

Characterization of ultra Ultra-fine Grained IF Steel and Al-1100 Sheets Processed by Accumulative Roll Bonding

S. Tamimiⁱ; M. Ketabchiⁱⁱ; N. Parvinⁱⁱⁱ

ABSTRACT

Severe Plastic Deformation (SPD), is a new method to reach ultra ultra-fine grain materials. The aim purpose of this project paper is to investigate whether Accumulative Roll Bonding (ARB), is an effective grain refinement technique for ultra-low-carbon steel strips containing 0.004% C and Al-1100. For this purpose, a number of aAccumulative rRoll bBonding processes were performed at 500°C for iInterstitial fFree and 250°C for Al-1100, with 50% reduction in area in each rolling pass. The mechanical properties after rolling and cooling were obtained, which includes yield and tensile strengths and elongation, were obtained. By increasing the number of aAccumulative rRoll bBonding cycles in iInterstitial fFree steel, the yield point and UTS of these samples increased by 430% and 330%, respectively. Also, elongation was reduced from 50.5% in annealed state to 2.6% after aAccumulative rRoll bonding process. In Al-1100, yield and tensile strengths increased by 130% and 164%, respectively while the elongation dropped from a pre-rolled value of 6.48% to 3.43%. The Ffracture surface of the specimens werefracture surface of the specimens was observed by SEM. For all samplenessamples, the hardness distribution along the thickness was directly proportional to the mean grain size according to Hall–Petch relation. The Hhardness of the samples was obtained using micro hardness tests. It was found that both the ultimate achievable grain size achieved, as well as the degree of bonding, depend on number of rolling pass and reduction in the area as a whole. In iInterstitial fFree steel, mean grain size was obtained using SEM and optical microscopy is about 225nm. Mean grain size in aluminum Al-1100, was estimated is determined about 480nm by SEM. The rolling process was stopped in after seven cycles 7 for Al-1100 and 10 ten cycles for iInterstitial fFree steel, when edge cracking of the edges became pronounced.

KEYWORDS

Severe Plastic Deformation, Accumulative Roll Bonding (ARB), Ultrafine Grain, Interstitial Free Steel, Al-1100

1. INTRODUCTION

Ultrafine grained (UFG) materials have grain size smaller than 1 μ m and present very high mechanical strength [1]. The high strength of UFG materials can largely reduce the weight of any constructions, and the strengthening without alloying elements would be preferable for recycling. Severe plastic deformation (SPD) techniques have emerged in the last decade as effective methods for the production of bulk metallic materials with very fine grain sizes. A rather large body of research has been published on the efficiency of techniques such as equal channel angular pressing (ECAP) and high-pressure torsion (HPT) for grain

refinement of a number of metallic materials [2]. The former, involves cycling a billet through an angular die repeatedly, which produces ultra-fine grain sizes ($d \approx 200$ – 300 nm). The use of HPT, where a disk-shaped sample is simultaneously pressed (with pressures in the order of GPa) and sheared by a plunger, has led to the production of genuine nanostructures ($d < 100$ nm). Though the ECAP and HPT can certainly fabricate bulk materials, the typical size of the samples is still small. Further, they are principally not a continuous process but a batch process [3]. In recent years, a number of alternative SPD technologies have been developed, including ARB, cyclic extrusion, cyclic bending, constrained grooved pressing,

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and others. The processing–microstructure relationships corresponding to these techniques are not clearly understood. ARB, in particular, has potential to be adopted by industry to produce fine-grained materials in the form of large sheets, due to its feasibility as a continuous process. This paper focuses on FCC BCC and BCC FCC structures in IF steel and Al-1100. IF steel does not have interstitial elements such as Carbon or Nitrogen, but has good formability. On the other hand, it does not have yield phenomenon in its stress-strain curve. This steel is used in industry today. Al-1100 is an important industrial material. It has high strength with low density. It is important to increase the strength of these materials by new methods. This paper deals with mechanical properties and microstructure studies of the mentioned materials after the ARB process.

2. EXPERIMENTAL PROCEDURES

A. Materials and process

Ultra-low-carbon steel and Al-1100 were used in this work. The chemical composition of these materials is given in Table 1.

Elements	Weight Percent	Elements	Weight Percent
Si	0.11	C	0.004
Fe	0.56	Ti	0.040
Mn	0.01	Mn	0.061
Mg	0.02	Ni	0.017
Zn	0.01	Cr	0.015
Ti	0.03	V	0.001
Al	Balance	Co	0.002
		Mo	0.002
		Sb	0.002
		Ta	0.002
		Nb ₂ N	trace
		Fe	Balance

TABLE 1. CHEMICAL ANALYSIS OF IF STEEL AND AL-1100.

The mean grain size of IF steel at the beginning was 17.6 μ m. The ARB was conducted under the conditions that the reduction in thickness per cycle was 50% (equivalent strain of 0.8). The ultra-low-carbon steel strips, nominally 2mm thick, were cut into samples of 30mm width and 170mm length. The surfaces of the strips were roughened using a wire brush, removing a thin layer of scale. After brushing, the surfaces were cleaned using acetone. The strips were then joined on the roughened surfaces and they were joined by aluminum rivet. The strips were placed in a furnace, pre-heated to 550°C and held there for 300s before rolling. Rolling at elevated temperature is advantageous for joinability and workability, though too high temperature would cause recrystallization and remove the accumulated strain. Strips were rolled dry to a nominal 50% reduction, at a velocity of 0.47 m/s (30 rpm, diameter of rolls were 30cm). No lubrication was used during the passes. The principle of the ARB process has been reported previously [4, 5]. The schematic illustration of the ARB process is shown in Fig. 1. The details of the ARB process have been explained by Lee [6] and Saito [5]. These procedures were continued up to 10 cycles. ARB process was done for Al-1100, too.

