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ISSN 0543-5846
METABK 48(1) 15-18 (2009)
UDC – UDK 669.14-418:539.37:620.17=111

FORMING OF ULTRAFINE GRAINED STRUCTURE IN ALUMINIUM BY CGP METHOD

Received – Prispjelo: 2007-08-03
Accepted – Prihvaćeno: 2008-03-02
Original Scientific Paper – Izvorni znanstveni rad

The severe plastic deformation method known as constrained groove pressing (CGP) was used to produce ultrafine grained (UFG) structure in recrystallized aluminium (99.99%) at room temperature. The impact of repeated groove pressing, upon microstructure refinement was investigated by transmission electron microscopy (TEM) of thin foils. The changes in mechanical properties measured by tensile and by hardness tests were related to microstructure development. The yield stress and ultimate tensile strength reached a maximum after four passes. The loss of ductility was observed in all processed plates. Hardness values measured in different area of the deformed plates indicated strain heterogeneity distribution, regardless the high straining was maintained.

Key words: Constrained groove pressing, aluminium, severe plastic deformation, microstructure evolution, mechanical properties.

Nastanak ultrafine zrnate strukture u aluminiju sa ograničenim utornim prešanjem (OUP). Intenzivna plastična deformacija, metoda poznata kao ograničeno utorno prešanje (OUP) je rabljena za nastanak ultrafine zrnate (UFZ) strukture u rekristaliziranom aluminiju (99,9%) na sobnoj temperaturi. Utjecaj ponovljenog utornog prešanja na poboljšavanje mikrostrukture istraživano je transmisijskim elektronskim mikroskopom (TEM) na tankim folijama. Promjena mehaničkih svojstava izmjerom vlačne čvrstoće i tvrdoće pokazivalo je razvitak mikrostrukture. Granica razvlačenja i vlačna čvrstoća dosežu maksimum poslije četvrte provlake. Gubitak plastičnosti primjećen je kod svih tretiranih uzoraka. Mjerenja tvrdoće u različitim površinama deformiranih uzoraka pokazalo je heterogenu raspodjelu naprezanja.

Ključne riječi: Ograničeno utorno prešanje, aluminij, intenzivna plastična deformacija, razvitak mikrostukture, mehanička svojstva

INTRODUCTION

In 2001, Zhu et al. described an SPD method based on the repetitive corrugating and straightening known now as CGP [1,2]. This method comprises bending of a straight billet with corrugated tools and then restoring the straight shape of the slab with flat tools. The repetition of the process is required to obtain a large strain and desired structural changes. It has been shown that ultrafine grained structure can be introduced into metals and alloys using this method [3,4]. Metals with ultrafine grained (UFG) structure exhibit a high strength compared to materials with micrometer grain structures. The drawback of ultrafine grained structure materials is their limited ductility [5,6]. One of the approaches to enhancing ductility in UFG metals is explaining the change in the deformation mechanism in a ultrafine grained microstructure which is mainly characterized by high angle boundaries and a large volume fraction of non-equlibrium grain boundaries [7,8]. The deformation mechanisms of UFG metals is often considered to be governed not only by the activity of the lattice dislocations, but also by mechanisms such as grain rotation and grain boundary sliding [9]. Because of the complexity of the microstructure in materials processed by ECAP it is not clearly understood, whether and how these grain boundary mechanisms act and whether there is some beneficial contribution to the mechanical properties. From this point of view to study microstructure evolution and the character of the boundaries appears as very important role with respect to evaluate their influence on the mechanical properties. By repeating CGP process, a very large amount of plastic strain can be accumulated in the sample without changing the initial dimensions substantially. The method has been found as potential method for structure refinement in metals. Considering processing condition the heterogeneity in microstructure formation was observed across the bulk specimen in dependence of the strain introduced [10]. The effectiveness of four passes of successive groove and flat

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pressing was examined with the aim to study the microstructural evolution in dependence of executed CGP passes. The ultrafine grained structure development is discuss with respect to introduced effective strain and mechanical properties in tension are presented and discuss in term of an observed ductility as the number of CGP passes increases.

EXPERIMENTAL PROCEDURE

Commercial purity aluminium was supplied in cold rolled condition. Prior to CGP pressing, the plate was annealed at the temperature of 250°C for 1,5 h to obtain recrystallized structure. Aluminium plate with dimensions of 70×50×7 mm was then pressed with the CGP technique. The details of CGP process when the experimental material is pressed in asymmetrically positioned grooves and then straighten between a set of flat die is described elsewhere [1,3]. In this experiment four complete deformations of the plate were conducted. The effective strain, ε_{eff}, in the deformed region corresponding to one pass becomes about 1,16 throughout the sample [1]. By repeating the CGP process, a large amount of plastic strain can be accumulated in the workpiece without changing its initial dimensions. Total strain of ~ 4,64 finishing four passes was accumulated in plate.

