Characterization of ultra-fine grained aluminum produced by accumulative back extrusion (ABE)

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ABSTRACT

In the present work, the microstructural evolutions and microhardness of AA1050 subjected to one, two and three passes of accumulative back extrusion (ABE) were investigated. The microstructural evolutions were characterized using transmission electron microscopy. The results revealed that applying three passes of accumulative back extrusion led to significant grain refinement. The initial grain size of 47 μm was refined to the grains of 500 nm after three passes of ABE. Increasing the number of passes resulted in more decrease in grain size, better microstructure homogeneity and increase in the microhardness. The cross-section of ABEed specimen consisted of two different zones: (i) shear deformation zone, and (ii) normal deformation zone. The microhardness measurements indicated that the hardness increased from the initial value of 31 Hv to 67 Hv, verifying the significant microstructural refinement via accumulative back extrusion.

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1. Introduction

Severe plastic deformation (SPD) is an effective approach to produce ultra-fine grain (UFG) and even nanograin (NG) materials. The UFG and NG materials exhibit superior mechanical properties comparing to their coarse grained counterparts, e.g. simultaneous enhancement of strength and ductility [1–3]. Recently, extensive studies have been carried out to develop various SPD techniques and to establish optimum processing parameters for fabricating UFG materials [4,5]. During the SPD process, the workpiece is subjected to intense plastic deformation resulting in the production of bulk UFG materials [6–8].

Among the SPD techniques, the ones based on extrusion process have attracted much more attention due to the capability of applying large deformations. This resulted in developing new SPD techniques based on extrusion process such as twist extrusion (TE) [9], cyclic extrusion and compression (CEC) [10], simple shear extrusion (SSE) [11] and torsion extrusion [12]. Recently, Alihossein et al. [13] developed a new SPD technique so-called cyclic forward-backward extrusion (CFBE).

Although ABE was introduced as a novel extrusion technique for the first time in 2009 [14], there are not many studies in the literature regarding this method. ABE is a kind of SPD in which the back extrusion (BE) and compression are carried out cyclically. As the BE and compression are performed many times on a workpiece, the term accumulate is used to differentiate it from the conventional extrusion process. The applied cyclic extrusion and compression leads to extensive grain refining. That is because severe plastic deformation causes an accumulated strain introduced into the workpiece when the material undergoes several extrusion–compression cycles. One significant advantage of ABE over BE is the absence of cracks and porosity in the processed workpiece.
Besides, it has been reported that ABE can be a very effective technique in producing the materials with ultrafine grains (UFGs) \cite{14,15}. Faraji et al. \cite{16} showed that the processing of AZ91 alloy by ABE led to an inhomogeneous microstructure. However, they showed that an increase in the inner punch diameter resulted in better microstructure homogeneity \cite{4,17}. Despite the extensive use of aluminum alloys, there are only a few studies regarding the ABE processing of these alloys. Alihosseini et al. \cite{18} indicated that after only one cycle of ABE process on AA6061 alloy, significant grain refinement was achieved.

The schematic of the ABE process is shown in Fig. 1. The inner punch with a given diameter is pressed into the work-piece. The movement of the inner punch causes the excess material to flow out through the gap between the die and the inner punch. The deformed material finally takes a cup-shape form at end of first-half pass. Then, the outer punch compresses back the cup-shaped specimen causing the inner punch move upwards to its initial state (end of second-half pass) \cite{19}. At this stage (end of first pass), the shape of workpiece is similar to its initial shape.

In the present work, the microstructural evolutions and mechanical properties of AA1050 subjected to several passes of ABE are investigated.

### 2. Experimental Procedure

The material used in this study was AA1050 aluminum alloy. Cylindrical specimens of 20 mm in diameter and 10 mm in height were machined, along the extrusion direction, from the as-received extruded rods.

