Microstructure development in pure aluminium processed by constrained groove pressing

J. Drnek[1], J. Zrník[1,2]*, M. Cieslar[3], Z. Nový[1]

[1] *Comtes FHT Ltd., Lobezska E987, 326 00 Pilsen, Czech Republic*
[2] *University of West Bohemia, Universitni 22, 306 14 Pilsen, Czech Republic*
[3] *Faculty of Mathematics and Physics, Charles University, Ke Karlovu 3, 121 16 Prague 2, Czech Republic*

Received 4 July 2006, received in revised form 17 September 2006, accepted 18 September 2006

Abstract

Commercial purity aluminium (99.99 %) plates were processed with a severe plastic deformation technique – the Constrained Groove Pressing (CGP) at room temperature. It has been reported that using this technique, the coarse structure of pure aluminium can be refined to submicron ranges. The plates prior to CGP had a coarse-grained recrystallized structure with large scatter in grain size. Specimens were formed with two passes, each pass introducing the strain of about 0.58 in the deformed regions. The impact of the experimental forming upon microstructure was investigated with light microscopy and transmission electron microscopy of thin foils. Mechanical properties of selected specimens from different regions of the plates were measured by tensile tests. Hardness values (HV 0.3) measured in different segments of deformed specimens matched the strain distribution, and indicated some non-homogeneity in strain distribution. Development of microstructure analysed by transmission electron microscopy (TEM) proved formation of elongated grains with subgrain structure was found already after the first pass. The existence of grains with high angle boundaries already present in deformed structure was surprising and unexpected. The results show that the CGP with two passes resulted in formation of non-uniform microstructures with some new grains. Considering the results, a higher number of passes is required to obtain more advanced microstructure homogenization and grain refining.

Key words: pure aluminium, constrained groove pressing, severe plastic deformation, structure refinement, mechanical properties

1. Introduction

Constrained groove pressing (CGP) is a processing method, in which a metal is subjected to an intense plastic deformation through repeated dominant shearing and pressing (flattening) of plate. In 2001, Zhu et al. described an SPD method based on the repetitive corrugating and straightening (RCS) which is more known now as CGP [1–3]. 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 via severe plastic deformation. One of the possible techniques used recently is also CGP [4–6]. Using this method, the coarse grains in pure metals and alloys were successfully refined to the grain size of tens to a few hundreds of nanometers. The submicron grain materials showed very high strength compared to materials with micrometer grain structures. The drawback of ultrafine-grained structure materials is their elongation to fracture. Because of low strain hardening in submicron grain structure, the elongation is then dramatically decreased [7–9].

Unlike the widely used ECAP process for structure refinement, the CGP process has the advantage that severe plastic deformation can be applied to metal in sheet or plate form [10]. The groove pressing is carried

*Corresponding author: tel.: +00420 377 327427; fax: +00420 377 422224; e-mail address: jzrnik@comtesfht.cz

out so that the dimension of the gap between the upper die and lower die is the same as the sample thickness and therefore the inclined region of the sample is subjected to theoretically pure shear deformation under plane strain deformation condition. If dies are designed with the groove flank angle (θ) of 45°, a single pressing yields a shear strain of about 1 in the deformed region. This is equivalent to an effective true strain of 0.58. By repeating this process, a very large amount of plastic strain can be accumulated in the sample without changing its initial dimensions very substantially. The method has been found to have the potential to produce ultrafine structure in plate shape materials.

This investigation is an attempt to refine rather coarse grain structure of commercial purity aluminium using CGP technique. The effectiveness of two steps successive groove and flat pressing (corrugation process) was examined with the aim to study the effect of pressing condition on the microstructural development and mechanical behaviour of aluminium. The evolution processes of fine-grained structure, grain boundary formation and mechanical characteristics developed due to straining process were investigated.

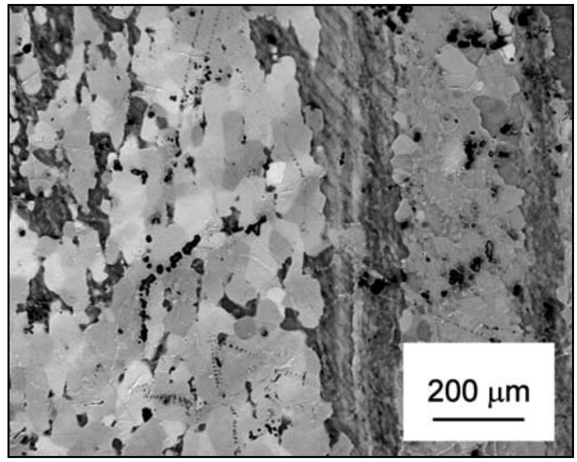

Fig. 1. LOM micrograph of initial recrystallized structure of aluminium.