The samples for transmission electron microscopy (TEM) study were selected from pressed plates after the first pressing from the top of the groove, which relates to a "undeformed" region (sample 1A), and from the inclined sheared region where resulting strain of ε_{eff} ~ 0,58 was achieved (sample 1B), Figure 1. The second pair of samples was cut off from the plate after first straightening matching the same position on corrugated plate as previous ones (samples 2A, 2B). When workpiece underwent the second pressing by grooved dies and flat dies, i.e. finishing one pass, a relatively homogeneous strain ε_{eff} of 1,16 regardless of the location was introduced. In order to check the effect of uniform straining, the third pair of samples for TEM analysis was selected from two different positions (3A and 3B) corresponding to sheared and flat region, as seen in Figure 1. By continuing pressing consequently more advanced substructure development was obtained after conducting two deformation cycles (ε_{eff} ~ 2.32), and four deformation cycles with total strain of ε_{eff} ~ 4.64. Thin foils were prepared by twinjet electro polishing technique using a solution of 6% perchloric acid in methanol. The microstructure of the pressed samples was examined by a JEOL 2000FX TEM microscope operating at 200 kV.

Tensile tests were performed at room temperature with hydraulic testing machine with multisensor strain gauge at a crosshead speed of 2 mm/min. The size of gauge part of the tensile specimen was 3 mm in diameter and gauge length was of 30 mm. Two tests were carried out for each selected condition.

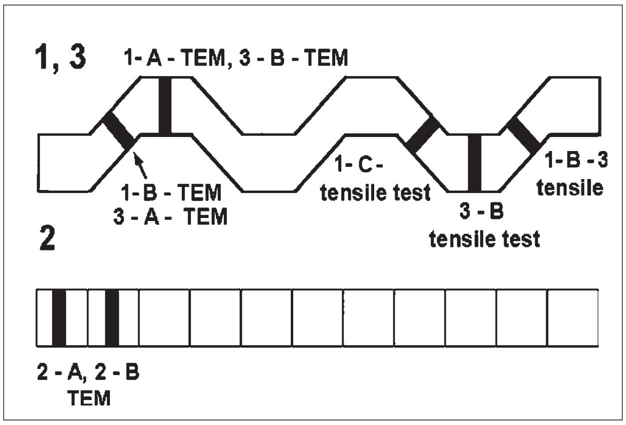

Figure 1. Schematic illustration of CGP plate for selection of TEM and tensile test specimens.

Hardness HV03 was measured on a Vickers hardness tester with a load of 300 g and loading period of 10 s. The hardness average value was the mean value of three measurements.

EXPERIMENTAL RESULTS AND DISCUSSION

Microstructure formation

Single pressing, as concerns "undeformed" flat top groove (1A), resulted in structure formation, consisting of elongated and/or equiaxed subgrains segmented by dislocation cells. The presence o subgrains and dislocation cells substructure is an evidence that the aluminium in this region has undergone also quite large amount of plastic deformation and it can not be denoted as an "undeformed" area. Detailed observation over wide areas of structure suggests that dislocation activities concerning the low angle subgrain boundaries formation were effective already in time of the first pressing.

The development of a deformed substructure in sheared grooved area subjected to single pressing (ε = 0,58) is illustrated in Figure 2. The microstructure con-

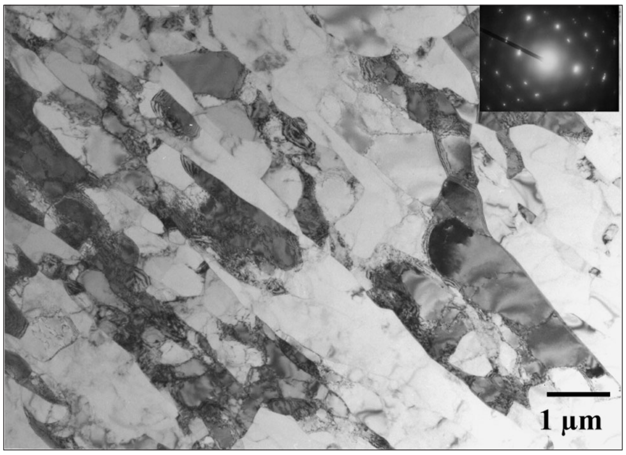

Figure 2. TEM micrograph of banded microstructure resulting from the first pressing in sheared region, (ε= 0,58).

sists of banded arrangement of elongated subgrains, which are segmented by dislocation cells. Dislocation substructure in this sample is more refined and the width of elongated subgrain is ~ 1μm and less. The small nuclei of polygonized grains were found inside of banded-like polygonized grains. Their appearance in structure of pure aluminium is probably resulting from the local dynamic recovery supported by local development of adiabatic heat at shear deformation. The SAED spot pattern indicates the crystallographic misorientation between subgrains is still very close, but spot diffusion and small streaks were evident.