Annealed sheets Aluminum were cut in 300 \times 20 \times 1.5mm dimension. The mean grain size of Al-1100 in raw material was 14.8 μ m. They were preheated to 250°C and held there for 300s after preparing the Al sheets. They were rolled up to 7 cycles.

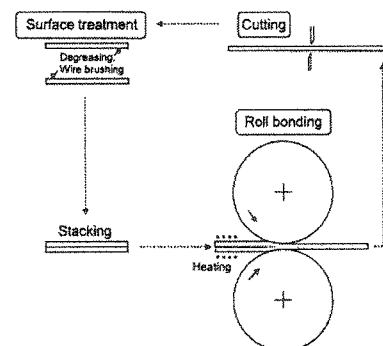


Figure 1: Schematic of ARB process.

B. Microstructure

The microstructure was investigated by optical microscopy and scanning electron microscopy (SEM) by using secondary electron probe. The microstructures were observed on the longitudinal (TD plane). IF samples were etched by nital solution after polishing. Also, Al samples were etched by 6ml HNO₃, 1ml HF(48%), 1ml H₂O.

C. Mechanical properties

The samples of IF steel and Al-1100 were strengthened with INSTRON instrument by strain rate approximately 8 \times 10⁻⁴ s⁻¹. The mean value of hardness in each samples evaluate by Vickers micro hardness with the

load of 50gr. The variation of Vickers hardness through the cross section of the specimens was measured too. Also, the effect of wire brushing on the surface of the samples was evaluated. For this, wire brushed sheet and the sheet without any wire brushing were joined with rivet followed by the ARB process. Also, the variation of hardness in the thickness of sample was measured. Finally, fracture surfaces were observed in both materials in the anneal state and after the ARB process.

3. RESULTS AND DISCUSSION

A. Microstructure

SEM macrographs of the ARB processed IF steel are shown in Fig. 2. In case of the specimen of ten cycles, 1022 interfaces exist across the thickness. However, only few unbonded regions of interfaces are seen in the specimen. This meant that the subsequent rolling improves the bonding of interfaces introduced in the previous cycle [7]. If the rolling reduction was insufficient for bonding, the bonded interfaces between the sheets would be clearly seen (Figs. 2.a, b.).

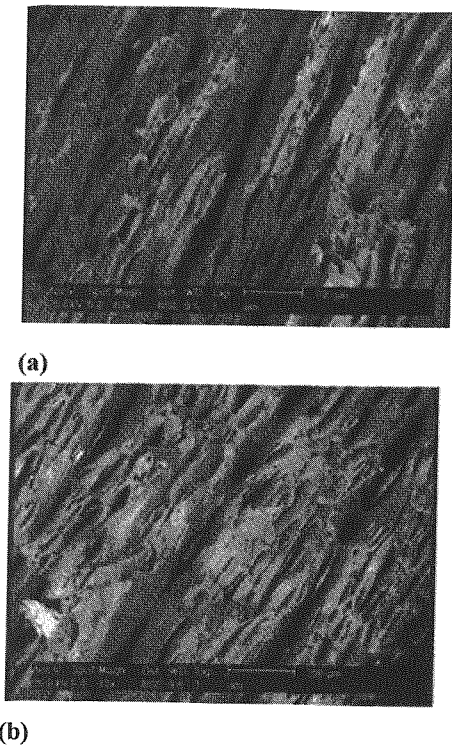


Figure 2: Interfaces in IF samples after ARB processed, a) 8 cycles, b) 10 cycles.

The ARB process has been successfully performed up to ten cycles without any shape defects in IF specimens and 7 cycles for the Al samples. Figs. 3 and 4 show the optical microstructures observed at the TD plane of the IF and Al specimen. Fig. 3.a. is related to the IF annealed sample. Grain shape in this sample is spherical with $17\mu\text{m}$ diameter. The microstructure of the specimen that was

produced by one and three cycles ARB are illustrated in Figs. 3.b, c. The microstructure of the specimen of one cycle ARB-processed showed relatively large grains, elongated to the rolling direction with clear grain boundaries [4, 8, 9]. As the number of ARB cycles increases, the microstructures become finer and more complicated. After 3 cycles it is difficult to detect any grain and their boundaries by optical microscopy. Fig. 4.a shows the optical microstructure of annealed Al-1100 alloy. It is possible to detect sphere grain with $14.8\mu\text{m}$ in diameter. Structure of Al samples after two and six cycles ARB are observed in Figs. 4.b, c. Figures depict that grains are elongated to the rolling direction, but at higher reductions microstructure become more complicated. Mean thickness size in two cycles sample is 920nm and in six cycles is about 480nm .

