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Development of ultrafine-grained microstructure in Al-Cu-Mg alloy through equal-channel angular pressing

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Abstract. Al-Cu-Mg alloys are extensively used for riveting applications in aerospace industries due to their relatively high shear strength coupled with high plasticity. The significant advantage of using V65 aluminum alloy ((Al-4Cu-0.2Mg) for rivet application also stems from its significantly slower natural aging kinetics, which gives operational flexibility to carryout riveting operation even after 4 days of solution heat treatment, in contrast to its equivalent alloy AA2024.Rivets are usually made by cold heading of wire rods. In order to form a defect free rivet head, grain size control in wire rods is essential at each and every stage of processing right from casting onwards upto the final wire drawing stage. Wire drawing is carried out at room temperature to reduce diameter as well as impart good surface finish. In the present study, different microstructures in V65 alloy bars were produced by rolling at different temperatures (room temperature to 523K) and subsequently deformed by equal channel angular pressing (ECAP) at 423K upto an equivalent strain of 7. ECAP was carried out to study the effect of initial microstructure on grain refinement and degree of deformation on the evolution of ultrafine grain structure. The refinement of V65 alloy by ECAP is significantly influenced by Initial microstructure but amount of deformation strongly affects the evolution processes as revealed by optical microscopy and transmission electron microscopy.

Keywords: Al-Cu-Mg alloy, equal-channel angular pressing, ultrafine grain structure.

1. Introduction

Aluminum alloys play an important role in aerospace applications because of their favorable engineering properties. Selection of riveting material is one of the most important problems in the construction of aerospace structures; since any rivet failure will have serious consequences. Good formability in solutionised condition followed by self-induced natural aging is required for riveting applications. Rivets are usually made by cold heading operation on wire rods which are obtained by wire drawing operation. Microstructure and mechanical properties of wire rods play an important role, in the formation of defect free rivets. The microstructure has to be controlled at each and every stage of processing i.e., from casting to final wire drawing.

Relatively high shear strength coupled with high plasticity supports extensive use of Al-Cu-Mg alloys for riveting applications. AA2024 alloy is the choice material for rivets due to its high-strength and good formability in solution-treated condition followed by self-induced natural aging. However, the alloy has higher magnesium content which promotes natural aging kinetics due to large number of quenched in vacancies after solution treatment. This limits its application as a rivet because the riveting operation must be completed within

30 min of the solution treatment else the rivet would harden through natural aging process and becomes less formable. This problem could be overcome either by completing riveting operation within 30 min of solutionising or keeping the solution-treated stock at subzero temperature to retard the aging process, or by selecting a new material with reduced natural aging kinetics. Russian grade, V65 aluminum alloy is ductile enough to be deformed at room temperature after solution treatment and precipitation hardening. This alloy has relatively high shear strength combined with a high plasticity and can be riveted even after days of solution heat treatment and natural aging at room temperature. This individual valuable feature makes this alloy superior as rivet material over AA2024. However, a failure of V65 aluminum alloy rivets [1] has also been reported, due to the coarse grained microstructure in the rivet.

The strength of metallic materials with refined microstructures increases according to the Hall–Petch relationship [2, 3]. Numerous methods can be found to obtain ultra fine-grained materials with high strength based on this relationship [4, 7]. An alternative technique to classical deformation hardening methods is severe plastic deformation (SPD) which aims at obtaining ultra-fine grain structure, lower than1μm, uniformly throughout the volume and free from the cracks or damage. Among the SPD techniques, Equal Channel Angular Pressing (ECAP) is an effective method to produce homogeneous ultra-fine grained materials by introducing repetitive intense plastic straining (IPS) by extrusion i.e., ultra-high strains can be imposed on the worked material without a change in their shape or dimensions. ECAP is most promising, comparatively simple and easy to perform on various alloys and composites. Application to even large billets provides complete homogeneity in the final product [8]. The ultra-fine grained (UFG) materials produced by ECAP have a very high strength due to their low grain size and high dislocation density [9].

