

AA6014 50% Solutioni sed (5200 C/1 h) water quenche d 8-10 1mm 1800 C artificial aging of the initially solutionised state Similar outcomes were also reported by Birol [12] who explored the pre-aging behavior of AA6014 in order to improve the bake hardening response. The aim is to estimate the necessary time for the completion of precipitation and averaging. The initial hardness of the test sample artificially aged for 20 hours at 180°C is 100 HV10 (arrow 3). On further annealing the hardness keep on the increasing and attains a maximum after an additional 10 hours annealing. After 30 hours of annealing, the hardness arrives at its maximum and then reduces due to averaging of the Mg_2Si precipitates, which rise in size and change their coherency. The precipitates cannot efficiently pin the dislocations and act as barriers to the dislocation motion, which indicates overall decrease in hardness. The hardness decreases after 30 hours can be pragmatic for all three material states

2) Annealing Response of ARB processed Aluminium Alloy AA6014:

In order to find out the thermal stability of the ARB processed materials, all test samples were annealed at 180°C up to 65 hours as shown in Fig. 3. In the ARB process, the AA6014-N2 and AA6014-N4 samples were annealed for total time of 11 min. and 18 min., respectively. After annealing for about 2-3 hours, a small increase in hardness is observed in the samples. This can contribute to the formation of two phase particles which may still grow up and vary in their morphology. The test samples rolled up to 6 numbers of ARB cycles which show no major difference in hardness during 2-3 hours of annealing at 180°C. On the other hand, the test samples of ARB rollbonded up to 8 times which demonstrate only softening during annealing. This shows that most of 2 phase particles have already precipitated out of the solution and achieved the greatest hardening effect during rolling. Therefore, the peaks in the hardness vs. annealing time curves move to the left in the direction of shorter annealing times with increasing number of ARB cycles.

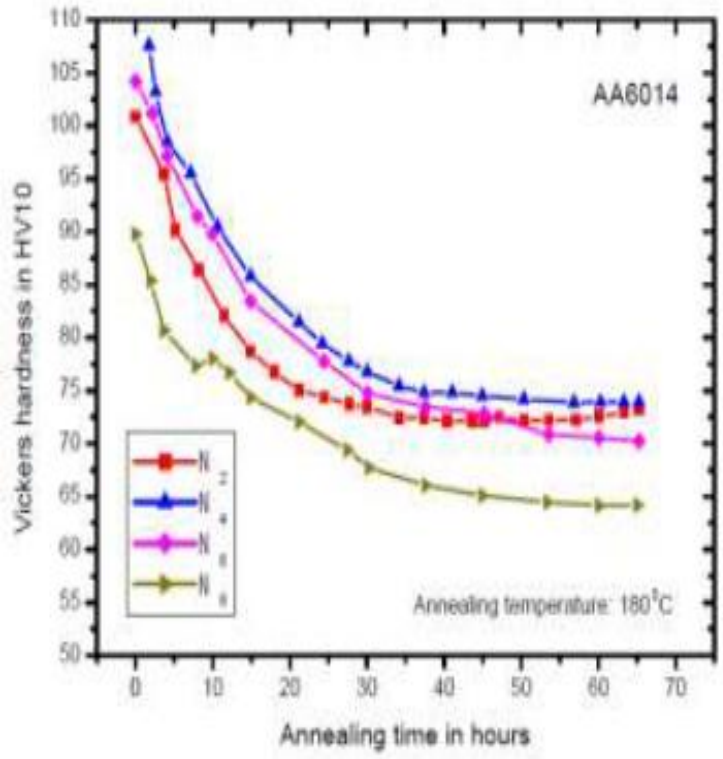

Fig. 3 Annealing responses of ARB processed aluminium alloy AA6014.

It appears that there is a transition phase after which no further material strengthening effect can be observed in annealing process. The transition phase lies most probably somewhere between the fourth and the sixth number of ARB cycle. All ARB processed test samples soften quickly after 10-20 min. of annealing at 180°C and attain a plateaubetween 45 and 65 hours of annealing. At this stage the ARB test samples are most likely re-crystallized. From Fig. 2 and 3, it can be observed that the ARB processed test samples precipitate sooner compared to the solutionised CG sample. The solutionised CG material accomplished saturation in hardness after about 30 hours, while the ARB processed samples need only ~60 min. (e.g. AA6014-N4) for the identical annealing temperature.