The microstructure after first pressing and straightening of the plate (ε = 1,16) indicates more frequent recovery and polygonization in banded structure. The increased occurrence of equiaxed subgrains of size scattered around of 1- 2 μm have been observed. The average size of these new equiaxed grains with not yet fully polygonized boundaries is ~ 2-3 μm. However, formation of polygonized dislocation-free grains after small number of pressings is not common [5]. In this case, it is supposed, that polygonization process was intensified due to development of latent heat.

The microstructure resulting form the second groove-shaping, upon the plate was rotated 180°, formed on in sheared region is documented in Figure 3. The micrograph presents more finer subgrain structure, where former longer elongated subgrains are fragmented to smaller and more equiaxed subgrains. Inside this microstructure, new tiny nuclei of polygonized grains with size about 1 μm and less, nucleated along former subgrains were seen. These small polygonized grains are dislocation-free and are formed due ordered dislocations arrangement within subgrains. The resulted structure after second groove pressing is mixture which consists of former, now sectioned, elongated subgrains and polygonized subgrains.

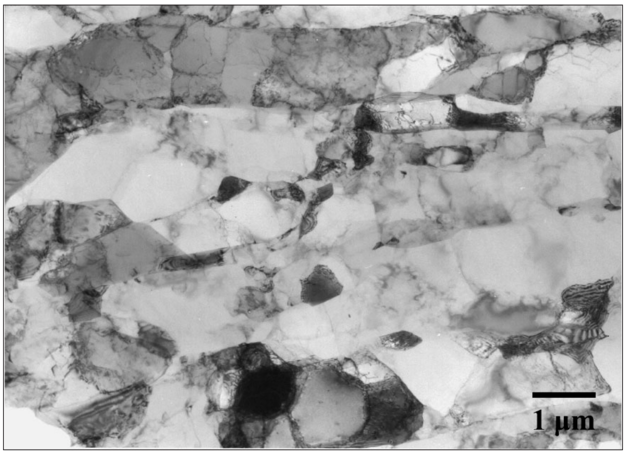

Figure 3. TEM micrograph of deformed microstructure resulting from the second shearing at the first pass (ε = 1,16 prior straigthening).

Completing the first deformation cycle by straightening pressing, the corresponding cumulative effective strain becomes ε_{ef} = 1,16 throughout the sample. The microstructure of deformed plate is shown in Figure 4. The elongated subgrain structure is severely banded due to the dominant shear stress. The fragmentation of subgrains is apparent as well. Inside of such deformed structure the formation of new equiaxed polygonized grains, depositied along the subgrains was observed. The dislocation structure recovery and formation of polygonized grains was observed as local process and is attributed to deformation heterogeneity distribution across the plate, which was detected at hardness measurement (see Table 1).

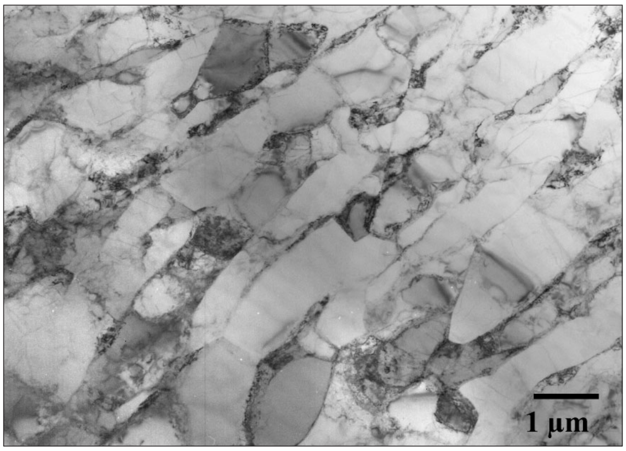

Figure 4. TEM micrograph of resulted structure completing the first pass, (ε = 1.16 after straightening).