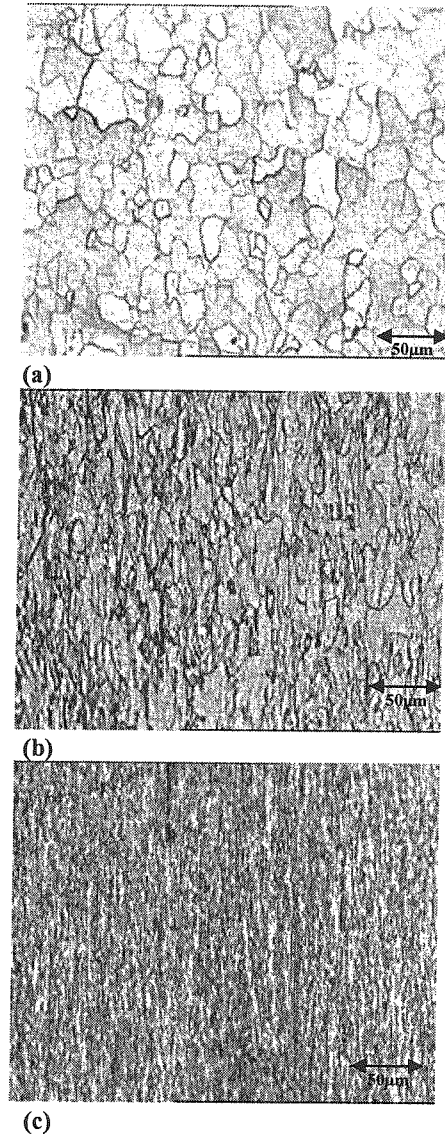
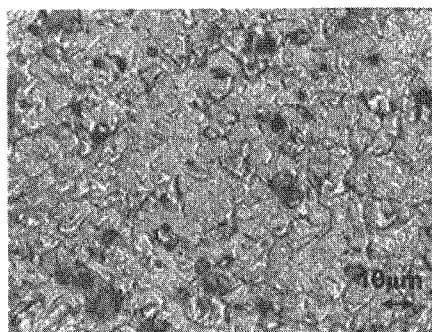
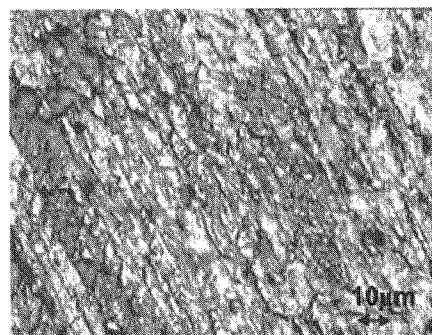


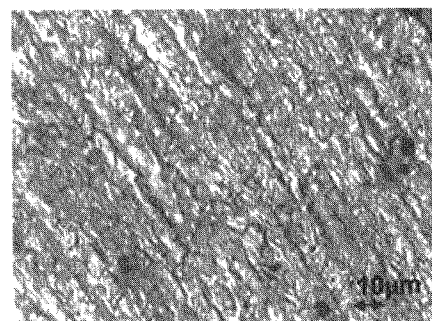
Figure 3: Optical micrograph of TD plane of IF steel samples, a) annealed, b) after one cycle, c) after three cycles ARB.



(a)



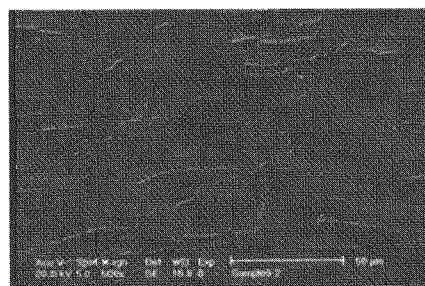
(b)



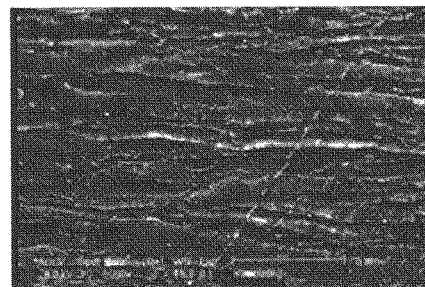
(c)

Figure 4: Optical micrograph of TD plane of Al-1100 samples, a) annealed, b) after two cycles, c) after six cycles ARB.

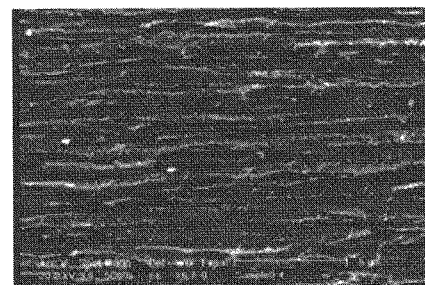
SEM micrographs of the ARB-processed IF steel after various cycles are shown in Fig. 5. The ultra-fine grains can be seen in the specimen. The fraction of these ultra-fine grains increased with the number of ARB cycles. The specimen after five cycles was covered with the ultra-fine grains of 543nm in average thickness surrounded by clear boundaries (Fig. 5.c). Mean grain size increases up to 8th cycle [6]. The ARB processed sheets after eight cycles were filled with the elongated ultra fine grains of 225nm in average thickness.