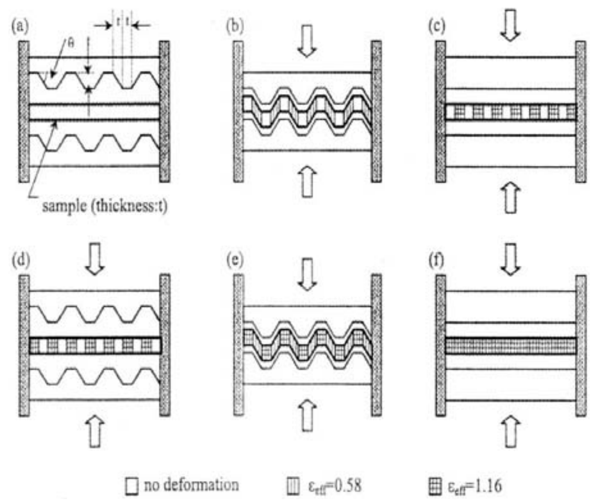

Fig. 2. A schematic illustration of CGP method [1].

2. Experimental procedures

Commercial purity aluminium was supplied in the form of cold-rolled 10 mm thick plate. Prior to CGP pressing, the plate was annealed at the temperature of 250 ℃ for 1.5 h in order to obtain the recrystallized structure. The light microscopy (LM) was used to analyse the structure characteristics. The microstructure, which developed in the recrystallized plate, is presented in Fig. 1. In the present study, a plate of aluminium with dimensions of 70 × 50 × 7 mm was pressed with the CGP technique. A schematic illustration of the CGP is presented in Fig. 2 [1]. In the first step of the groove pressing the plate is placed in the die and pressed between asymmetrically grooved dies. The deformed plate is then removed from the dies and pressed between a set of flat dies. In this time, the deformed regions are subjected to the reverse shear deformation while the undeformed region remains unchanged. After the second flat pressing the plate is rotated by 180° and pressed again using the grooved dies (the 3rd pressing). This ensures that the previously non-deformed region is strained by further pressing due to the asymmetry of the grooved die. The pressing is finished with deformation between the flat dies, restoring the original shape of the plate. In this experiment, only one complete deformation of the plate was conducted, which is equivalent to two pressing and two straightening steps. The cumulative (effective) strain, ε_{eff}, in the deformed region following the second pressing, and 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. All the passes were carried out using 100 tonne hydraulic pressing operating at a constant press speed of 15 mm$\cdot$ s^{-1} at room temperature.

Tensile tests were performed at ambient temperature using hydraulic Zwick testing machine with multisensor strain gauge at a crosshead speed of 2 mm/min. The size of gauge part of the tensile specimen after CGP pressing was 3 mm in diameter and gauge length was 30 mm. The gauge length was aligned along the longitudinal direction of the pressed plate. Two tests were carried out for each selected condition. Hardness was measured on a Vickers hardness tester with a load of 300 g and loading time of 10 s. The hardness average value was the mean value of five measurements.

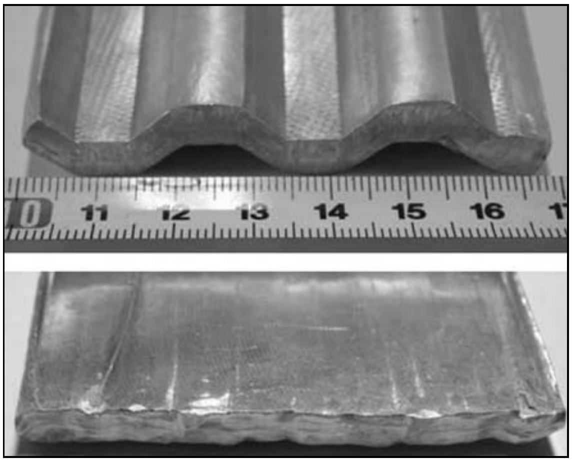

Fig. 3. The deformed plate shape after the pressing and straightening.