For understanding the mechanical behavior of UFG materials produced by ECAP, it is necessary to characterize their microstructure. Thus, the paper presents development of an ultra-fine grained microstructure in V65 aluminum alloy through ECAP. Some problems do occur during the ECAP at low temperatures. Intense shearing occurring during each pass in ECAP can result frequently in a premature failure of such materials due to the development of micro cracks under IPS conditions [10-12]. Therefore increasing the pressing temperature or warm ECAP [13,14] leads to improvement in the plastic workability which provides a greater flowability of material during ECAP.

2. Experiment

The method of processing of V65 grade bars from direct chill cast billets has been reported in detail elsewhere [15]. The bars rolled at RT and 523K, were subsequently cut and machined into rods with dimensions of 10mm diameter and 55mm length for ECAP. The die used for ECAP consists of two intersecting channels of same cross-section that meet at an angle Φ. The geometry of this tool provides that the material is deformed by simple shear at ideal, frictionless conditions. The cross-section of the specimen is subjected to ECAP several times in order to reach highest degree of plastic deformation. In the present study ECAP die of 10mm diameter circular cross section is employed with Φ=120° and Ψ=60°. It produces an equivalent strain of 0.60 for each pass using route B_c.

Specimens were then subjected to ECAP at peak aging and over aging temperatures i.e., 423K and 463K. The rods were preheated to the ECAP temperatures and then they were pressed through the die preheated to 423K. Repetitive pressing of the same rod was attempted by maintaining the temperature. Route B_C is adopted in this study by rotating the specimen counter clockwise 90° around its longitudinal axis between each pass, Molybdenum disulphide (MoS_2) was used as lubricant.

The ECAP samples were sectioned parallel and perpendicular to the longitudinal axis of the rods for TEM specimens. Thin foils (ground to a thickness of ~100μm) were prepared by grinding and electro polishing for TEM observation. The electrolyte consisting of 15%

nitric acid, 85% methanol was used and the temperature was maintained in between 233 and 243 K.

Hardness testing was carried out to evaluate the strength of the ECAP processed materials. Vickers hardness (VHN) was measured on the plane parallel to the longitudinal axes (designated as y-plane [16]), by imposing a load of 1kg for 15s.

3. Results and Discussion

Microstructure of as-received V65 aluminum alloy rolled at room temperature consists of elongated grains of average size of 21.6 μm (Fig. 1(a)). When rolled at 523K the alloy contains equiaxed grain structure of average grain size of 42.4 μm (Fig.1(b)). When ECAP was performed at 423K on a V65 Aluminum sample rolled at room temperature, it developed cracks after 12 passes and on a V65 Aluminum sample warm rolled at 523K sample, it developed cracks after 9 passes. When ECAP was performed at 463K for a sample rolled at room temperature there was a seizure of the sample in the die. Therefore warm ECAP is carried out at 423K (peak aging temperature of V65 Aluminum alloy) for all samples. Transmission electron microscopy images of the alloy rolled at room temperature before subjecting to ECAP (Fig.1(c)), show a microstructure with elongated grains and a uniform dispersion of the precipitate particles of very fine size and a minor volume fraction of blocky precipitates. The same sample when subjected to ECAP shows a significant increase in the volume fraction of fine precipitates in the initial passes, as observed in Fig. 1(d), while in the later stages of deformation the fine precipitates redissolve and only the blocky precipitates remain with a concurrent refinement of the matrix to an equiaxed grains (Fig. 1(e and f)).