In case of that more intense straining (ε_{ef} = 4,64) was imposed to plate after performing four passes the observed structure characteristics were similar to that with lower deformation, no matter that the total effective strain was four times larger. The structure is the mixture of severely deformed elongated subgrains and well developed equiaxed polygonized grains, which size is about ~ 1 μm, Figure 5. The presence of polygonized

Table 1. **Results of tensile tests and hardness measurement**

	RK	1A	1B	2A	2B	3A	3B	4 prs	8 prs	12 prs	16 prs
ε_{ef}	0	0	0.58	0	1.16	0.58	1.16	1.16	2.32	3.48	4.64
HV03 / MPa	25.1	21.7	36.7	19.8	30.9	32.7	32.2	37.6	37.8	37.7	31.3
0.2PS / MPa	50.1	-	106	-	-	100	100	104	127	122	124
UTS / MPa	59.3	-	110	-	-	105	111	105	128	127	131
A / %	4.2+	-	13.4	-	-	16	7.5	1+	3.5+	9.8	12.5
R of A / %	75*	-	70	-	-	69	61	63.6*	64.8*	65.8*	65.5*

+ failure aside extensometer * specimen not complete failure

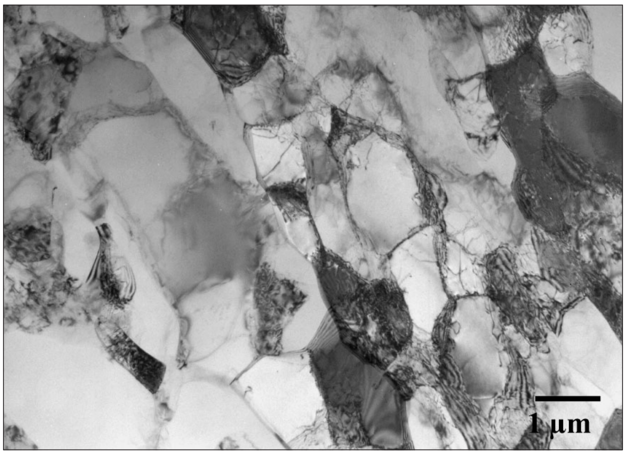

Figure 5. TEM micrograph of polygonized grains present in deformed structure experienced four passes.

grains was observed more frequently in deformed structure. The transformation process appears to be local and leads to grain coarsening.

Mechanical properties

The mechanical properties have been measured in the initial recrystallized aluminium and in the pressed plates by means of conventional tensile test and by hardness measurements. The locations of the tensile test specimens (3 mm diameter and 30 mm gauge length) in plates no. 1 (single stroke – corrugated shape) and 3 (three strokes – corrugated shape) and also locations of TEM specimens are shown in Figure 1. Hardness was measured in the locations of the TEM specimens. Tensile specimens, in case of higher straining was introduced, were cut off the plate without defining the location, because it was expected that uniform deformation was received over the plate after multiple pressing.

The values resulting from the tensile and hardness tests are shown in Table 1. There is a clear distinction between the values characterizing the initial state and the deformed plates subjected to CGP straining due different number of pressings. Generally, the strength measured in the initial annealed state is about 50 MPa and the corresponding hardness is 25 HV0,3. In the strained specimens subjected to CGP, the strength ranges from 101 to 109 MPa and the hardness from 31 to 37 HV0,3. The yield stress of strained specimens is twice higher as compared to recrystallized aluminium state. However, there are very small differences between the strengths and hardnesses of 1-B and 1-C tensile specimens (1 shearing deformation) and 3-A and 3-B specimens (3 and 2 shearing deformations, respectively). Considering the strength values, work of hardening and deformation behaviour there is not principal difference among the specimens subjected to different number of pressing. In order to clarify the CGP effect on grain refinement, transformation of subgrain boundaries to high angle boundaries, and appearance of small fraction of dynamically recrystallized grains, all these structural changes may substantially contributed to modification of plastic deformation behaviour of strained aluminium specimens, regarding the number of pressings. The ductility values point out that there was not observed the work hardening effect on deformation curve, and reaching the maximum stress value the continuous softening resulting from local necking sets in.

CONCLUSION

Severe plastic deformation by groove pressing of commercial purity Al at room temperature was investigated. The amount of straining resulted from applied passes is sufficient to refine the coarse recrystallized grains. The evolution of microstructure in deformed plate subjected to between 1 and 4 passes indicates formation banded subgrained structure with presence of dislocation cells in the subgrains. Significant refinement of microstructure is achieved in shear regions already after the first pressing. The structure characteristics after more intense straining ($\varepsilon_{eff} \sim 4.64$) were similar to that with lower straining. TEM observation revealed that mechanical fragmentation dominates at structure refinement due to the latent heat developed at straining, which supported the dynamic recovery and polygonization to sets in heavily deformed region.

The impact of the straining upon the mechanical properties was observed in tensile strength increase. The most pronounced hardness increase was measured after the first pressing.

Strain distribution heterogeneity was observed across the plate.

Acknowledgement – This paper contains results of investigation conducted as part of the MSM2631691901 project funded by the Ministry of Education of the Czech Republic.

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Note: The rensposible for English language is Author J. Zrnik.