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Microstructure of ultrafine-grained Al produced by severe plastic deformation

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ABSTRACT

Purpose: The structure of Al subjected to severe plastic deformation by means of compression with oscillatory torsion and by combined method (compression followed by compression with oscillatory torsion) were investigated.

Design/methodology/approach: Al samples were deformed at torsion frequency (f) changed from 0 Hz (compression) to 1.6 Hz under a constant torsion angle (α) ≈6° and compression speed (v)=0.1mm/s. For combined methods the samples were compressed for strain ε= 0.7 and next deformed at mentioned parameters of compression with oscillatory torsion. Structural investigations were conducted by using light microscopy (LM) and transmission electron microscopy (TEM).

Findings: The structural analysis made by TEM shows that the processing by compression with oscillatory torsion ensures obtaining a structure (at selected parameters) with a mean grain size ≈ 1.6μm. Combined methods of deformation lead to grain refinement to about ≈ 0.9μm moreover material with uniform ultra-fine grained (UFG) microstructure was obtained.

Research limitations/implications: The understanding in refinement of Al structure could help to modify the process and design deformation parameters.

Practical implications: The knowledge of the characteristic features of unconventionally deformed materials will provide the usefulness of the employed method to produce materials having the desirable functional properties.

Originality/value: Oscillatory compression is a deformation procedure applied to achieve large strains. However there is no studies on evolution of the microstructures during deformation by using mentioned mode. This paper provides these information's.

Keywords: Nanomaterials; Al; Transmission Electron Microscopy; Severe plastic deformation

MATERIALS

1. Introduction

Significant grain refinement may be achieved in bulk polycrystalline metals through the application of severe plastic deformation (SPD) [1-7]. Several SPD processing techniques leads to microstructure refinement with various grain size and various contributions of high-angle boundaries [8-12]. Combined method for refining grain size equal channel angular pressing (ECAP) + cold rolling and ECAP + cold rolling + high pressure torsion (HPT) was employed for Ni, this processing allows to obtain equal grains of a sizes 330 nm and 120 nm respectively [13]. In [14] compression following to deformation by ECAP induces an increase of the most frequent grain size by about 10% and the size distribution gets much broader. Meanwhile combined torsion-compression following to ECAP yields a 50 % increase of the most frequent grain size whereas the width of size distributions does not change. In both cases the number of adjacent grains with large misorientations is higher as

compared to ECA deformation only. In [14] argued that compression can be used to break down an initial coarse structure, refine grain size, and increase the strength of the material. The experimental results present in [15] very clear demonstrate of the advantage of processing materials throught a combination of ECAP followed by HPT.
The aim of the present study was to determine changes in microstructure during methods of deformation as:

- compression
- compression with oscillatory torsion
- compression and next compression with oscillatory torsion (combined method).

For presented deformation methods, the analysis has been based on a detailed structural characterization by transmission electron microscope (TEM). By applying different deformation processes to the same metal (aluminum 99,7% purity), significant variations in microstructural parameters were obtained.

2. Experimental details

Commercial aluminum 99,7% purity was used. The material was homogenized at 500°C for 5h and then cooled slowly, and the grain size was about 100 μm. The bars were machined into cylinders of diameters 18 mm and length 36 mm for compression processing. Samples were compressed in room temperature for reach strain ε = 0.7. The next samples were compressed with oscillatory torsion. The samples were deformed at torsion frequency (f) changed from 0.4 Hz to 1.6 Hz under a constant torsion angle (α) ≈6° and compression speed (v)=0.1mm/s. The states of the samples are listed in Table 1.

Table. 1.
The states of the samples

Sample No	Processing
Sample 1	only compression ε= 0.7
Sample 2	0.4 Hz
Sample 3	1.6 Hz
Sample 4	Combined methods (ε= 0.7 + 0.4 Hz)
Sample 5	Combined methods (ε= 0.7 + 1.6 Hz)

Light microscopy (LM) and transmission electron microscope (TEM) were used to examine the microstructure. For optical microscopy, metallographically polished samples were electrolitically etched with a 5% aqueous solution of HBF_4. For TEM, longitudinal sections were taken from the 0.8 value of sample radius. Foils were prepared using conventional techniques by electropolishing using: 600 ml CH_3OH, 340 ml C_4H_9OH, 60 ml 60% $HClO_4$. TEM observations were carried out at 100 kV. Quantitative analyses were determined from TEM images. For quantitative analysis of grain size distribution more than 200 grains were measured for each specimen. Grain size is defined by the equivalent circle diameter. Hardness test were performed to characterize mechanical property by using Vickers Hardness testing ($HV_{0,05}$).

3. Results

The mean unit pressure (p) versus real reduction (ε_h) of cold compressed aluminum and compressed with oscillatory torsion (sample 3) is shown in fig.1. The samples were deformed for constant value of ε_h = 0.6. It is apparent, that the mean unit pressure (p) is about two twice smaller for compression with oscillatory torsion samples than compression only.