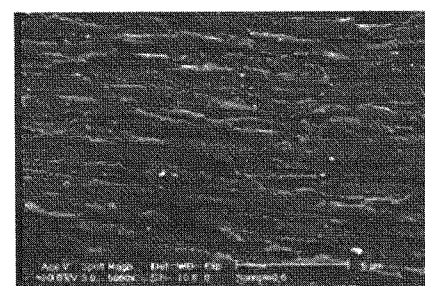
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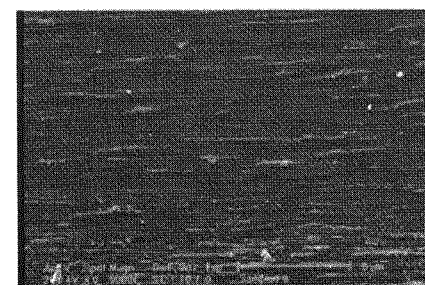
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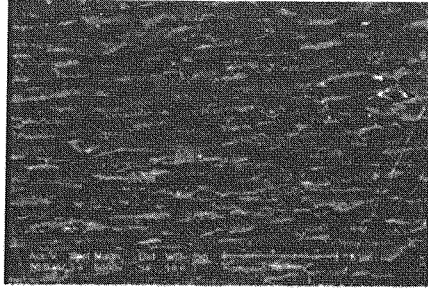
(c)



(d)



(e)



(f)

Figure 5: SEM micrographs in TD plane of IF steel, a) 1 cycle 500×, b) three cycles 5000×, c) five cycles 5000×, d) six cycles 5000× e) eight cycles 5000×, f) ten cycles 5000×.

Increasing ARB cycles led to increased grain thickness up to 389nm in the 10th cycle. This behavior can be discussed by dynamic and static recrystallization. The larger amount of strain energy accumulated in the material as a consequence of the higher reduction in thickness per pass might constitute a driving force for grain growth [2]. The finest grain size was observed in 8 cycle ARB process. Fig. 6. shows the variation of average grain size with respect to various ARB cycles in IF steels.

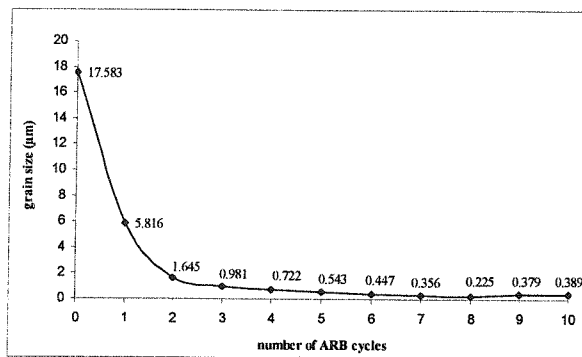


Figure 6: Variation of average thickness of grain after various ARB cycles in IF steel.

The formation process of these ultra-fine grains with the number of ARB cycles is same in various materials. Tsuji et al. suggested the formation mechanism of these ultra-fine grains in ARB process [10]. The materials ARB-processed by many cycles have severely strained structures with large local misorientations consisted of geometrically necessary (g-n) dislocations suggested by Ashby [11]. Tsuji et al. explained that the continuous changes in misorientation are converted into the planer boundaries by rearrangement of the g-n dislocations by short-range diffusion. The short-range diffusion is possible even at ambient temperature due to the temperature rise by plastic work. For both sample, IF steel and Al-1100, temperature of ARB process was high and diffusion can occur simply and it concluded grain refinement in specimens.

B. Mechanical properties

One of the most important aspects of the ARB process is tensile properties. Figs. 7 and 8 show variations of yield point, tensile strength and elongation in various cycles of ARB in IF steel and Al-1100. By increasing the reduction in area, values of yield point and tensile strength rise and their elongation lowers in both materials.

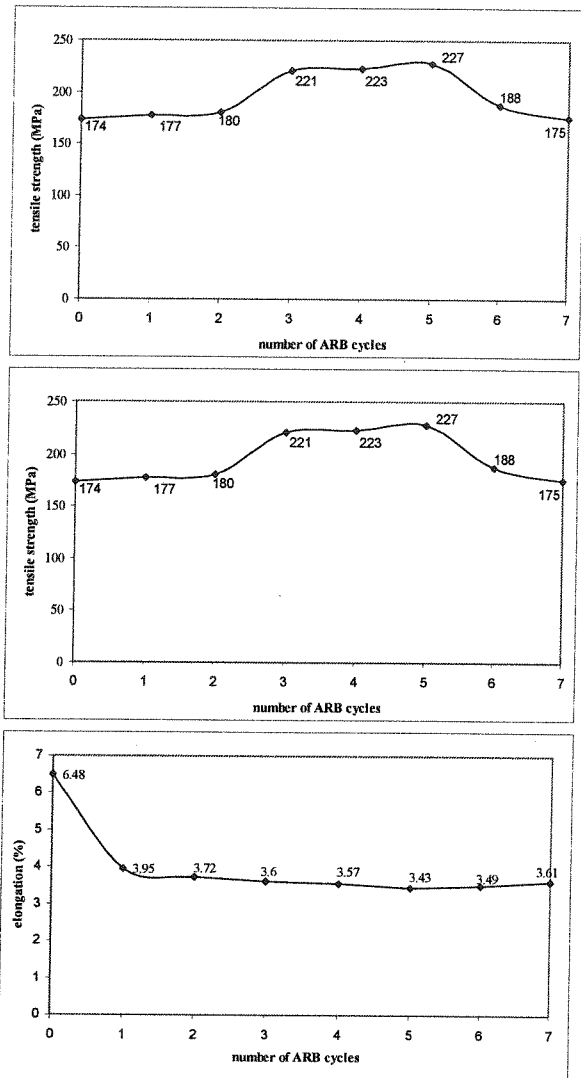


Figure 7: Variation of mechanical properties in Al-1100 by number of ARB cycles.