The second set of samples of the alloy rolled at 523K show only blocky precipitates even before ECAP along with a small fraction of the precipitates having a typical capsule shape (Fig. 2(a)). The matrix grain structure remained equiaxed and large in size. With progressive ECAP passes, grain refinement took place along with a noticeable decrease in the volume fraction of the precipitates (Fig. 2(b-d)). The two types of precipitates could be identified to be Al_2Cu, the capsular shaped, and Al_2CuMg, the blocky ones, through electron diffraction patterns and corresponding dark field images, as shown in Figs. 3 and 4 respectively, The grain sizes were estimated for both set of samples by repeated measurement of the average cross-section of the grains in different orientations and is plotted against equivalent strain in Figs. 5 and 6. Grain size of as-received materials were measured from optical micrographs (Fig.1 (a) and (b)). There is a drastic decrease in grain size from the intial value by first pass in both cases. Further reduction in grain size by more number of passes takes place though it is imperceptible in the graph due to the scale chossen on Y-axis.

The vickers hardness values (VHN) are represented in Fig. 7 and 8 for the two sets of samples. The samples that were rolled at room temperature prior to ECAP (Fig. 7) show a decrease in hardness value as expected from their initial condition and reaches towards a constant value. The second set of samples, subjected to rolling at 523K prior ECAP show an increasing trend in hardness values with number of passes reaching saturation.

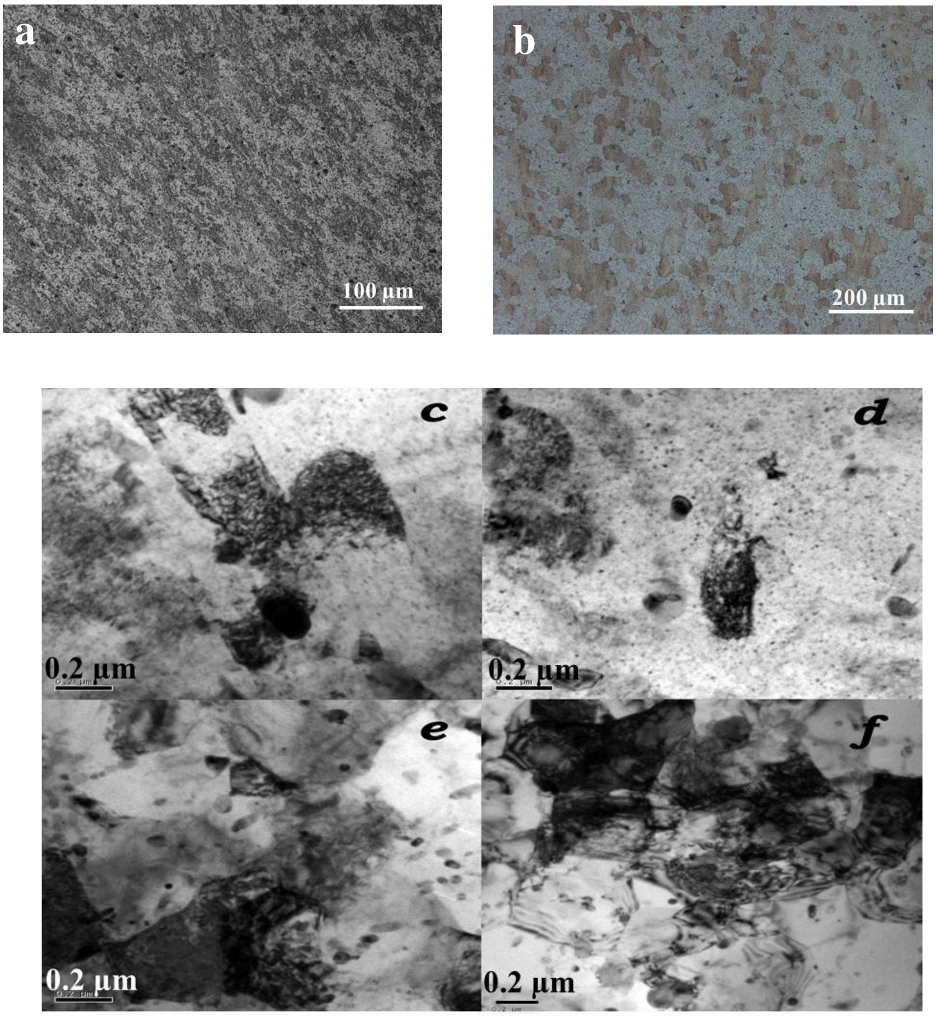

Fig. 1: Optical microstructure of as-received V65 aluminum alloy (a) rolled at room temperature and (b) rolled at 523K. TEM bright field images of V65 Aluminum alloy rolled at room temperature (c) as-received and ECAPed at 423K for (d) one pass (e) four passes and (f) ten passes.

