STRUCTURE REFINEMENT BY SPD AND PROPERTIES RESPOND OF AA6082 ALUMINUM ALLOY

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Abstract

In this study ultrafine grain structure evolution during high pressure torsion of commercial aluminum alloy AA6082 at increased temperature is presented. Two different initial states of the alloy were prepared by thermal treatment. The progress in structure refinement in dependence on the shear strain level was investigated by TEM in thin foils. The impact of different amount of strain ε_{ef} was analyzed with respect to the effect of increased temperature. The results of microstructure analyses confirmed that ultrafine grain (UFG) structure was already formed in deformed disc upon the first turn, regardless the initial structure of alloy. The heterogeneity in UFG grain structure development across the deformed discs was observed in thin foils, supporting by micro hardness results scattering. By increasing strain value, applying different number of turns (N-2,4,6), more effectively homogenized UFG microstructure was observed across the deformed discs. The effect of increased deformation temperature became evident and dynamic recrystalization modified locally received UFG structure. The retardation of new grains growth and higher thermal stability of UFG structure was observed, when two steps thermal treatment of alloy, introducing quenching and ageing, was executed prior sample deformation. The strength measurement results, yielded from tensile tests, showed that the effect of structure strengthening was degraded by local recrystallization of deformed structure. The results received at torque measurements versus the time then showed that the torque required to deform the sample was increasing until the first turn and then kept stable or even decreased.

Keywords: Aluminum alloy, ultrafine grain structure (UFG), pressure torsion, torque, deformation behavior

1. INTRODUCTION

Severe plastic deformation (SPD) of metals and alloys can lead to grain refinement and finally to the formation of nanocrystalline structure. During the last two decades bulk nanostructure materials or materials with submicron structure, prepared by severe plastic deformation, have been investigated intensively. The production of fine grained metallic materials by SPD led to a large extent of investigation focusing on the microstructure development and related to mechanical properties. It is well known that SPD of metallic materials, involving different deformation processing ECAP, ARB, HPT and further deformation techniques is capable of producing ultrafine grained materials with sub-micrometer, or even with nanometer grain size [1, 2].

High pressure torsion is an effective deformation process of severe plastic deformation for producing ultrafine grained structure in metallic materials. Usually a sample for the HPT deformation is used in a form of disc, recently also the ring samples used for deformation [3]. Compared to other SPD processes the HPT offers a large number of advantages, as stated in [4]. At this deformation method shear strain is introduced in proportion to the distance from the disc centre, so that an inhomogeneous microstructure develops across the deformed disc. Among all available deformation methods HPT deformation represent a simple and effective method, which allows producing reproducible and well defined structure in samples. High applied pressure prevents fracture of sample and quantitative parameters of deformed sample response during torsion test can be compared with developed microstructure. The wide range of results, available for various materials deformed by HPT, confirmed saturation in structural refinement across the deformed discs and the strength and hardness

increase as strain increases [5, 6]. The results point out that there are differences in the development of microstructure among pure metal, multiphase steels and alloys [7, 8]. The improvements in the tool design led to a relatively well defined conditions of torsion deformation what resulted markedly in increase of HPT experimental deformation.

In this study, aluminum alloy AA6082 (EN AW6082), of two different initial structural states, was processed with HPT at increased temperature. The differences in fine structure development were analyzed with respect to different initial alloy structure, due different applied thermal treatment of alloy. Furthermore, microstructure evolution was examined and related to the hardness and torque in dependence of the effective strain applied.

2. EXPERIMENTAL PROCEDURE

A commercial aluminum alloy EN AW 6082 was supplied in as-cast rods with diameter of 20 mm. Prior HPT deformation two structural states of alloy were prepared by thermal treatment as follows:
S1 - annealing at 540°C/1.5 h followed by water cooling (solution + quenching applied);