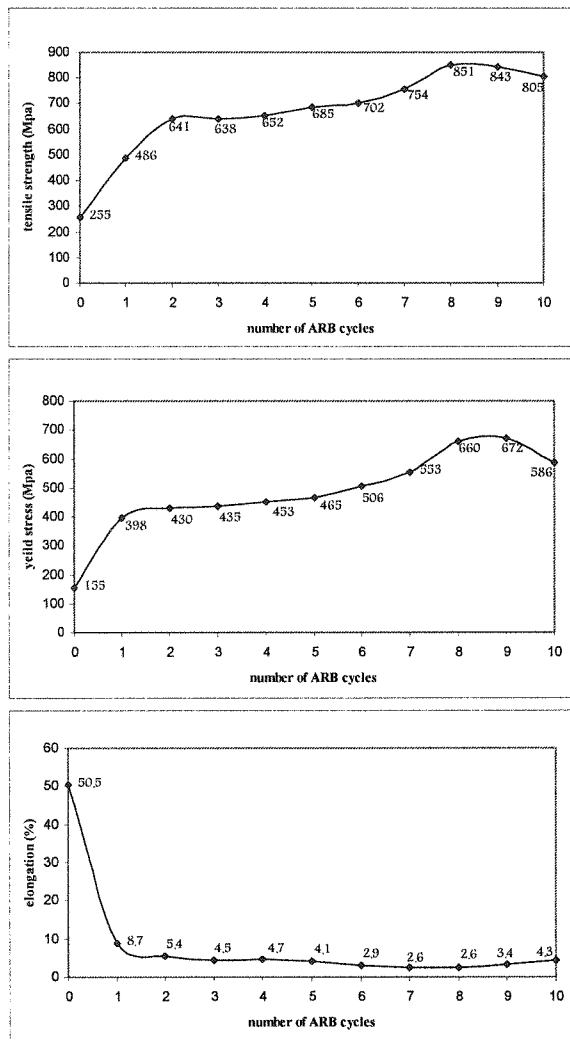


Figure 8: Variation of mechanical properties in IF steel by number of ARB cycles.

The strength properties of Al-1100 alloy are seen in Fig. 7. Yield stress in annealed state was 101.3MPa and it increased to 165.5MPa after five cycles. Also UTS value in these specimens, increased from 173.8MPa in annealed state to 227.4MPa after five cycles. On the other hand, elongation during ARB process was reduced. From these results, it can be concluded that 154% increase in yield stress and 130% increase in UTS and 52% reduction in elongation are all due to the favorable effect of ARB on mechanical properties in Al-1100. By continuing the process in higher cycles, yield stress and UTS decreased and elongation increased a little. These phenomena can be seen in IF steel, too. Yield stress at anneal state is 155MPa and it rose to 672MPa in cycle nine. Tensile strength in IF steel increased 255MPa in anneal state to 851MPa after eight cycles. After mentioned cycles the value of yield stress and UTS reduced a little (Fig. 8). Elongation decreased in primary cycles and it approximately remained constant after cycle three.

Elevation in yield stress and UTS can be justified by work hardening at the first. Large value of plastic deformation can occur in samples by ARB process. These plastic deformations cause work hardening. At this stage, grain refinement can be effective. Since the grain size in ARB samples is low, it concludes yield and tensile stress rise according to the Hall-Petch relationship. These two causes can help together to improve tensile properties [12]. However, previous observation can be affected by the formation of sub grain in materials. Increasing in misorientation angles in grain boundaries is the other reason for the improved tensile properties. By increasing the number of ARB cycles, the misorientation value of grain boundaries in both materials increased and the boundaries with low angles converted to boundaries with high angles. These high-angle boundaries and formed subgrains can prevent dislocation movements and cause higher yield and tensile. Decreased elongation in both materials can be justified by work hardening, too. By increasing the number of ARB cycles, values of yield and tensile strength decreased a little in both materials. These phenomena are for two reasons. The first cause is saturation in high level of accumulated strain in bulk material. At this stage, grains contain a large number of dislocations and it is impossible to produce a new dislocation in these grains. At this stage, the misorientation angle of grain cannot increase and in maximum state, it is about 36° for parallel boundaries with roll plane [13, 14, and 15]. Kyung-Tae Park et al., who worked on the behavior of ARB processed samples by temperature, believe dynamic recrystallization can affect samples [2]. Defect zones are higher in strained materials. In the thermodynamic aspect, this value of defects can provide conditions for recrystallization, which can be of two kinds. Dynamic recrystallization during rolling process and, also, static recrystallization when samples are in the furnace. Thus, saturation, static and dynamic recrystallizations can affect the tensile strength in high strained materials.