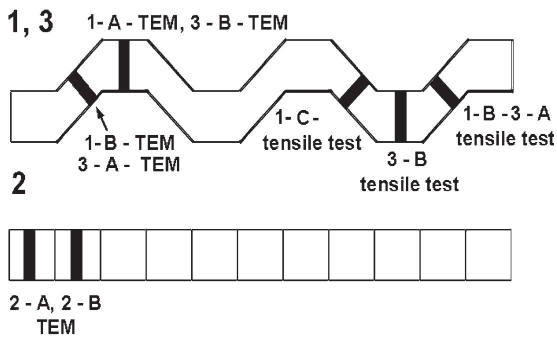

Fig. 4. Shapes of the 3 plate specimens and locations of TEM and tensile test samples.

The samples for TEM study were taken from pressed plates after the first pressing from the top of the groove, which relates to an "undeformed" (softly deformed) region (sample 1A), and from the inclined region (sample 1B) – see Fig. 4. The second pair of samples was cut off from the plate after first straightening. Sample selection was in coincidence with (matching the previous) positions on corrugated plate (sample 2A, sample 2B). When workpiece underwent the second pressing by grooved dies and flat dies, i.e. finishing one pass, it was introduced a relatively homogeneous effective strain of 1.16 regardless of the location within the flat sample. In order to check the effect of uniform straining, the third pair of samples was selected from two different positions corresponding to inclined and flat region prior to the second straightening. The sketch of thin plate's selection is documented in Fig. 4.

Thin foils were prepared from plates by abrasive grinding and mechanical polishing. Thin discs of 3 mm in diameter were twinjet electro-polished using a solution of 6% perchloric acid in methanol at an applied

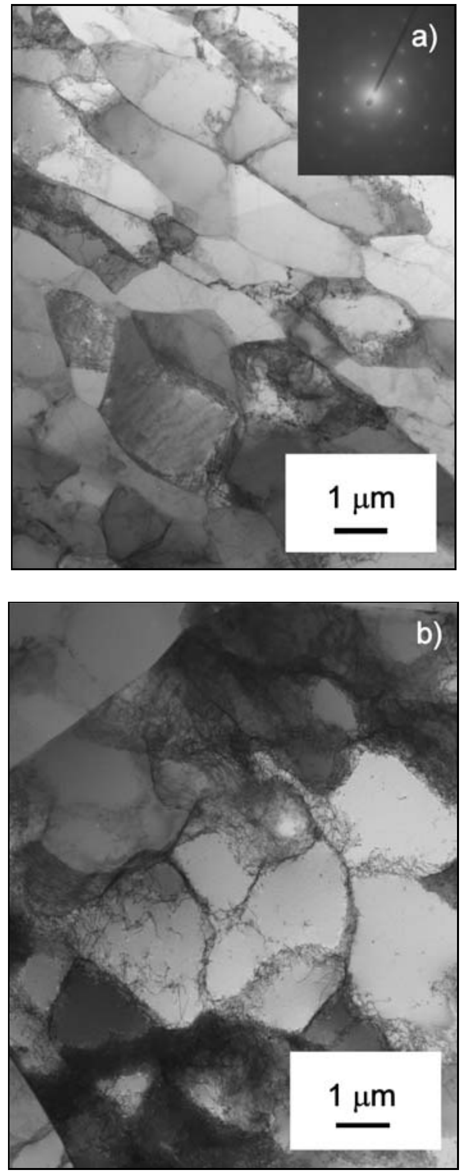

Fig. 5. TEM micrograph of subgrain structure (a) and dislocation cell structure (b) formed in the flat area of the grooved plate (segment 1-A-TEM).

potential of 15 V and a temperature of −15 ℃. The microstructure of the pressed samples was examined using a JEOL 2000FX transmission electron microscope operating at 200 kV.

3. Experimental results and discussion

3.1. Microstructure formation

After the single pressing, the microstructure has been investigated using the slices cut off from flat (1A) and sheared (1B) regions as marked in Fig. 4. Single pressing concerning "undeformed" flat top groove,

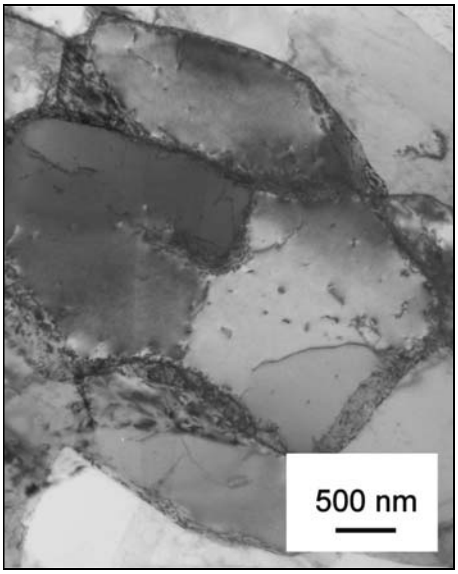

Fig. 6. TEM micrograph of high-angle grain boundaries formation on subgrains (1-B-TEM).