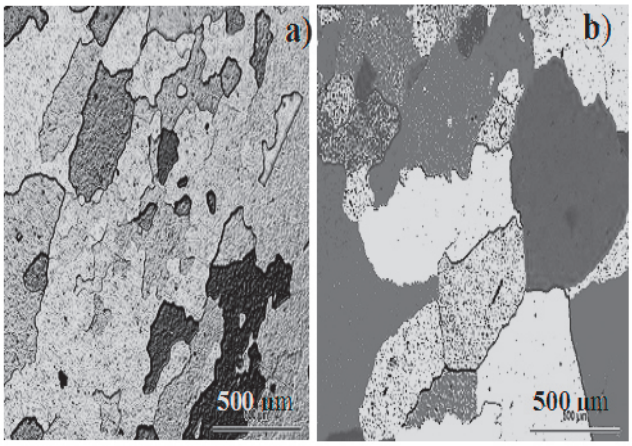

Fig. 1 Micrographs of initial microstructures of the alloy: a) as-cast; b) solutioning and ageing

S2 - S1 + ageing at 160°C for 12 hours; (precipitation hardened alloy);

The resulting microstructures prepared using thermal treatments S1 and S2 are presented in **Fig. 1**. The prior ageing treatment of alloy was to prepare different initial structure characteristics in alloy with respect to search a precipitation effect in alloy on structure refining. The presence of the secondary particles, was an aim to investigate the effect of these secondary particles β' phase precipitates (Mg_2Si) on deformed structure formation due to the presence of the secondary phases as precipitates in the alloy matrix. The initial microstructure in bulk resulting from casting process and thermal treatment, consisting of solutioning and ageing treatment is presented in **Fig. 1 a, b**. The as-cast structure modification was then apparent after rod forming (**Fig. 1a)**, but there were not observed a detectable changes in structure after solutioning a) and ageing b) treatment of the alloy prior ECAP deformation. The disc samples of 10 mm for HPT deformation were prepared from thermally treated rods (according S1 and S2 procedures) in **Fig. 1 a, b**. The as-cast structure was modified with aim to homogenize the alloy as-cast microstructure.

The samples for HPT were prepared from as-cast and thermally treated rods in form of discs with thickness of diameter of ~ 8 mm. HPT experiment was conducted using the deformation facilities able to produce high shear straining. The facilities consisted of upper and lower anvils having a shallow hole of 8 mm in diameter and 0.8 mm in depth at the centre. The discs were deformed by torsion up to 6 turns. Each sample was placed in the hole and the lower anvil was rotated with respect to the upper anvil at increased temperature of 350 °C with a rotation speed of 0.4 rpm under pressure of 4 GPa. The rotation was terminated after an N turns of 1, 2, 4 or 6. The equivalent Von Misses strain ε_{eq} as a function oh the number of turns N was calculated according to the relationship $N = \varepsilon_{eq} = 2\ \pi\ n\ r/\sqrt{3}$. The size effective strain ε_{eq} conducting N = 1, 2, 4 and 6 turns was ε_{eq} ~ 15, 30, 60 and 90. The discs were deformed by torsion up to 6 turns.

The changes in mechanical properties in relation to straining level (number of turns N performed) were

The changes in mechanical properties in relation to straining level (number of turns N performed) were determined by micro hardness across the deformed disc by static tensile tests using sub-size tensile pieces cut off aside the centre of the discs (in radial direction) and by in-situ measurement of the torque.

The disc samples subjected to HPT straining were ground using emery paper Grit P1200 and thus the Vickers micro hardness was measured at deformed discs edge (across) and in centre. The load of 50 g (HV10) was applied and the average value was taken from the 4 measurements approximately at the same distance from the centre of the disc.

Transmission electron microscopy (TEM) was used to evaluate the ultrafine grain microstructures evolution with respect to site on deformed discs and the number of turns at corresponding ε_{eq}. Discs with 3 mm in diameter were cut from the deformed disc at the centre and at its periphery. After disc thinning and polishing the TEM micrographs were obtained using JEOL JEM 2000FX operating at 200 kW. The purpose of the selected conditions was to evaluate the effect of difference in strain magnitude across the deformed disc at peripheral and axial position of the deformed disc, on ultrafine grain microstructure development.

3. RESULTS AND DISCUSSION

Investigated material, the commercial aluminum alloy AA6082, initially was of the coarse structure as documented in **Fig. 1**. Using HPT deformation method in order to refine a coarse steel structure to submicron size needs the application of large strain, usually with an equivalent strain more then 2. However, the strain heterogeneity across the deformed disc when using HPT deformation method, the larger straining (ε_{eq} - 6) is needed to obtain uniform UFG microstructure. Electron microstructure detailed analysis of thin foils prepared from deformed discs experienced different equivalent strain ε_{eq} revealed formation of various structural characteristics in dependence of strain used and selected localization on the deformed disc.