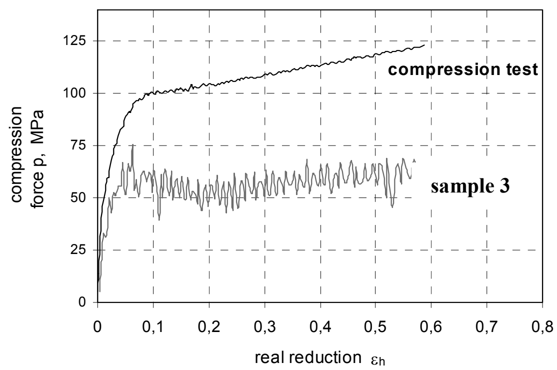

Fig. 1. Dependence of p= f (ε_h) for the investigated samples

It has been found that the mean unit pressure for sample 3 after initial increase, stabilizes at the level of p≈60 [MPa]. Comparable value of p for ε_h =0.6 was obtained for sample 5 (Fig.2), but the slope of p= f (ε_h) is different for combined methods. After initial increase of p≈ 90 [MPa], the (p) value shows stabilization for sample 4 on the same level p ≈ 90 [MPa] and rapid change in the slope of the curve in direction of smaller value of p for sample 5.

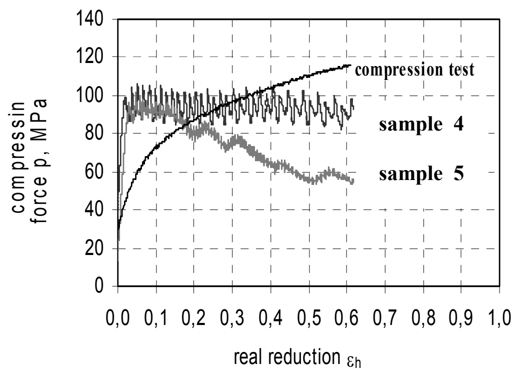

Fig. 2. Dependence of p= f (ε_h) for the investigated samples

A series of true stress (σ_p)- total equivalent strain (ε_i)- curves of Al tested in our experiment are shown in Fig. 3 and Fig. 4. After hardening the flow stress decreases with deformation (ε_i) for sample 4 and sample 5. The value of (ε_i) for sample 5 is about three twice higher than for sample 4. The observed difference may have its cause in the microstructure difference.

The evolution of microstructure processed by compression indicates the significant role of microbands and also such elements as Dense Dislocation Walls (DDWs). (More information about microstructure after compression at ε= 0.7 will be presented elsewhere). The subdivided microstructure by lamellar boundaries (LBs) is in contrast to the evolution towards an equiaxed subgrain structure observed in specimens deformed under a changing strain direction.

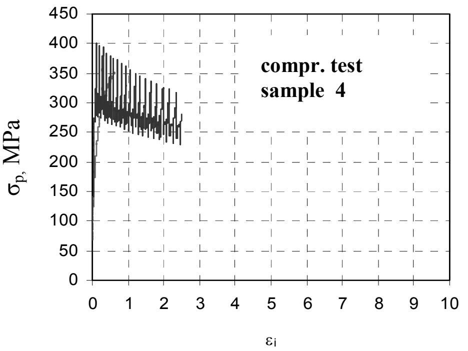

Fig. 3. Stress-strain curves obtained for Al under compression test and combined methods of compression and oscillatory compression at 0.4 Hz

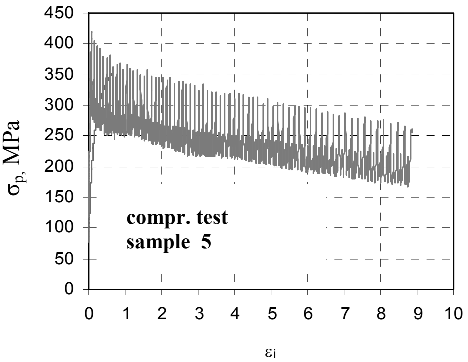

Fig. 4. Stress-strain curves obtained for Al under compression test and combined methods of compression and oscillatory compression at 1.6 Hz

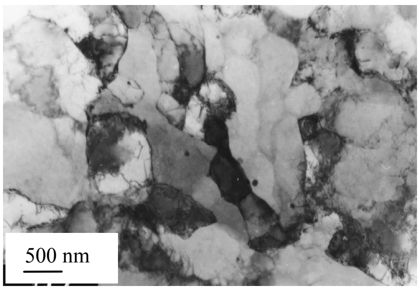

Fig. 5. The microstructure of sample 2

(Compression with oscillatory torsion is cyclic methods, i.e. the strain direction changes). The results of ill-defined boundaries after processing with low Hz (Fig.5) were typical with that reported in Cu [8,9]. The increase of Hz (relatively high strains) seems to be attributed to subgrain boundaries wall formation. After processing at high (1.6) Hz structure looks like a recovered containing many subgrain boundaries with dislocation inside subgrains (Fig.6). Figure 7 show a grains which has polygonized dislocation wall and grain boundary.