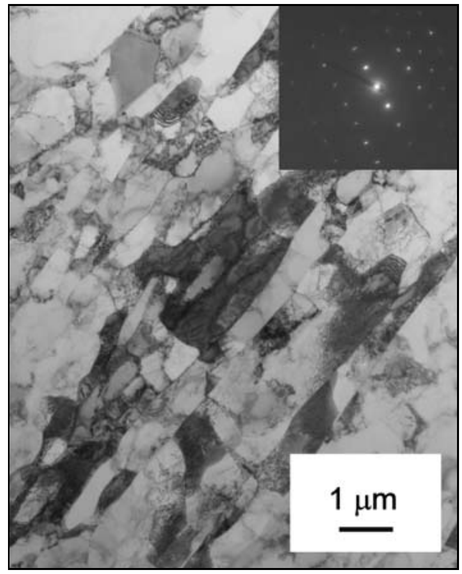

Fig. 7. TEM micrograph showing the subgrain formation in sheared region (1-B-TEM).

where strain ε should be zero, resulted in structure formation, consisting of elongated and/or equiaxed subgrains, and dislocation cells, as documented in Fig. 5. These micrographs evidence that the aluminium in this region has undergone also quite large amount of plastic deformation and it cannot be denoted as an "undeformed" area. Careful observation over wide areas of the sample suggests that dislocation activities (rearrangement on subgrain boundaries) concerning the low angle subgrain boundaries transformation towards to formation of high angle grain boundaries were effective and initialized already in time of the first pressing, Fig. 5b. The detail of such transition where more distinctive boundaries are apparent is evident in Fig. 6. The selected area electron diffraction (SAED) pattern, however, confirms that some of these boundaries are still having relatively low angle misorientation (Fig. 5a).

The development of a deformed substructure in grooved sample subjected to single pressing in region of effective shearing ($\varepsilon = 0.58$) is illustrated in Fig. 7. The microstructure consists of banded arrangement of elongated subgrains. Most subgrains are segmented by presence of dislocation cells or still dense dislocation network is formed within. Comparing the substructure development to the previous sample, dislocation substructure in this sample is more refined and the width of elongated subgrain is ~ 1 μm and less. When analysing deformed substructure the small nuclei of polygonized grains were observed inside of banded-like polygonized grains, as documented in Fig. 8. These newly formed polygonized grains are dislocation-free and with well defined boundaries recognized by fringed contrast, which is characteristic for formation of high angle grain boundaries in structure. We suppose that these high angle grains are not forming as result of grain fragmentation due to imposed severe plastic deformation. Their appearance in structure of pure aluminium is probably resulting from the local polygonization process and supported by local development of adiabatic heat generated by shear deformation, which probably increased a temperature in pure aluminium, as it was detected. The SAED spot pattern is clear at this stage, indicating the crystallographic misorientation between subgrains is still very close, but spot diffusion and small deviation can be observed.

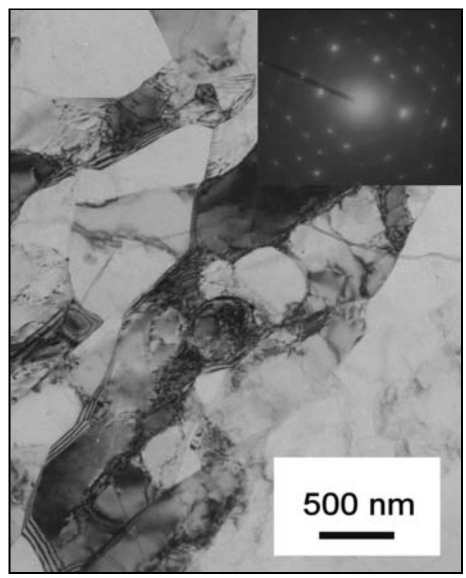

Fig. 8. TEM micrograph of polygonized structure (1-B-TEM specimen).