3.1. Microstructure analysis

In order to compare the magnitude of torsion straining at different position across the deformed disc the TEM micrographs in **Fig. 2** present microstructures of differently thermally treated alloy (state S1 and S2), which developed in deformed discs experienced torsion deformation resulting from the first turn (N1).

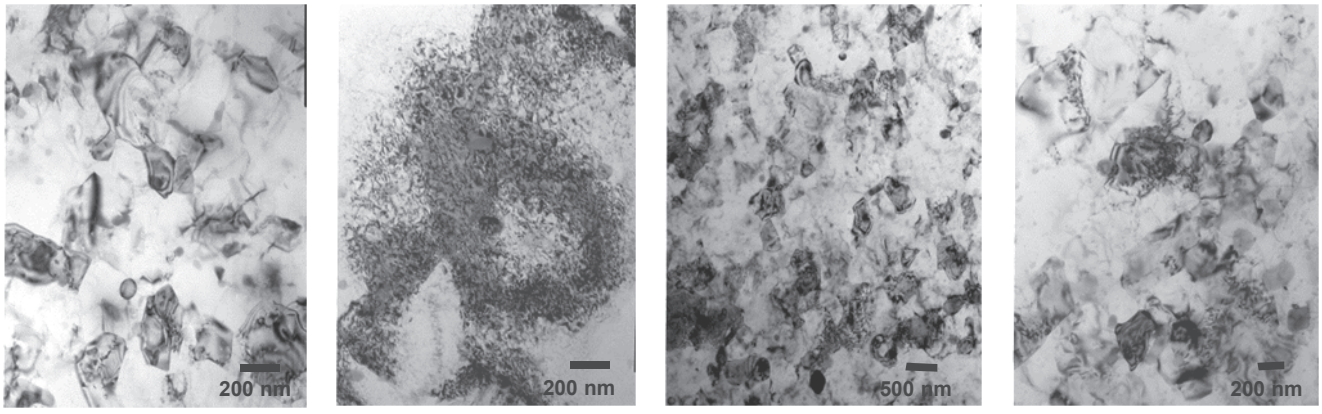

Fig. 2 TEM micrographs of HPT deformed structure developed at edge and centre of deformed discs. a) S1 edge; b) S1 centre; c) S2 edge; d) S2. The first turn (N1)

The set of individual figures represents the ultrafine grain structures and tangled dislocation network structure in deformed discs of both alloy states, which were developed at the edge and at the centre of deformed discs at temperature of 350°C. There is clear evidence that finishing the first turn (ε_{eq} ~ 15) the heterogeneity in development of a fine grain structure was still observed across the discs, no matter what thermal treatment of

alloy was applied. At disc periphery, the new grains having high angle boundaries were found applying the first turn and others. In the central area of the disc, tangled dislocation networks or partially recovered and rearranged dislocation network (almost sub grains), were found together with new grains. There were not observed evidences that increased temperature of deformation, by any way or another could modify or eventually supported growth of newly formed grains.

Conducting N = 6 turns ($\varepsilon_{eq} \sim 90$), the initial equiaxed coarse grained structure was found significantly modified across the deformed disc and ultrafine grained microstructure was formed and found almost across whole deformed discs, for both initial structure condition of alloy, as can be seen in **Fig. 3**.