The ABE die with 15 mm inner-punch diameter and 20 mm outer-punch diameter was manufactured from the tool steel having a hardness of 55 HRC. The die stroke, i.e. the maximum distance that the inner punch penetrates into the material, was 7.5 mm. The workpiece was subjected to three ABE passes using a 30 ton INSTRON machine operating at a cross-head speed of 10 mm/min. The friction between the specimen and the die was reduced by applying MoS2 as a lubricant \cite{4}. Microstructural evolutions were characterized using TEM technique (Philips CM 200 operated at 200 kV). Thin foils were prepared from ABEed specimens by twin-jet electro-polishing using a 400 ml HNO3 and 800 ml CH3OH solution. Microhardness measurements were conducted in the mid-diameter of specimens in their radial direction using a load of 200 g for 15 s.

### 3. Results and Discussion

#### 3.1. TEM Observations

The initial grain size of workpiece (about 47 μm) was refined to the grains of 500 nm after three ABE passes. Such a significant microstructural refinement can be attributed to severe shear deformation applied to the workpiece due to repeated back extrusion. In this regard, the cross-section of ABEed specimen was divided into two different zones: (i) shear deformation zone (region I in Fig. 2), and (ii) normal deformation zone subjected to normal strains (region II in Fig. 2). This is consistent with the findings of Faraji et al. \cite{16,17} who indicated that there were two distinct areas: (i) a shear deformation zone, and (ii) a normal strain zone. They proved that the grains located in the shear deformation zone were much finer than the ones formed in the zones subjected to normal strains. Faraji et al. \cite{17} reported that in the ABE processing with the same die geometries used here, in the region (I) the plastic strain was about 1.5, while in the region (II) it was about 5 after the first pass.

In this paper the terms “(sub)grain” and “(sub)grain size” were used when discussing the microstructure of the first two passes of ABE. Due to an absence of an experimental proof for the occurrence of a very large fraction of high angle boundaries in this work, the pioneers of SPD have defined that a structure consists of grains when at least 85% of the grain boundaries are high angle boundaries.

Figs. 3 to 5 show the TEM micrographs and the histograms regarding the size distribution of (sub)grains formed in the specimens subjected to different ABE passes.

According to Fig. 3, the microstructure after one ABE pass is inhomogeneous due to a difference in local accumulated plastic strain. After the first pass, the (sub)grain size in the shear and normal deformation zones is about 950 nm and 5 μm, respectively which corresponds to the strain level of 5 and 1.5 respectively, as reported in Ref. \cite{17}. The microstructure consists of deformed and non-deformed grains containing some kind of dislocation tangles. This is in agreement with the past works \cite{20–22} who attributed this phenomenon to the inherent inhomogeneous plastic deformation when the work-piece is subjected to only one ABE pass. The microstructure illustrated in Fig. 3 indicates a typical cell structure with its SAD pattern. This is in agreement with the results reported by Park et al. \cite{20}. After the first pass of ABE, some original coarse grains are divided into (sub)grains. The structure in zone I consisted mainly of (sub)grains and dislocation cell structures \cite{20,21}. In the region II (Fig. 3), the grain refinement is noticeable because of lower accumulated strain which is about 1.5, as mentioned in Ref. \cite{17}. In this region, the work-piece is likely affected by normal strain rather than by shear strain.

Fig. 4 shows the TEM micrographs taken from the regions I and II of the sample subjected to two ABE passes. From Fig. 4, it is clear that the region I contains the (sub)grains of 650 nm (which corresponds to a strain of ~10 \cite{17}); while in the region II, the average size of (sub)grains is about 815 nm, which corresponds to the strain level of ~3 as already discussed in Ref. \cite{17}. The (sub)grains are mostly equiaxed and the grain
Boundaries are sharp. The ultra-fine grained structure became more dominant and there are also some regions with slightly elongated (sub)grains which present a band-like structure as shown in Fig. 4 [22,23]. The relatively larger (sub)grains are elongated and subdivided by dislocation boundaries. Therefore, the grain structure consists of mainly the (sub)grains or dislocation cell structures [22,24,25]. Comparing the microstructures of region II in the samples subjected to one and two ABE passes, the grain boundaries are visible in the latter case. Considering the histograms illustrated in Fig. 4, by increasing the number of ABE passes, a more homogenous microstructure is attained and the distribution of (sub)grain size becomes narrower. This implies that the ABE process can be a practical approach for grain refining and formation of ultra-fine (sub)grains.