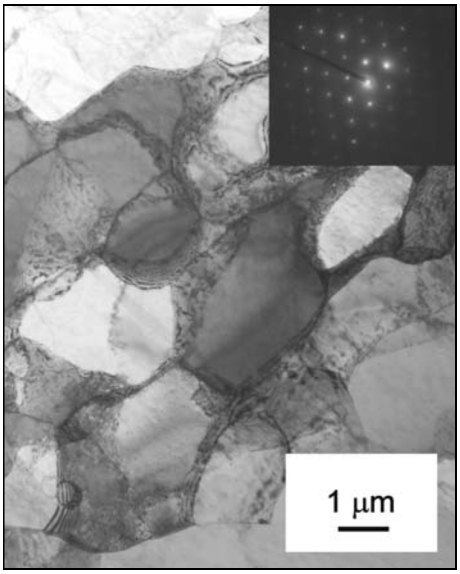

Fig. 9. TEM micrograph formation of new grains with high angle boundaries (2-B-TEM specimen).

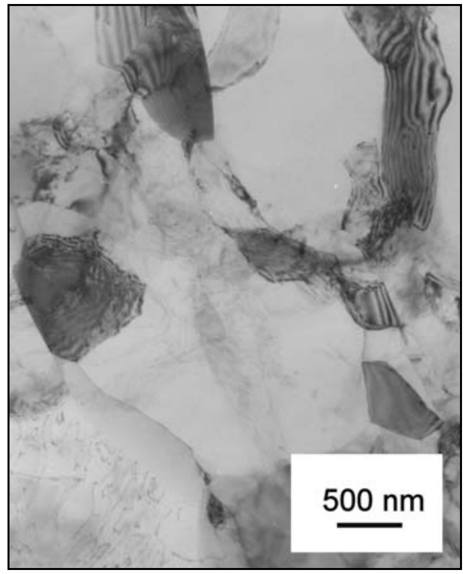

Fig. 11. New high angle grains formed on former subgrains (3-A-TEM).

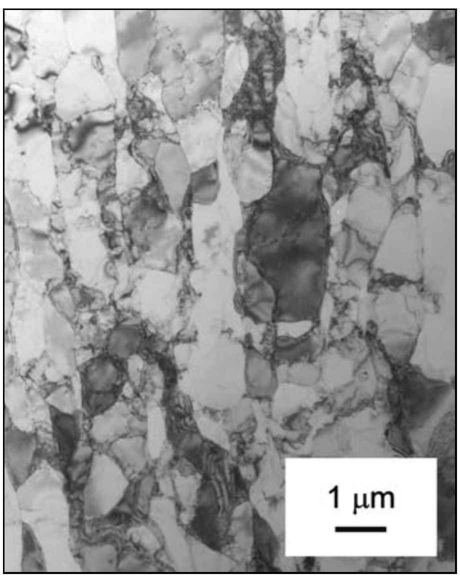

Fig. 10. TEM micrograph of subgrains microstructure after second pressing between grooved dies (3-A-TEM).

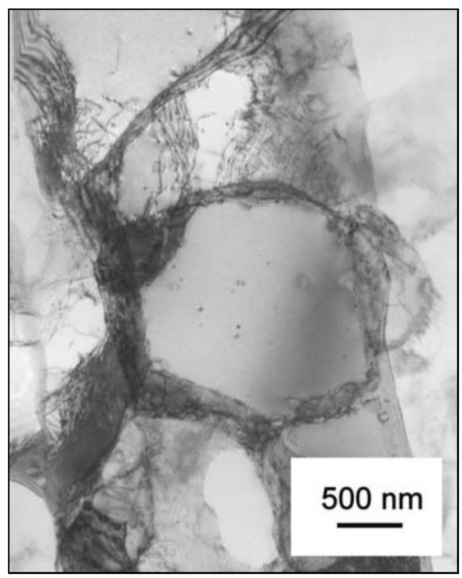

Fig. 12. Formation of new grains by dislocation rearrangement (3-A-TEM).

The microstructure after first pressing and straightening of the plate indicates more advance polygonization took place in deformed structure. The microstructure can be characterized by the presence of equiaxed subgrains of sizes scattered around of 1–2 μm. Dislocation density is low in subgrains and the most of them there are cleared out of dislocations. The characteristic microstructure is documented in Fig. 9 (specimen 2-B).