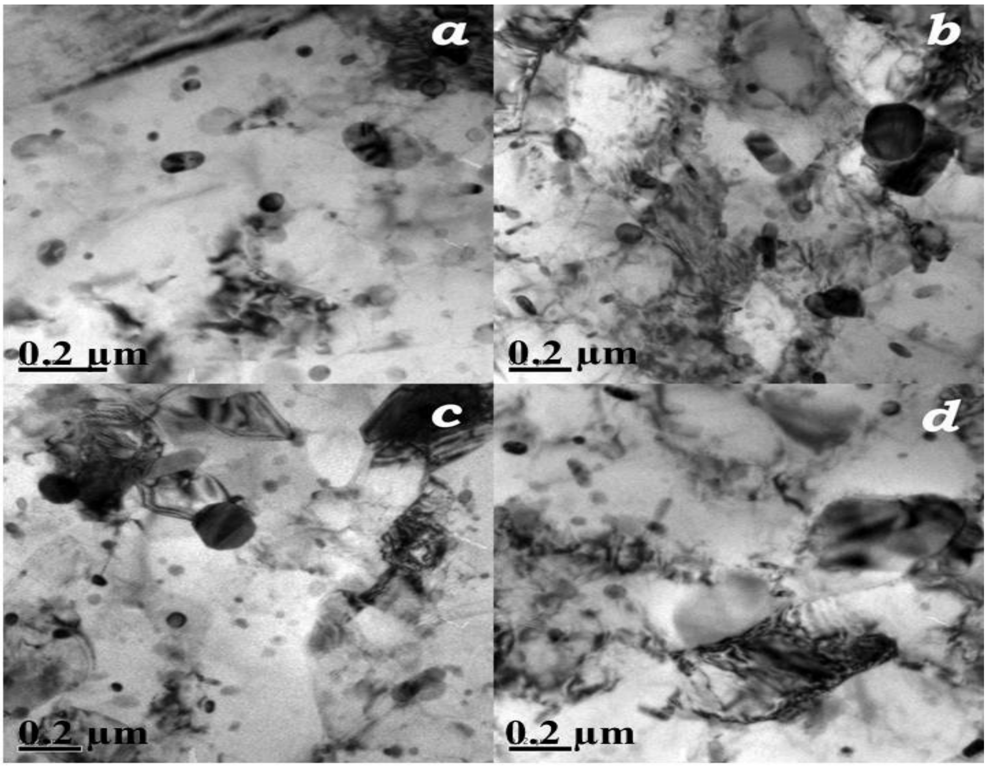

Fig. 2: TEM bright field images of V65 Aluminum alloy (a) rolled at 523K and rolling at 523K followed by ECAP at 423K for (b) one pass, (c) three passes and (d) eight passes.