Fig. 5 compares the microstructure and histograms taken from both regions of samples after three passes of ABE. According to this figure, the ABE can lead to more homogenous microstructure after three passes. The microstructure is mainly consisted of ultra-fine grains with the average grain size of about 500 nm. Besides, their distribution is also more uniform. In this case, most of the grain interiors are dislocation-free [25,26]. Obviously, the microstructure is quite different from the one obtained after one and two cycles of ABE (Figs. 3 and 4). In the case of three passes, the histograms of both regions indicate that the homogeneity of microstructure is significantly improved. In addition, the size distribution of grains becomes much narrower compared to the one obtained after the first and second passes.

By increasing the number of passes from one to three, the shape of (sub)grains changes from elongated to equiaxed one. Meanwhile, the (sub)grain size decreases by increasing the strain level from the first pass to the third one. These microstructural evolutions are obvious by referring to Figs. 3–5. To provide a better comparison, the quantitative values regarding the (sub)grain sizes in different regions, are gathered in Table 1 for three passes of ABE.

Considering the microstructural evolutions presented in Figs. 3–5, it is clear that by increasing the number of passes, the inhomogeneous microstructure has disappeared and a homogeneous one is obtained instead. Fig. 6 schematically shows the extension of region having much refiner grain to the whole region of sample by increasing the number of ABE passes from one to three. In other words, increasing the number of ABE passes leads to better microstructure homogeneity through the whole microstructure. It is well known that in the SPD process such as ECAP, the increase in the number of passes in the grain refinement is more effective in the early SPD passes (e.g. during the first and second passes) [27]. Fu et al. [27] showed that the grain size of AA6061 alloy after the first four passes of ECAP was decreased to 3.7 μm from 66.3 μm, while it was decreased to 0.86 μm after the second four passes of ECAP. This implies that in a sample subjected to SPD, although the distribution of introduced strain may not be uniform, the microstructure looks homogeneous. Since the ABE process divides the sample into two distinct areas having different equivalent plastic strains [16,17], one can conclude that the (sub)grains in the regions of lower accumulated strain can be much finer than those in the regions of higher equivalent plastic strains. Faraji et al. reported that one pass ABE divides the sample to two lower and higher accumulated strain levels of ~1.5 and ~5 respectively [17]. In the 2 passes ABE process these two accumulated strain levels increase to about ~3 and ~10. Increase in the number of passes from one to three may lead to divide the sample to two lower and higher accumulated strain levels of ~5 and ~15 respectively. This statement needs some finite element validations that will be reported in the near future.

The grain refinement could occur due to the accumulation of strain as well as due to the change of a strain path [28,29]. During the first-half cycle of ABE, the material passes through
Fig. 3 – TEM micrographs, corresponding SAD patterns and grain size histogram of one pass ABE processed sample in: (a, b, c) region I, and (d, e, f) region II.
the "shear-deformation channel". This means that while the inner punch moves down, the material is deformed by a shear straining process [15,16]. Considering the above discussion, during the half cycle of the ABE process, severe deformation is employed to the material when passing through the shear-deformation channel. However, during the second step, the material undergoes an inverse route while the deformation channel becomes broadened [15]. In such a case, the normal deformation is dominated. In other words, as back extrusion is applied, the workpiece is compressed back to its initial shape during the second-half cycle. This means that the normal strain zone prevails over the shear deformation, i.e. the deformation geometry associates with less severe shear-deformation. Moreover, the fine recrystallized (sub)grains, which can be formed at the end of first-half cycle, may relax the strain hardening generated during the second-half cycle. This is in accordance with the work of Lapovok et al. [30]. Generally speaking, by increasing the passes of ABE, the shear-deformation channel is more broadened due to an increase in the shear deformation zone (region I), as is shown in Fig. 6. In fact, the ABE technique introduces severe localized shearing, especially at the vicinity of grain boundaries, as well as extensive shear bands [30–32] in the matrix. It is reported [19] that due to the occurrence of continuous dynamic recrystallization during SPD, the (sub)grain size was significantly decreased resulting in a microstructure consisted of very fine and equiaxed (sub)grains.