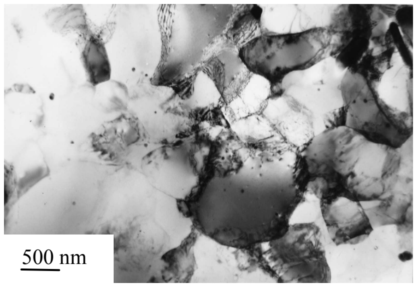

Fig. 6. The microstructure of sample 3

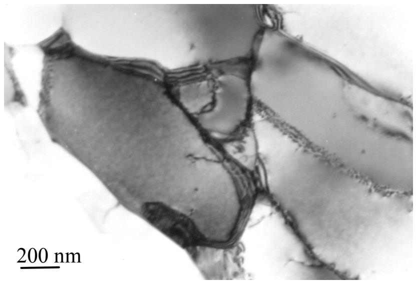

Fig. 7. The microstructure of sample 3. TEM micrograph showing a grain, which is surrounded by a polygonized dislocation wall and by grain boundaries

Compression with oscillatory torsion applied to compressed samples leads to formation of a structure with equiaxial grains below 1μm diameter (Fig.8).

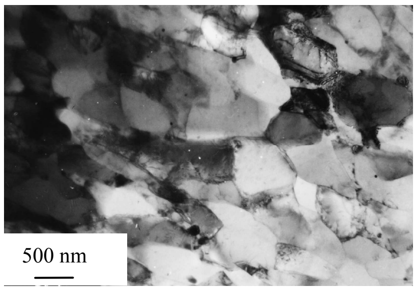

Fig. 8. The microstructure of sample 5

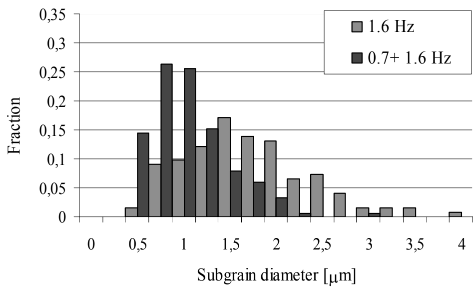

Fig. 9. Subgrain diameter distributions developed in samples 3 and 5

Presented on Fig. 9. distribution of the subgrain diameters deduced from longitudinal sections of samples 3 and 5 exhibit a more extended distribution of subgrain diameter for sample 3 than sample 5. It is mean that sample 5 is with more uniform microstructure compared to sample 3. The half of analyzed subgrains for sample 5 have a diameter in the range at 0.75 to 1μm. Meanwhile for sample 3, the 50 % contributions of subgrains is in range 1.25-1.75. It can be seen that severe plastic deformation through compression and next compression with oscillatory torsion results in ultrafine grained structure (Fig.10). The mean equivalent diameter decreases from about 1.6 μm for sample 3 to 0.9 μm for sample 5.

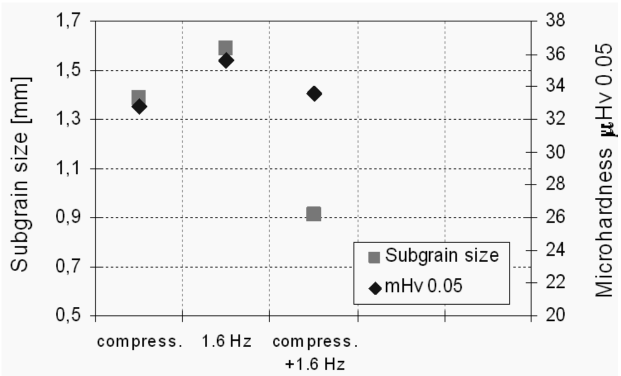

Fig. 10. Subgrain size and microhardness of Al after successive mode of deformation

Moreover, during severe plastic deformation, the microhardness do not changes particularly. The values of microhardness changes from about 32 $\mu HV_{0,05}$ to 35 $HV_{0,05}$ (Fig.10).

4. Conclusions

During deformation at low strain (low value of Hz), fine dislocation cells were formed by an operation of multi-directional slip. With increasing strain, dislocation cells were developed into ultrafine subgrains. At large strains (high value of Hz), the microstructural change was dominated by a conversion of low-angled subboundaries to high-angled boundaries, rather than grain refinement. Combined methods (compression followed by compression with oscillatory torsion yields specimens of uniform structure and controlled grains size below 1 μm. Reported research ought to be completed with grain boundary misorientation research.

Acknowledgements

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