The formation of new grains with well-defined high angle grain boundaries morphology is in progress. The spot pattern of SAED indicates the crystallographic orientation of formed polygonized grains. The average size of these new equiaxed grains with not yet fully polygonized boundaries is $\sim$ 2–3 μm. However, formation of polygonized dislocation-free grains after small number of deformation passes is not common. Usually such polygonized structure is observed in aluminium during more intense plastic straining [11, 12]. In this case it is supposed, that polygonization process was intensified due to developed deformation heat.

The microstructure after second groove-shape

Table 1. Results of tensile tests

Specimen	0.2 proof stress (MPa)	UTS (MPa)	UTS (MPa) small	Ductility (%)	Reduction in area (%)	No. of shear deformations
1	50.09	59.26	–	4.2*	70.6^{+}	0
2	50.16	59.35	–	3.49*	75.3^{+}	0
1-B	106.50	110.39	111	13.4	59.5	1
1-C	107.7	109.11	–	13.2	59.3	1
3-A	100.99	104.81	105	16.1	68.6	2
3-B	109.47	111.14	–	7.5	60.9	2
2-A	–	–	52	–	–	0
2-B	–	–	111	–	–	2

* failure aside of extensometer, + not completely failured

formation, upon the plate was rotated 180°, formed on sheared region is documented in Fig. 10. The micrograph shows the more finer subgrain structure, where former longer elongated subgrains are fragmented to smaller more equiaxed subgrains. Inside this microstructure, new tiny nuclei of polygonized grains, with size about 1 μm and less, nucleated along former subgrains, as can be seen in Fig. 11. These small grains are dislocation-free and are formed due to ordered dislocations arrangement within subgrains as documented in Fig. 12. The resulting structure after second groove pressing is a mixture, which consists of former, now sectioned, elongated subgrains and polygonized grains.

3.2. Mechanical property testing

The mechanical properties have been measured in the initial recrystallized aluminium and in the pressed plates by means of conventional tensile testing, testing using small tensile specimens cut off from the deformed plates and by hardness measurements. The locations of the tensile test specimens (3 mm diameter and 30 mm gauge length) in plates no. 1 (single pressing – corrugated shape) and 3 (three pressings – corrugated shape) and also locations of TEM specimens are shown in Fig. 4. The small tensile specimens had a square cross-section of 2 by 2 mm and gauge length of 5 mm. Hardness was measured in the locations of the TEM specimens.

The values resulting from the tensile and hardness tests are shown in Tables 1, 2. There is a clear distinction between the values characterizing the initial state and the deformed regions of the CG pressed plates. Generally, the strength measured in the initial annealed state is about 50 MPa and the corresponding hardness is 25 HV 0.3. In the CG strained specimens subjected to CGP, the strength ranges from 101 to 109 MPa and the hardness from 31 to 37 HV 0.3. 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 spe-

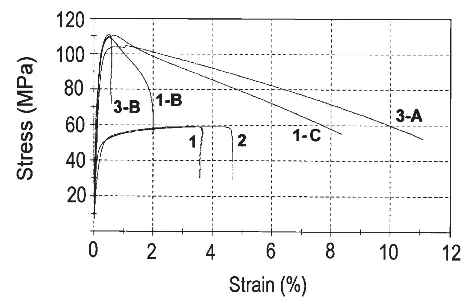

Fig. 13. Mechanical properties of specimens. No. 1 and 2 denote the initial as-annealed plate, whilst the remaining curves correspond to tensile specimens as shown in Fig. 4.

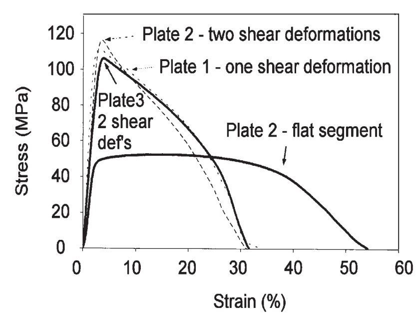

Fig. 14. Records of tensile tests performed with small specimens for different segments of plates.