3) DSC Measurements of CG and UFG Aluminium

Alloy AA6014: The DSC measurements were performed on the CG and UFG AA6014 samples in order to examine the precipitation development and annealing response during the increase in continuous temperature. Fig. 4 shows three different initial conditions of aluminium alloy AA6014, namely: the solutionised, solutionised and 1 day aged, and solutionised and 7 days aged state. The three DSC curves illustrate the same development, but slightly different precipitation growth.

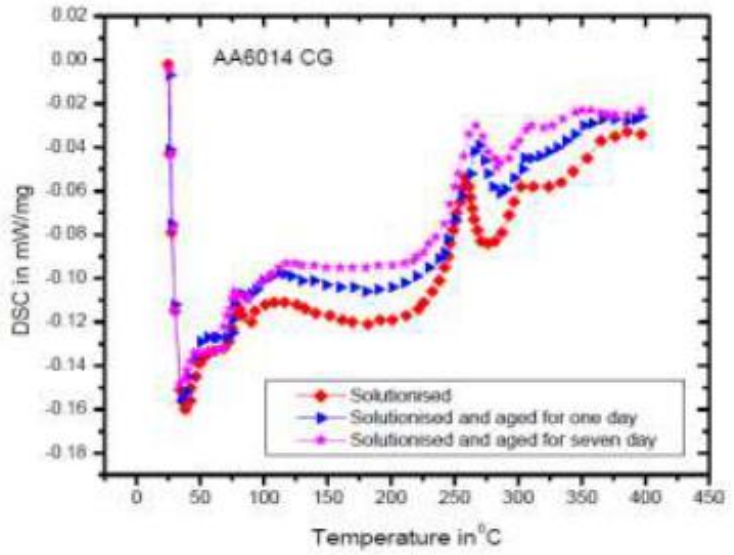

Fig. 4. DSC curves of aluminium alloy AA6014 of CG All samples were heated to a maximum temperature of 400°C at a rate of 10 K/min.

The solutionised state shows two leading peaks at in the region of 260 °C and 300 °C. Similar observations were also reported in the literature for the similar alloy and suggest the pattern of the β″-phase and β′- ase,respectively [13]. One day and seven days room temperature aging or natural aging results in the formation of β″-phase and β′-phase at elevated temperatures. Some Mg, Si and Mg-Si cluster form during early condition of heating at about 100 °C and influence the development of the β″-phase and the β′-phase. It looks like that it is essential for the dissolution of clusters occurring prior to the formation of the two states, since they act as nuclei for further formation of the β″-phase and β′-phase, and finally the stable Mg_2Si precipitates are observed [14, 12]. It indicates that theclusters in the naturally aged test samples are initially dissolved in the matrix, before the formation of β″ and β′phases begin. The precipitation order of the naturally agedsamples is slow down and the DSC curves are hence shiftedto elevated temperatures (Fig. 4). The pre mature clusteringat room temperature is usually damaging for the mechanicalproperties and usually not viable. In the paint bake cycleused in the automotive industry, annealing must beperformed at elevated temperatures and longer annealingtimes in order to get a maximum strength. This makes thepaint bake cycle of the naturally aged AA6014 sheetseconomical.

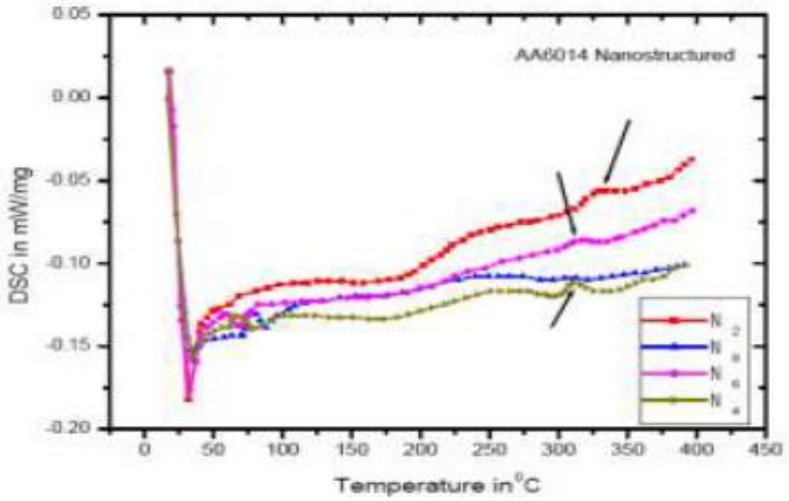

Fig. 5 DSC curves of aluminium alloy AA6014 of UFG samples processed by accumulative roll bonding. All samples were heated to a maximum temperature of 400°C at a rate of 10 K/min