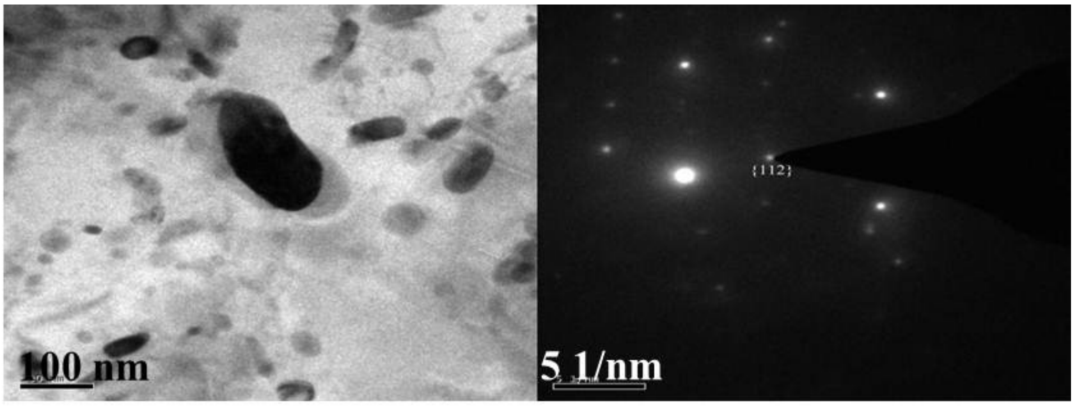

Fig. 3: TEM bright field image showing Al_2Cu precipitates in V65 Aluminum alloy rolled at 523K followed by ECAP at 423K for four passes, along with corresponding diffraction pattern. Reflection at arrow is from (112) of Al_2Cu.

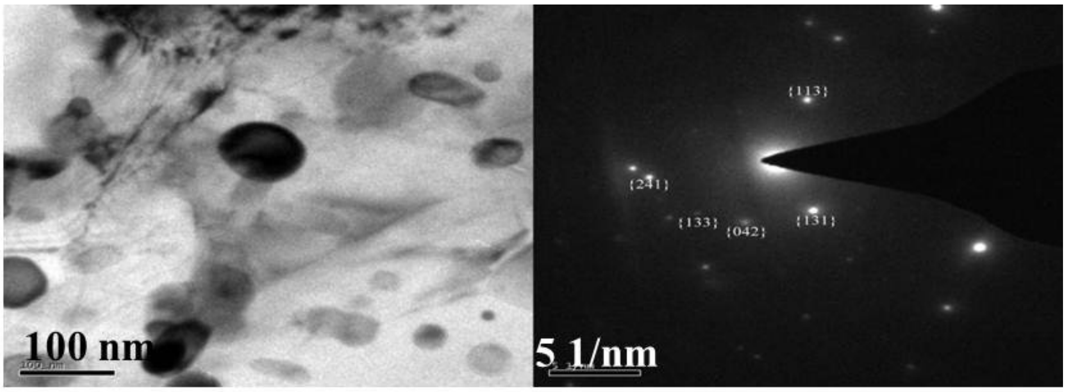

Fig. 4: TEM bright field image showing Al_2CuMg precipitates in V65 Aluminum alloy rolled at 523K followed by ECAP at 423K for eight passes, along with the corresponding diffraction pattern. Indexed reflections are from the Al_2CuMg precipitates.

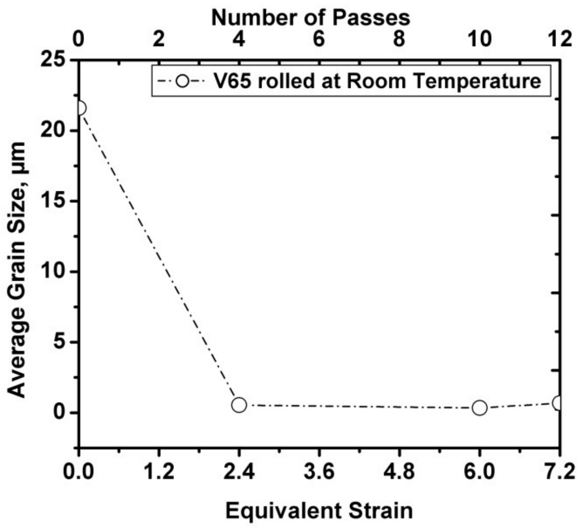

Fig. 5: Variation of average grain size (in µm) with equivalent strain, of the V65 aluminum alloy rolled at room temperature and rolled at 523K followed by ECAP at 423K.