Fig. 9 illustrates the changes in Vickers hardness with ARB cycles in IF steel and Al-1100 alloy. In IF samples micro hardness increased rapidly at initial stage. After 7 cycles, the rate of increasing hardness reduces. Maximum hardness was reported after 8 cycles being 247HV. After this stage, the hardness of the samples decreased and was saturated by further rolling. These behaviors can be seen in Al-1100 alloy too. Initially, micro hardness increases up to five cycles. Next, in the final cycles, the value of micro hardness was decreased. Maximum hardness in this sample is related to five cycles sample by 48.9HV (Table II). These behaviors can be seen in tensile properties. During work hardening, the formation of subgrains and increasing in misorientation of grain boundaries can occur and also at final cycles, saturation, static and dynamic recrystallization are dominating procedures.

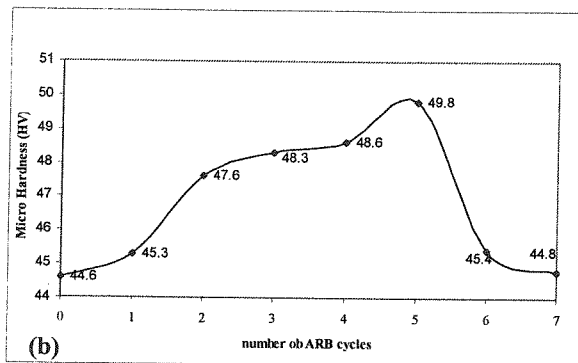
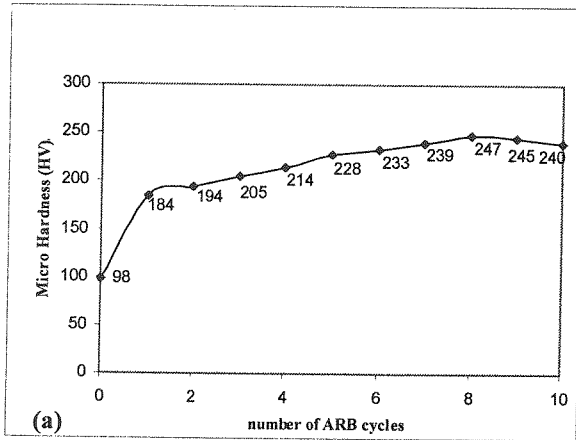


Figure 9: Variation of Vickers Hardness after various ARB cycles a) IF steel b) Al-1100.

Valiev et al. [16] and Furukawa et al. [17] showed the hardness due to ultra-fine grains in highly strained Al-Mg alloys. However, it was not ensured that the ultra-fine grained polycrystals show the same deformation mechanism as that of conventional materials which can be explained by the dislocation theory [18].

Fig. 10. shows the variations of hardness with thickness in IF steel samples. The hardness of the specimens is a constant value before the ARB. However, each specimen after one, five and eight cycles in the IF steel and after five cycles in the Al-1100 alloy shows inhomogeneous distribution in the thickness direction having higher values near the surface and center. The high value of hardness near the surface is caused by work hardening due to large redundant shear strain induced by high friction between roll surface and specimen during the ARB [19]. This hardening by the redundant shear strain near the surface has also an effect on the hardness at the center of the ARB processed specimens, except for specimen produced by one cycle, because the surface of specimen will be placed into the center of the specimen in the next cycle [6].

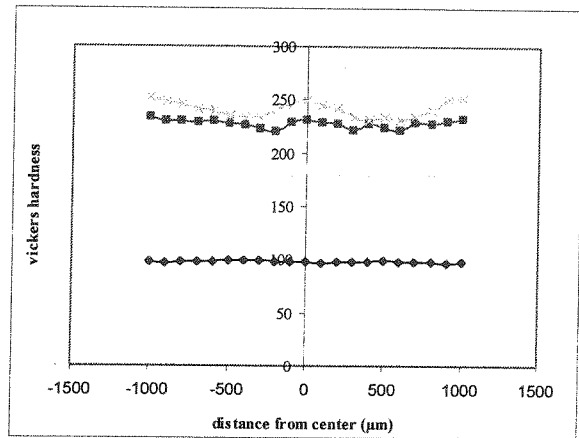


Figure 10: Variation of hardness with thickness.

The high value in hardness near the center in the ARB samples is caused by wire brushing as well. To investigate the effect of wire brushing on hardness, two IF steel sheets were tested in which only one sheet was wire brushed and it was found that the brushed sheet has higher hardness of about 7HV compared to the unbrushed sheet. This difference may be discussed by work hardening occurred during brushing (Fig. 11.)

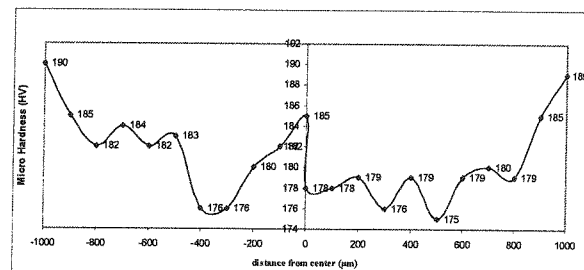


Figure 11: Effect of wire brushing on surface of IF samples.