As aluminum is among the materials of high stacking fault energy (SFE), it will be restored by the dynamic recovery (DRV) rather than the discontinuous dynamic recrystallization (DDRX). Besides, as recovery normally occurs at about 0.3 Tm, which is the case here, it is therefore more likely that DRV or continuous dynamic recrystallization (CDRX) is responsible for the grain refining, as already reported by many researchers [19]. In general, CDRX refers to the gradual conversion of (sub)grains to grains during SPD, whereas DDRX occurs by the nucleation of new (sub)grains through the bulging of initial grain boundaries followed by their conversion to new recrystallized grains. The latter normally takes place in the materials of low SFE, which cannot be the case in the present work.

3.2. Microhardness

The hardness profiles obtained across the cross section of ABeed samples are compared in Fig. 7. In general, the hardness is increased by increasing the number of passes. The most increase in the hardness was achieved after the first pass of ABE. Although, there is also a considerable increase
in the hardness after the second pass of ABE, a moderate increase in the hardness was observed after the third pass. Comparing to the initial hardness (31 Hv), there is a significant increase in the hardness after three passes of ABE (67 Hv), i.e. an increase by a factor of ~2.2. This is in accordance with the observed changes in the hardness during the other SPD techniques such as equal channel angular pressing (ECAP) [33]. The variations in the hardness after the first pass of ABE are in good agreement with those reported by Faraji et al. [16]. They found that the lowest hardness occurred in the outer region of the AZ91 alloy subjected to ABE [16]; the reason was that the outer regions were subjected to normal strains rather than the shear ones. In the other regions subjected to shear strain, the obtained hardness was much higher resulting in more grain refinement. According to the well-known Hall–Petch relationship, a decrease in the (sub)grain size leads to an increase in both strength and hardness [14]. Therefore, by increasing the number of ABE passes, more grain refinement and higher hardness can be achieved because of higher strain applied. Although, there are some irregularities in the hardness profile after the first pass, almost a uniform hardness profile is attained after the third pass. The changes in hardness, from the first to third pass of ABE, are in good agreement with the microstructural evolutions discussed in the previous sections.

### Table 1 – The summary of quantitative values of the grain sizes regarding the different regions and passes.

<table>
<thead>
<tr>
<th>Number of passes</th>
<th>Grain size</th>
<th>Region I</th>
<th>Region II</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>950 nm</td>
<td>5 μm</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>650 nm</td>
<td>815 nm</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>500 nm</td>
<td>550 nm</td>
<td></td>
</tr>
</tbody>
</table>

4. Conclusions

The microstructural evolutions of AA1050 aluminum alloy subjected to three passes of ABE were investigated. The main results are summarized as follows:

1. A significant grain refinement can be achieved by applying different passes of ABE. A homogeneous microstructure
with the grain size of about 500 nm (initial grain size of 47 μm) was achieved after the third pass of ABE.

2 Microstructural homogeneity of ABEed samples increased by increasing the number of ABE cycles.

3 Microhardness measurements verified the microstructure results. The microhardness was increased from 31 Hv to 67 Hv after 3 passes of ABE.

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Fig. 6 – Schematic of expanding high possibility grain refinement zones in different passes of ABE.

Fig. 7 – Microhardness profiles of ABEed samples for different passes.

REFERENCES

31 El-Danaf EA. Mechanical properties, microstructure and texture of single pass equal channel angular pressed 1050, 5083, 6082 and 7010 aluminum alloys with different dies. Mater Des 2011;32:1383–53.