Fig. 5, gives an idea about the DSC curves of the ARB processed AA6014 test samples. In terms of plastic deformation in the rolled samples the high quantity of stored energy results in a high driving force for

recovery and recrystallization. Thus, there are simultaneously three possible exothermal processes which may take place during heating of UFG test samples. The test samples may precipitate, recover during early phase of heating and later recrystallize. All three processes are overlapped to each other and it is not clear whether the exothermic peaks are due to precipitation or recovery. Samples AA6014-N2 andAA6014-N4 demonstrate minute exothermic peaks at 320°C and 340 °C, respectively. Thus, it is possible that there isstill some precipitation action such as precipitation growthand transformtion in the precipitation morphology. Due tothis reason results can be detected in small peaks in the DSCcurves (Fig. 5) and increase its hardness after annealing(Fig. 5). The AA6014-N6 test sample does not show anydifferent peaks in the DSC curves, while it can be arguedthat a small exothermal reaction is present at about 310°C. Conversely, the AA6014-N8 sample does not confirm anyexothermal peaks. These results match well with the resultsobserved in Fig. 5, where the AA6014-N8 test samplecontinually softens during post-roll annealing there was noadditional increase in hardness. Both results achieved fromthe DSC curves and the hardness values after annealing ofthe AA6014-N8 test sample, which indicate that theprecipitation evolution and growth may have alreadyaccomplished during the ARB process.

4) Thermal Stability of ARB processed Materials: It isexpected that SPD materials have low thermal stability. In order to examine the temperatures, which can be used forthe subsequent metal forming processes of UFG materials, itis significant to verify the appropriate process temperatures where the UFG microstructure is established.

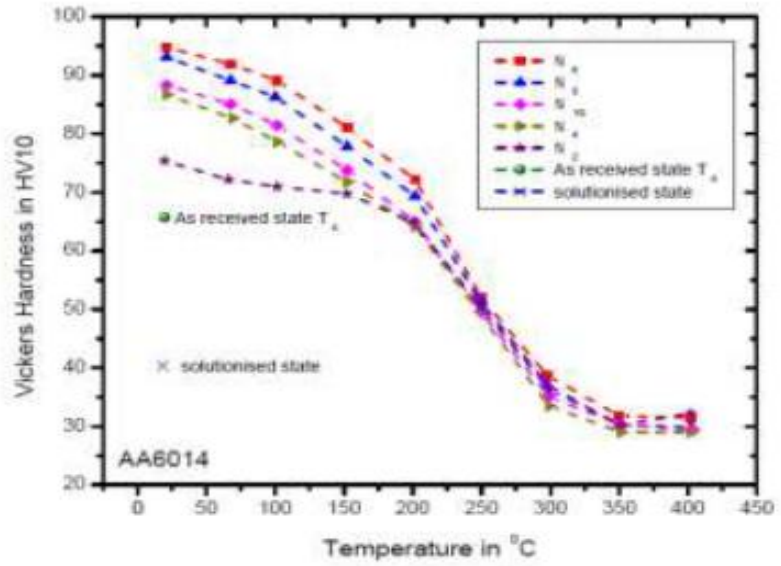

Fig. 6 Thermal stability curves of ARB processed AA6014 aluminium alloy roll bonded to 2, 4, 6, 8 and 10 ARB cycles. All samples were annealed for one hour at 100 °C, 200 °C, 300 °C and 400 °C.

Fig. 6 gives an idea about thermal stability curves of the ARB processed AA6014. The hardness values can be
evaluated to the as-received, T4 state and the solutionised state. It can be noticed that hardness varies from 40 HV10 in the solutionised state to 95 HV10 after 6 ARB cycles. The ARB test samples start to drop their thermal stability once they are exposed to higher temperatures. The ARB test samples are stable to 200 °C and start to soften quickly between 200°C - 300 °C. After 60 minutes annealing at 300 °C, the ARB samples attained just about the same hardness as the solutionised state. This specifies that the UFG microstructure is completely lost and the material is completely re-crystallized, related observations were also prepared by Park et al. [15] and Slamova et al [16]. Thus, itcan be understood that the thermal stability of the ultrafinegrained AA6014 is analogous to that of the UFG commercial pure aluminium AA1050 [12].