C. Fractography

The fracture surface of the IF steel and the Al-1100 are illustrated in Figs. 12 and 13. By increasing the number of cycles, the size of dimples is smaller than the size of them in low cycles. Ductile and brittle are terms that describe the amount of macroscopic plastic deformation that precedes fracture. Ductile fractures are those that occur by micro void formation and coalescence, whereas brittle fractures can occur by either transgranular or intergranular cracking. Brittle fractures are observed in higher cycles. The ductile fracture can be detected in annealed samples [20]. The shape of the dimples produced is determined by the type of loading the component experienced during fracture, and the orientation of the dimples reveals the direction of crack extension. Equiaxed or hemispheroidal dimples are cup shaped, and they form under conditions of uniform plastic strain in the direction of applied tensile stress. In Fig. 12 and also Fig. 13 elongated dimples result from non-uniform plastic-strain condition such as shear overloads.

These are elongated in the direction of crack extension and reveal the origin of the fracture.

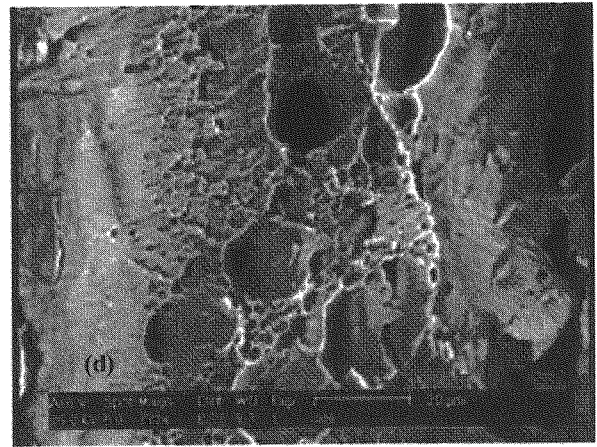
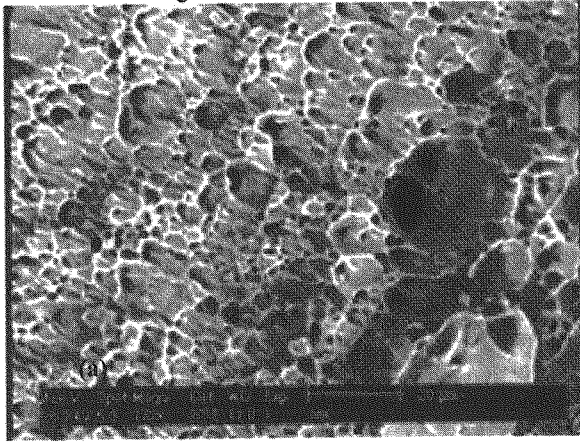


Figure 12 Fracture surfaces in IF steel after tensile test in a) anneal state b) 3 c) 6 d) 10 cycles ARB.

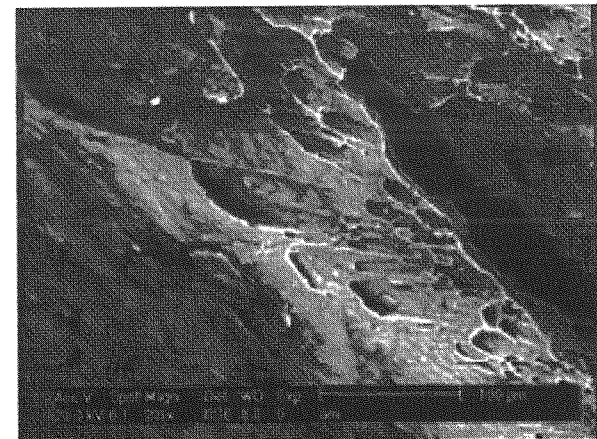
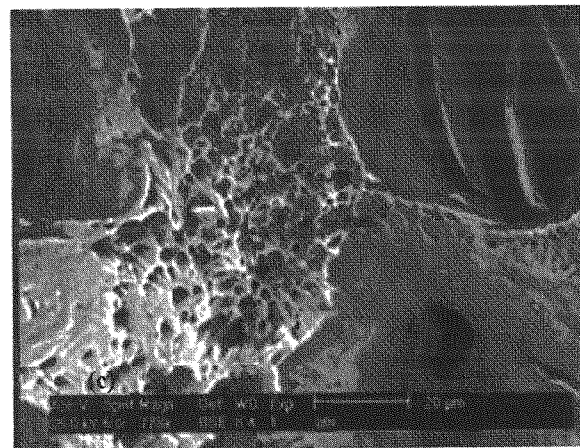
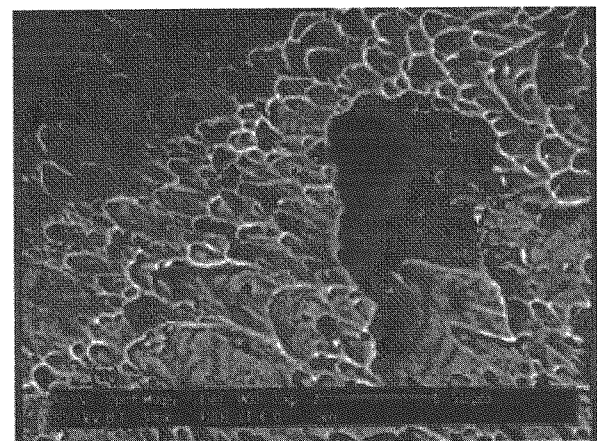
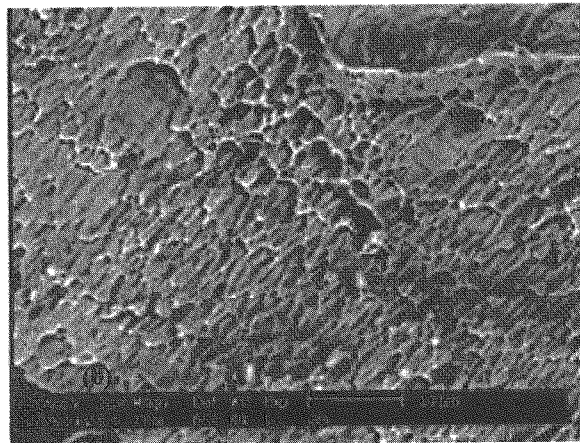