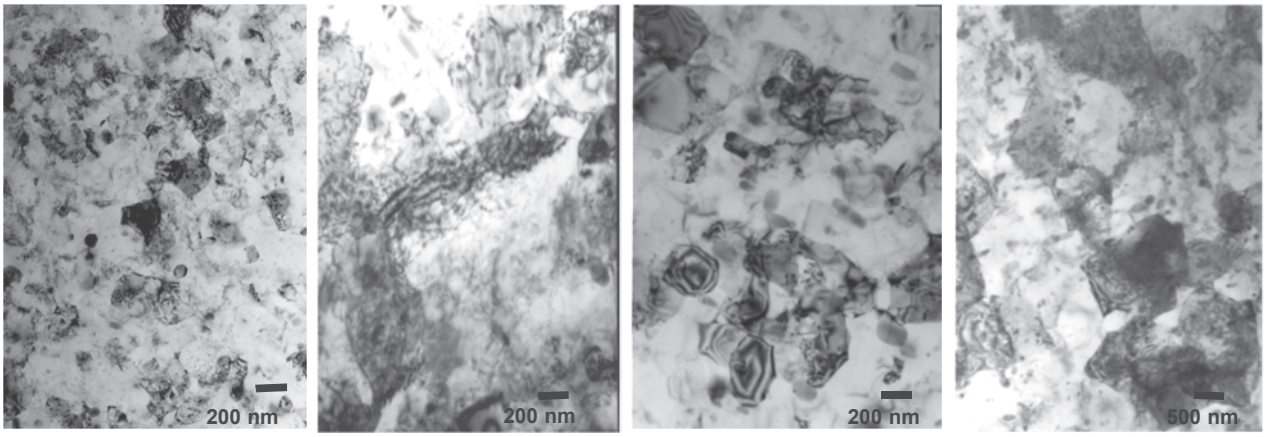

Fig. 3 TEM micrographs of HPT deformed which structure developed at edge and centre of deformed discs. a) S1 edge; b) S1 centre; c) S2 edge; d) S2. The sixth turn (N1)

The size of individual new grains were scattered over the wide region from 200 nm to 1 μm. Randomly, in central part of deformed disc, which was subjected to ageing for 12 h at temperature of 160 °C after solutioning and then quenched in water, the areas with partially recovered dislocation structure were found, as can be seen in **Fig. 3b**. Probably, this fact can be attributed to lower straining at the centre at the centre of the deformed disc, driving force for formation of the less deformed structure, due to lover driving force, resulted from straining at increased deformation temperature in this central area of disc.

3.2. Mechanical properties

In order to characterize the changes in mechanical properties with respect to applied large shear deformation and in dependence of applied straining, the various methods of testing were applied. Local Vickers microhardness data presented in **Table 1** were measured across the deformed discs. The tensile properties, using small tensile samples, were evaluated at room temperature. The torque, which was measured in time of loading, contains next to the torque necessary to deform the sample solely a contribution from region of the burr as well [7]. So this contribution to deformation process in some way then appears to be difficult for solely evaluation.

3.3. Hardness results

Microhardness development after different deformation exposures was the most available method to estimate the mechanical strength [8, 9]. In present work, this experimental exposure method was mainly used to determine the change of the strengthening across the deformed disc, (at the disc periphery and in the disc centre), after application of different strain ε_{eq} and compared with hardness of initial alloy. The measured Vickers hardness records (HV3) for both alloy states and for executed different number of turns is stated in **Table 1**. The hardness dependencies manifest different deformation behaviour of treated alloy in dependence of the initial state of alloy. The received results point out to an effect of softening, which was detected in

deformed discs after performing sixth turns in quenched state and after the second and more turns for quenched and aged state, regardless of the position on the disc, see **Table 1**. While for solution and followed by ageing treated alloy (S2) the gradual decrease of hardness was measured for higher number of turns that is for Nos. 2, 4 and 6. The effect of aging process included into alloy treatment procedure, with respect to structure modification due to increase an intermetallic precipitation, showed only negligible effect for structure modification. Considering this fact, the local dynamic recovery and recrystallization may contribute to structure softening. The resulted UFG grain structural characteristics at deformed disc edge supports this selective growth of fine grains, as can be seen in **Figs. 2** and **3**.

Table 1 HV hardness as the function of the turns and indent position (Q - quenching, Ag - ageing)

Alloy state	Turns *N*	DIT (mm)	DFT (mm)	HV3 L	HV3 C	HV3 R
S1 Q	N1	0.92	0.64	83	112	83
S1 Q	N2	0.94	0.65	83	111	82
S1 Q	N4	0.95	0.63	110	118	108
S1 Q+Ag	N6	0.95	0.61	102	110	102
S2 Q+Ag	N1	0.94	0.65	116	120	105
S2 Q+Ag	N2	0.94	0.65	105	113	105
S2 Q+Ag	N4	0.95	0.61	108	121	109
S2 Q+Ag	N6	0.95	0.63	101	112	101