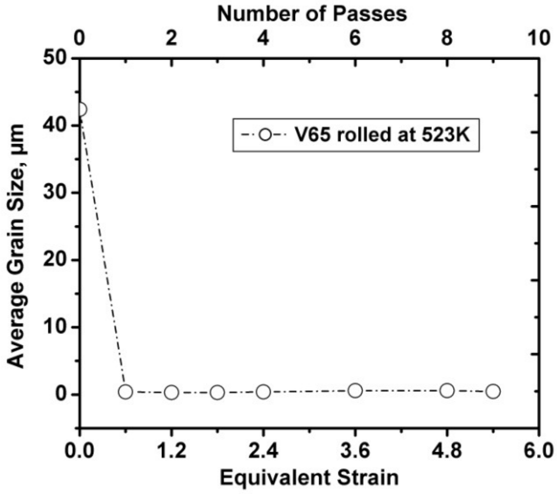

Fig. 6: Variation of average grain size (in µm) with equivalent strain, of the V65 Aluminum alloy rolled 523K and rolling followed by ECAP at 423K.

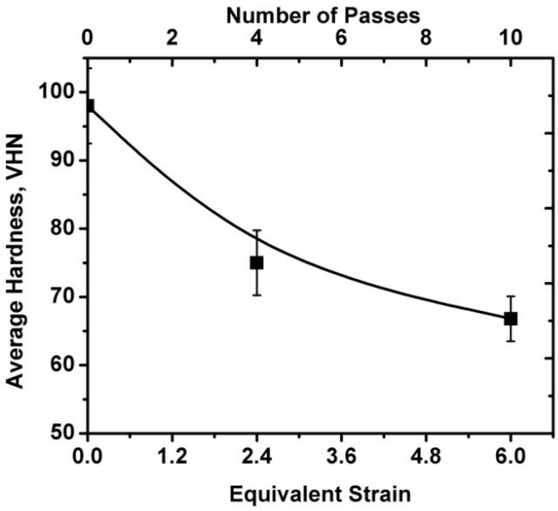

Fig. 7: Variation average hardness (in VHN) with equivalent strain, of the V65 aluminum alloy rolled at room temperature and rolled at room temperature followed by ECAP at 423K.

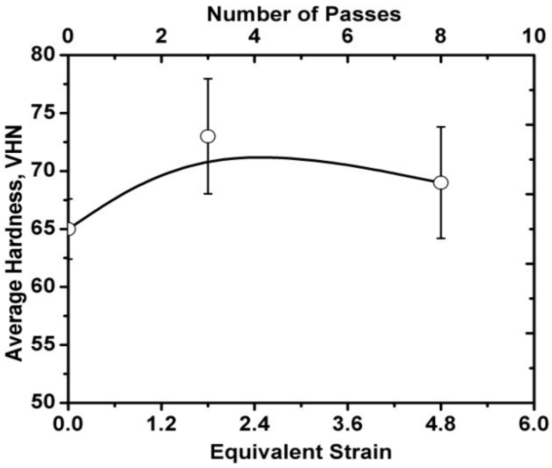

Fig. 8: Variation of average hardness (in VHN) with equivalent strain, of V65 aluminum alloy rolled at 523K temperature and rolled at room temperature followed by ECAP at 423K.

These observations reveal that significant contribution from grain refinement of the matrix did not give rise to any increase in strength. This trend could be understood from the way in which the grain size reduction and re-dissolution of strengthening precipitates were competing in both sets of samples. This behaviour also is in consonance with the decrease in hardness value with increasing number of passes (Fig. 7). Though an increase in hardness value is expected due to refinement in grain size (0.342 μm at equivalent strain of 6), the contribution to the hardness arising due to precipitation, which is a principal factor in these alloys, also need to be correlated with the above behaviour. The lack of contribution to hardness from precipitates can be understood by an observation of Fig. 1(e) which shows a large decrease in volume fraction of the precipitates due to their dissolution during ECAP (when compared to the initial condition)/re-precipitation in course size. In the initial condition, the alloy had high density of dislocations accumulated from cold rolling leading to a high hardness value 66.8 (VHN). Prior to ECAP these dislocations might have assisted heavy precipitation of θ′. Upon ECAP considerable rearrangement of the existing dislocations is understood to take place, along with the dissolution of existing precipitates in some cases. For this reason, the refinement in grain size may not be able to compensate for the fall in hardness values as function of equivalent strain.