Figure 13: Fracture surfaces in Al-1100 after tensile test in a) four, b) seven cycles ARB.

The size of dimples is largely a function of the relative ductility of the material. Therefore, in annealed state, the

size of the dimples is large and by increasing the number of cycles, the sizes of the dimples are smaller. This suggests that annealed samples fail by ductile fracture and samples having accumulated strain exhibit a brittle fracture. It means, ductility reduces by increasing in reduction in area.

4. CONCLUSION

1- IF steel with an ultrafine grain structure was obtained by accumulative roll bonding. The grain size of 225nm was achieved by conducting up to 8-cycle ARB.

2- The Al-1100 alloy with an ultrafine grain structure was obtained by accumulative roll bonding. The grain size of 480nm was achieved by conducting up to 5-cycle ARB.

3- The hardness of the ARB processed IF steel increases with the number of ARB cycles, so that the specimen after eight cycles achieved the highest hardness of 247 HV, which is about 250% higher than the initial value.

REFERENCES

- [1] A.L.M. Costa, "Ultra grain refinement and hardening of IF-steel during accumulative roll-bonding", *Materials Science and Engineering A* 406 (2005) 279–285.
- [2] J.A. del Valle, "Accumulative roll bonding of a Mg-based AZ61 alloy", *Materials Science and Engineering A* 410–411 (2005) 353–357.
- [3]. N. Tsuji, "ARB (Accumulative Roll Bonding) and other new techniques to produce bulk ultra fine grained materials", *advanced engineering materials*(2003),5 ,no. 5.
- [4]. N. Tsuji, "Strength and ductility of ultra.fine grained aluminum and iron produced by ARB and annealing", *Scripta Materialia* 47 (2002) 893–899.
- [5] Y.Saito, Utsunomiya H, Tsuji N, Sakai T., "Novel ultra-high straining process for bulk materials—development of the accumulative roll-bonding (ARB) process" *Acta Mater* (1999) 47:579.
- [6] S.H. Lee, "Microstructures and mechanical properties of 6061 aluminum alloy processed by accumulative roll-bonding", *Materials Science and Engineering A* 325 (2002) 228–235.
- [7] Y.Saito,"Novel Ultra-high straining process for bulk materials development of the Accumulative Roll Bonding process", *PII: S1359-6454(9d8)00365-6*.
- [8] Inoue K, Tsuji N, Saito Y. In: *Proc of Int Symp on Ultrafine Grained Steels (ISUGS)*, ISIJ. (2001). p. 126.
- [9] Tsuji N, Saito Y, Utsunomiya H, Sakai T. In: *Ultrafine Grained Materials*. TMS;(2000). p. 207.
- [10] N. Tsuji, Y. Saito, H. Utsunomiya, T. Sakai, In: *The Proceeding of the Fourth International Conference on Recrystallization and Phenomena*, The Japan Institute of Metals, Proceeding, 13 (1999), p. 309.
- [11] M.F. Ashby, "The deformation of plastically non-homogeneous materials" *Phil. Mag.* 21 (1970) 399.
- [12] Z. P. Xing, S. B. Kang, H. W. Kim, 'Structure and properties of AA3003 Alloy Produced by Accumulative Roll Bonding Process', *Journal of Materials Science*, 2002,37,717– 722.
- [13] K-T.Park, "Microstructural characteristics and thermal stability of ultrafine grained 6061 Al alloy fabricated by accumulative roll bonding process", *Materials Science and Engineering A* 316(2001) 145–152.
- [14] K. Nakashima, Z. Horita, M. Nemoto, T.G. Langdon, *Mater. Sci. Eng. A* 281 (2000) 92.
- [15] Y. Iwahashi, Z. Horita, M. Nemoto, T.G. Langdon, *Acta Mater.* 46 (1998) 3317.
- [16] R. Z. Valiev, N. A. Krasilnikov, and N. K. Tsenev, *Mater. Sci. Eng. A* 137, 35 (1991).
- [17] M. Furukawa, Z. Horita, M. Nemoto, R. Z. Valiev, and T. G. Langdon, *Acta Mater.* 44, 4619 (1996).
- [18] Y. Saito, "Ultra Fine Grained Materials in bulk Aluminium Produced by ARB Process", *PII S1359-6462(1998)00302-9*.
- [19] S.H. Lee, T. Sakai, Y. Saito, H. Utsunomiya, N. Tsuji, *Mater.Trans., JIM* 40 (1999) 1422.
- [20] B.L.Gabriel, 'Scanning Electron Microscopy', *SEM Handbooks*, 1999, V.12, 173- 175.