3.4. Tensile deformation behaviour

In order to obtain mechanical strength values the small size specimens for tensile tests were machined out of HPT deformed samples, **Fig. 4**. Tensile deformation behavior of HPT samples was evaluated at room temperature and the data on load and elongation for all samples were recorded. The deformation results in form of load and elongation are documented in **Fig. 5 a, b**. As deformation results point out, it was observed difference in strength values with respect to higher stress applied for both structural states of alloy, as number of the turns was increasing. It means that reverse phenomenon, regarding the strength, was observed at tensile testing. The effect, however resulted in strength decrease. As the effective strain (number of turns) increased the strength values were decreasing for all exposed specimens, regardless the initial heat treatment this experimental alloy experienced. Evaluating the deformation results with respect to received deformed substructure, the reason of this "softening" was initiated by applied processing conditions (temperature and straining level) of deformation process. In deformed substructure, as structure characteristics show, not only ultrafine structure formation is evident but also recovery and recrystalization process contributed to modification of the final structure and locally to the growth of the initial fine grains especially on the pheripheral part of the deformed discs. The drop in the strength values and also appearance of short hardening period on deformation records (see **Figs. 5 a, 5 b**) in case of the highest straining (N4, N6 turns), is probably the support of recovery process.

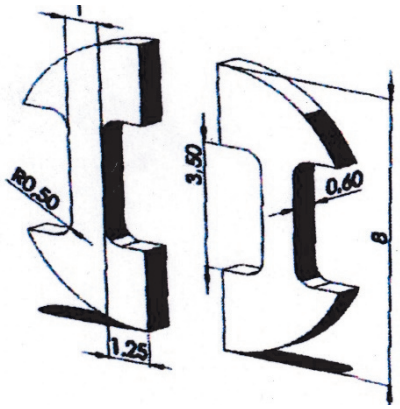

Fig. 4 The specimen for tensile testing

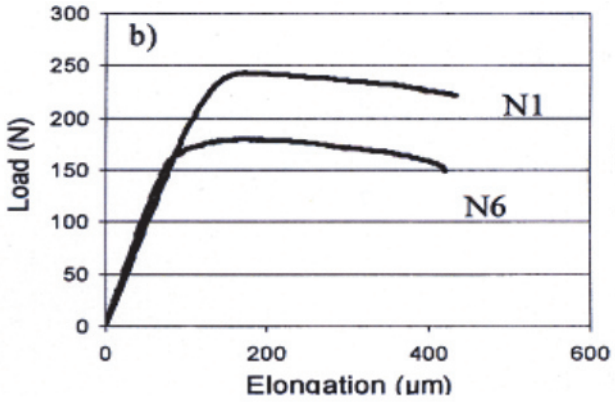

Fig. 5 a Tensile test records expressed in terms of load and elongation: a) S1 initial state

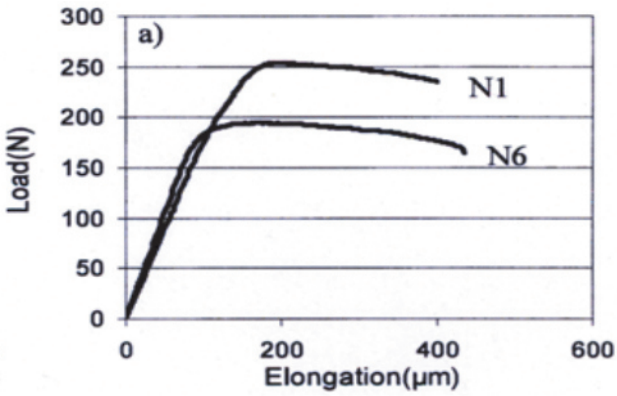

Fig. 5 b Tensile test records expressed in terms of load and elongation: a) S2 initial state

The softening, which appeared in both structural states of the alloy more or less deny the efficiency of ageing for alloy matrix strengthening by Mg_2Si (ß') precipitates, what was anticipated aim of íntroduced heat treatment schedule.