The second set of samples had an initial microstructure (Fig.1(a)) with equiaxed large grains which experienced a steep fall in size by three passes of ECAP as normally expected. There is a concomitant increase in hardness value (VHN) (Fig. 8). The initial hardness value (VHN) of this set of samples was lower than the first set due to the high temperature at which rolling was carried out (523K). The strengthening effect due to precipitates may be almost negligible due to large size and sparse distribution of particles. The precipitates at this stage are largely Al_2CuMg phase and not Al2Cu (Fig 2(c)). Increase in grain size after 3rd pass (note that there is a minor positive to the curve in Fig.6) is difficult to understand. Some reports in literature show that such increase in grain size (grain growth) takes place in pure metals after a considerably large number of passes.

4. Conclusions

The decreasing hardness values for room temperature rolled V65 Aluminum alloy with progressive ECAP passes is primarily attributed to removal of strain hardening and decrease in volume fraction and an increase in particle size of the precipitates under ECAP processing conditions. Hardening due to decrease in grain size by ECAP is unable to compensate softening effect by coarsening of precipitates and reduction in volume fraction of precipitates. ECAP of hot rolled V65 alloy show strengthening due to grain refinement. Softening effect by coarsening of precipitates and reduction in volume fraction of precipitates bring hardness to a constant value or slightly decreasing trend. A re-solutionising treatment prior to ECAP may be desirable to effect the refinement of grain size through ECAP, subsequent to which an aging treatment can enhance precipitation in the alloy for achieving higher peak hardness.

References

[1] Jha A.K., Sreekumar K. and Sinha P.P., 2011, J. Fail. Anal. And Prven. **11(2)**:123-128.
[2] Hall E.O., 1951, Proc. Phys. Soc. B., **64,** 747–753.
[3] Petch N.J., 1953, J. Iron Steel Inst., **174**, 25–28.
[4] Segal V.M., 1995, Mater. Sci. Eng. **A 197**, 157–164.
[5] Mao J., Kang S.B., Park J.O., 2005, J. Mater. Process. Technol. **159,** 314–320.
[6] Wang Y.Y., P.L. Sun, Kao P.W., Chang C.P., 2004, Scripta Mater. **50,** 613–617.
[7] Furukawa M., Iwahashi Y., Horita Z., Nemoto M., Langdon T.G., 1998, Mater. Sci. Eng.A **257**, 328–332.
[8] Valiev R.Z., Langdon T.G., 2006, Prog. Mater. Sci. **51**, 881–981.
[9] Torre F. Dalla, . Lapovok R, Sandlin J., Thompson P.F., Davies C.H.J., Pereloma E.V., 2004, Acta Mater. **52**, 4819.
[10] Lee S., Utsunomiya A., Akamatsu H., Neishi K., Furukawa M., Horita Z., Langdon T.G., 2002, Acta Mater. **50**, 553–564.
[11] Vinogradov A., Washikita A., Kitagawa K., Kopylov V.I., 2003, Mater. Sci. Eng. **A349**, 318–326.
[12] Popovic M., Verlinden B., 2005, Mater. Sci. Technol. **21**, 606–612.
[13] Chakkingal U., Thomson P. F., 2001, J Mater Process Technol, **117**, 169–177.
[14] Ning J. L., Jiang D. M., Fan x. G., Lai Z. H., Meng Q. C., Wang D. L., 2008, Mater. charact, **59**, 306–311.
[15] Nayan Niraj, Murty S.V.S. Narayana, Jha Abhay K., Sharma S.C., Sreekumar K. and Sinha P.P., 2012, Mater. Sci. Forum **710**, 167-173.
[16] Furukawa M., Iwahashi Y., Horita Z., Nemoto M., Langdon T.G., 1998, Mater. Sci. Eng. **A 257**, 328-332.