III. CONCLUSION

The effect of the second phase magnesium-silicon particles were investigated in the CG and UFG AA6014 samples. At room temperature aging of the solutionised AA6014 in the CG state leads to a rapid hardness increase from 40HV10 to 70HV10 within 7 days. Artificial aging shows that the material hardness may even increase after 20 hours of annealing at 180°C. Similar observations were also made by the DSC

measurements, which show a clear shift of DSC curves of aged specimens to higher temperature. Since the evolution of second phase particles contributes to the continuously changing mechanical properties, it is important to define the initial state of the material. For this reason, the conventionally grained AA6014 sheets were always solutionised before conducting the ARB process. It was found that the ultrafine-grained AA6014 alloy loses its thermal stability at approximately 200°C and recrystallised at 300°C. As a rule of thumb, it can be said that most ARB processed materials lose their thermal stability at approximately the same temperature at which they were processed or at a temperature 100°C higher than their process temperature. Thermal stability is strongly dependent on the material purity, second phase particles and/or oxide particles which may break up during rolling and lead to some dispersion strengthening.

ACKNOWLEDGMENT

Principal Author is very thankful to Prof. P. B. Sharma, Vice Chancellor Amity University, Haryana for his kind support and motivation.

REFERENCES

[1]. Langdon, T.G.: 'The current status of bulk nanostructured materials', Rev. Adv. Mater. Sci., 2012, Vol.31, pp. 1-4
[2]. Segal, V.M.: 'Equal channel angular extrusion: from macro mechanics to structure formation', Materials Science and Engineering: A, November 1999, Vol. 271(1-2), pp. 322–333
[3]. Zhilyaev, A; Langdon, T.G.: 'Using high-pressure torsion for metal processing: Fundamentals and applications', Progress in Materials Sci., August 2008, 53 Vol. 6, pp. 893–979
[4]. Richert, M; Liu, Q; Hansen, N.: 'Microstructural evolution over a large strain range in aluminium deformed by cyclic extrusion compression', Materials Science and Engineering: A, November 1999, Vol. 260(1-2), pp.275–283
[5]. Ratna, S. B.: 'Repetitive corrugation and straightening of sheet metals', Materials and Manufacturing Processes, 2015, Vol.30 (10), pp. 1262–1271
[6]. Verlinden, B.: 'Severe plastic deformation of metals', Metalurgija, Journal of Metallurgy, 2002, pp.165-182
[7]. Saito, Y., Tsuji, N., Utsunomiya, H., Sakai, T. and Hong, R.G.: 'Ultra- Fine Grained Bulk Aluminum Produced By Accumulative Roll-Bonding (ARB) Process', Scripta Materialia, 1998, Vol. 39 (9), pp. 1221-1227
[8]. Tsuji, N., Saito, Y., Utsunomiya, H. and Tanigawa, S.: 'Ultra-fine grained bulk steel produced by Accumulative roll-bonding (ARB) process', Scripta Materialia, 1999, Vol. 40 (7), pp. 795–800
[9]. Saito,Y, Utsunomiya, Tsuji, N. and Sakai, T.: "Novel ultra-high straining process for bulk Materials of development of the accumulative roll- bonding (ARB) process', Acta Materialia,1999, Vol. 47 (2), pp. 579-
[10]. 583
[11]. Kammer, C.: 'Aluminum hand book fundamentals of metals' 2002, Vol. 3
[12]. Kamikawa, N.; Tsuji, N.; Huang, X.; Hansen, N.: 'Quantification of annealed microstructures in ARB processed aluminum', Acta Materialia, 2006, Vol.54, pp. 3055-3066
[13]. Birol, Y.: 'Pre-aging to improve bake hardening in a twin-roll cast Al– Mg–Si alloy', Materials Science and Engineering A, Vol. 391, 2005, pp. 175- 180
[14]. Birol, Y.: 'Pre-aging to improve bake hardening in a twin-roll cast Al– Mg–Si alloy', Materials Science and Engineering A, Vol. 391, 2005, pp.175- 180
[15]. Edwards, G. A.; Stiller, K.; Dunlop, G. L.; Couper, M. J.: 'The precipitation sequence in Al-Mg-Si alloys', Acta Materialia, Vol. 46, 1998, pp.3893-3904
[16]. Park, K.T.; Kwon, H.J., Kim, W. J., Kim Y.S.: 'Microstructural characteristics and thermal stability of UFG 6061 Al alloy fabricated by ARB process', Materials Science and Engineering A, Vol.316, 2001, pp.124-152
[17]. Slamova, M.; Homola, P.; Slama, P.; Karlik, M.; Cieslar, M.; Ohara, Y.;Tsuji N.: 'Accumulative roll bonding of AA8006, AA8011 and AA5754 sheets', Materials Science Forum, 519-521,2006, 1227-pp.1232

*Sanjeev Sharma. "Annealing Response of Aluminum Alloy AA6014 Processed By Severe Plastic Deformation." International Refereed Journal of Engineering and Science (IRJES) 6.7 (2017): 59-65.