3.5. Torsion deformation

In order to characterize the changes in mechanical properties due to the large shear deformation (high pressure) and under constant nominal pressure of 4 GPA was performed. The deformation method is convenient to developed large shear deformation, which then can influences mechanical behaviour of severely deformed materials. The selected in-situ measured torque recorded at increased temperature of 350°C and for different strains ε_{eq} in dependence of turns number (N1 and N6) for quenched (a) quenched and aged (b) experimental alloy were performed. The results on torsion behaviour are presented in **Figs. 6** and **7**.

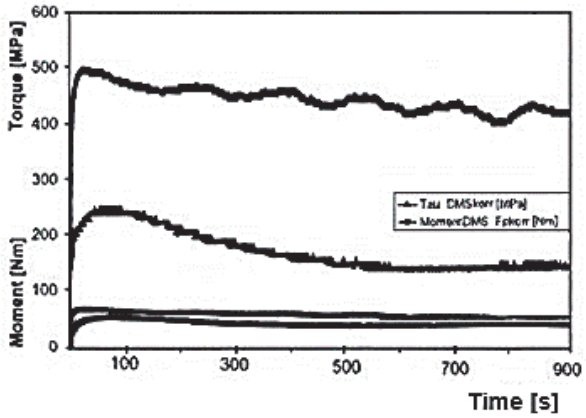

Fig. 6 In-situ measured torque curves experienced the first turn N1, N2, N4 and N6 turns for structural S2 alloy state

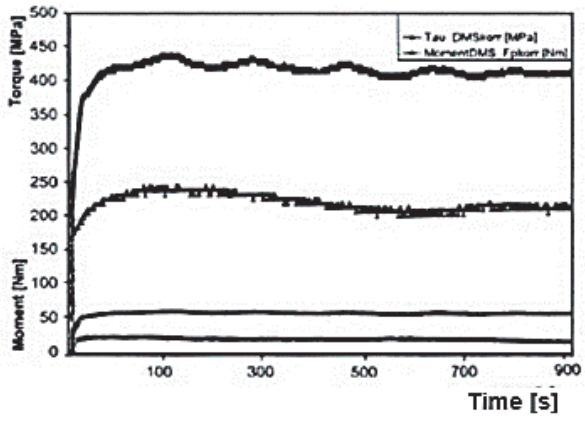

Fig. 7 In-situ measured torque curves experienced the first turn N1, N2, N4 and N6 turns for structural S1 alloy state

At the beginning of deformation a region of intense strain hardening was effective. Such specific behaviour resulted as strain was increasing as the number of turns was increasing, as well. In both structures it was accompanied by the drop of torque. A one of the reason behind of this softening appearance could be the progress in recrystalization process, probably due to applied quite high deformation temperature. Possible effect to make contribution to deformation behaviour appears to be a precipitation effect of Mg_2Si (β) particles due ageing, however this effect is controversial. Conducting N = 6 turns ($\varepsilon_{eq} \sim 90$), the initial equiaxed coarse

grained structure was found significantly modified across the deformed disc and ultrafine grained microstructure was formed and found almost across whole deformed discs, for both initial structure condition of alloy, as can be seen in **Fig. 3**. The sizes of individual new grains were scattered over the wide region from 200 nm to 1 μm. Randomly, in central part of deformed disc, which was subjected to ageing for 12 h at temperature of 160°C after solutioning and then quenched in water, the areas with partially recovered dislocation structure were found. as can be seen in **Fig. 3**. Probably, this fact can be attributed to lower straining at the centre of deformed disc, driving force for formation of the less deformed structure, resulted from straining at increased deformation temperature in this central area of disc.

4. CONCLUSIONS

The technology of severe plastic deformation was applied to aluminum alloy AA 6082 with aim to study the influence of large monotonic shear on formation of deformed microstructure development and mechanical properties respond. Two different initial Al alloy states, for high pressure torsion deformation technology were prepared to evaluate the effect of present secondary particles in alloy matrix, resulted from prior thermal treatment of the alloy. The idea to evaluate the presence of the secondary phases in alloy matrix on formation of ultra fine grained structure, have been realized. The results received from structural analyses and from mechanical testing showed some controversial deformation behaviour of the alloy, regardless the different initial state of the alloy. In refine deformed were not found the fine precipitates after severe deformation. On the other side the effect of softening was observed for the both initial states of the alloy, experienced different initial treatment. The effect of softening was found for both initial alloys states.

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