Table 2. Results of hardness measurement tests

Specimen	HV 0.3	No. of shear deformations
1	25.1	0
2	–	0
1-B-TEM	36.7	1
1-A-TEM	21.7	0
2-A-TEM	19.8	0
2-B-TEM	30.9	2
3-A-TEM	32.7	2
3-B-TEM	32.2	2

cimens (1 shearing deformation) and 3-A and 3-B specimens (3 and 2 shearing deformations, respectively). Considering the strength values and deformation behaviour of conventional testing specimen size and small specimens they do not differ substantially, as shown in Figs. 13 and 14. 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 substantially contributed to modification of plastic deformation behaviour of strained aluminium specimens subjected to one and/or two passes. It could be observed there is not work hardening effect and after reaching the maximum stress value the continuous softening resulting from local necking sets in. The similar plastic behaviour was found in case of conventional tensile test specimens and small size specimens were used.

The difference in ductility shown in Fig. 13 for conventional tensile test and stated in Table 1 for specimens 1-B and 3-B is caused by the failure of specimens near the end of the gauge length, aside of extensometer. The ductility value was calculated from recorded tensile test machine crosshead movement.

4. Conclusions

A new set of tools for processing specimens with 7 mm thickness allowing conventional mechanical property testing was manufactured and used for initial trials. Three plate-shaped specimens were prepared for initial testing of the technology with 1, 2 and 3 pressings. The obtained microstructures showed rather small grain in all specimens, compared to initial coarse grain (several tens of micrometers). Recrystallization in this high-purity aluminium occurred in some higher-strain locations of specimens, which was due to high local straining and associated generation of deformation-related heat. The reason for similar strengths and hardnesses in locations, which were subjected to different total shear strains, may be seen in Figs. 5a, 6, 9, and 11, where similar grain structures are shown upon zero through 3 deformations. The impact of strain upon the mechanical properties at low number of passes is observed in tensile strength increase. However, even the "non-deformed" segments of specimens undergo certain, chiefly tensile-type, strain, as evidenced by numerical simulation [13]. The amount of strain resulted from pressing passes is sufficient to refine coarse recrystallized grains and latent heat originated at pressing supported the dynamic recrystallization process and formation of refined polygonized grains with distinctive high angle boundaries in areas of extensive shearing. Appearance of subcrystalline and partially polygonized microstructure resulted in continuous stress decrease after reaching the tensile yield strength. Greater refinement of aluminium microstructure and further increase in strengths is expected to occur with larger number of executed passes.

Acknowledgements

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

References

[1] SHIN, D. H.—PARK, J. J.—KIM, Y. S.—PARK, K. T.: Mater. Sci. Eng. A, *375*, 2002, p. 178.

[2] ZHU, Y. T.—LOWE, T. C.—JIANG, H.—HUANG, J.: U.S. Patent No. 6197129 B1, 2001.

[3] HUANG, J.—ZHU, Y. T.—JIANG, H.—LOWE, T. C.: Acta Mater., *49*, 2001, p. 1497.

[4] LEE, J. W.—PARK, J. J.: J. of Mater. Proces. Technol., *130–131*, 2002, p. 208.

[5] KRISHNAIAH, A.—UDAY CHAKKINGA—VENUGOPAL, P.: Scripta Mater., *52*, 2005, p. 1229.

[6] ZHU, Y. T.—JIANG, H.—HUANG, J. Y.—LOVE, T. C.: Mater. Trans A, *32*, 2001, p. 1559.

[7] JIA, D.—WANG, Y. M.—RAMESH, K. T.—MA, E.—ZHU, Y. T.—VALIEV, R. Z.: Appl. Phys. Letters, *79*, 2001, p. 611.

[8] WANG, Y. M.—MA, E.: Mater. Sci. Eng., *A375–377*, 2004, p. 46.

[9] PARK, K. T.—SHIN, D. H.: Metall. Mater. Trans., *33A*, 2002, p. 705.

[10] SHIN, D. H.—PARK, J. J.—KIM, Y. S.—PARK, K. T.: Mater. Sci. Eng. A, *328*, 2002, p. 8.

[11] YVAHASHI, Y.—HORITA, Z.—NEMOTO, M.—LANGDON, T. G.: Acta Mater., *46*, 1998, p. 3317.

[12] YAMASHITA, A.—YAMAGUCHI, D.—HORITA, Z.—LANGDON, T. G.: Mater. Sci. Eng., *A287*, 2000, p. 100.

[13] ZRNÍK, J. et al.: Course study report of research proposal MSM 2631691901 "Metallic materials with submicron and nanostructure prepared by methods of severe plastic deformation". Pilsen, Comtes FHT 2006.