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ANALYSIS OF MICROSTRUCTURE AND MECHANICAL PROPERTIES CHANGES IN AA1050 ALUMINUM SUBJECTED TO ECAP AND KoBo PROCESSES

ANALIZA ZMIAN MIKROSTRUKTURY I WŁASNOŚCI MECHANICZNYCH ALUMINIUM AA1050 PO PROCESIE ECAP I KoBo

Analysis of the results of the microstructure and the mechanical properties change in AA1050 aluminum alloy of technical purity processed using ECAP (Equal Channel Angular Pressing) and KoBo deformation methods are presented in the paper.. ECAP process was performed according to Bc scheme in the range from 1 up to 10 passes. Changes of microstructure were analyzed using scanning electrone microscope equipped with electron backscattered diffraction (EBSD) system. Microstructure and fraction of high-angle grain boundaries in KoBo processed samples were similar to those observed in ECAP processed samples after four passes. The most significant microstructure refinement was observed in ECAP processed sample submitted to 10 passes. In ECAP method the systematic increase of mechanical properties was observed along with increase of deformation degree.

Keywords: SPD, aluminium, ECAP, KoBo, microstructure, mechanical properties

W pracy przedstawiono wyniki analizy zmian mikrostruktury i właściwości mechanicznych aluminium technicznego z serii AA1050 po procesie ECAP (Equal Channel Angular Pressing) i KoBo. Proces ECAP prowadzono wg schematu Bc w zakresie od 1 do 10 przepustów Zmiany mikrostruktury analizowano za pomocą mikroskopu skaningowego z systemem do analizy dyfrakcji elektronów wstecznie rozproszonych (EBSD). Mikrostruktura oraz udział granic ziaren dużego kąta po procesie KoBo był zbliżony do uzyskanych po 4 cyklach ECAP. Największe rozdrobnienie mikrostruktury uzyskano w procesie ECAP po 10 przepustach. W metodzie ECAP obserwowano systematyczny wzrost własności mechanicznych wraz ze wzrostem stopnia odkształcenia.

1. Introduction

Methods of severe plastic deformation belong to techniques of intensive microstructure refining in polycrystaline materials [1]. Depending on selected technique and processed material it is feasible to obtain ultra-fine grained material (UFG) of grain size in the range from 1μm up to 100 nmor even nanometric – below 100nm. Obtaining the highly refined microstructure characterized with high-angle grain boundaries results in high mechanical properties accompanied with high ductility of given material according to the Hall-Petch relation [2]. ECAP technique is one of the best known and analyzed among the SPD techniques and allows to obtain materials of high refinement level of microstructure [3-7]. Nevertheless, constantly new attempts are being done by modifying this technique to achieve even better intensification of plastic deformation process and to obtain more efficient refinement of microstructure [8-10]. Worth noting is the fact that processes of severe plastic deformation may be applied using combination of the various methods. Few of the most relevant are high pressure torsion HPT [11, 12], twist extrusion TE [13, 14], accumulative roll-bonding ARB [15, 16], cyclic extrusion compression CEC [17]. Relatively new and promising one is a KoBo method. Its grave advantage is imposing accumulated plastic deformation under only one cycle [18-19]. This paper considers comparison of microstructure and mechanical properties change in technical pure aluminum alloy AA1050 subjected to ECAP and KoBo processes.

2. Experimental procedure

Commercial aluminum AA1050 of technical purity was utilized as working material. Chemical composition was confirmed using Spark spectrometer (Q4TASMAN, Bruker) and was presented in Table 1. In the beginning, samples

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underwent annealing process lasting 8 hours at 500°C then were cooled down together with furnace chamber. For ECAP processing samples of dimensions 10x10x70 mm were used. The ECAP angles were taken to be: Ψ = 20° and φ = 90°. Ψ angle represents outer arc of curvature where the two channels intersect while φ angle describes angle at which channel is bent. The samples were lubricated using graphite based lubricant. Process of deformation were carried out in room temperature with feeding rate of 10mm/min applying Bc scheme i.e. after each pass the sample was rotated of angle of 90° around length axis. Samples subjected from 1 up to 10 passes were obtained. In KoBo technique as precursor material of dimension 10mm and height 10mm were used. Extrusion process was performed in room temperature using extrusion rate of 0.1mm/s and torsion angle of matrix equal to 8° and frequency of 3Hz. Initial extrusion force was equal to 850kN and was successively lowered down to 550kN at the end of extrusion process. The KoBo process outcome was deformed rod-shape material of diameter of 4 mm.

TABLE 1

Chemical composition of the AA1050 (wt.%)

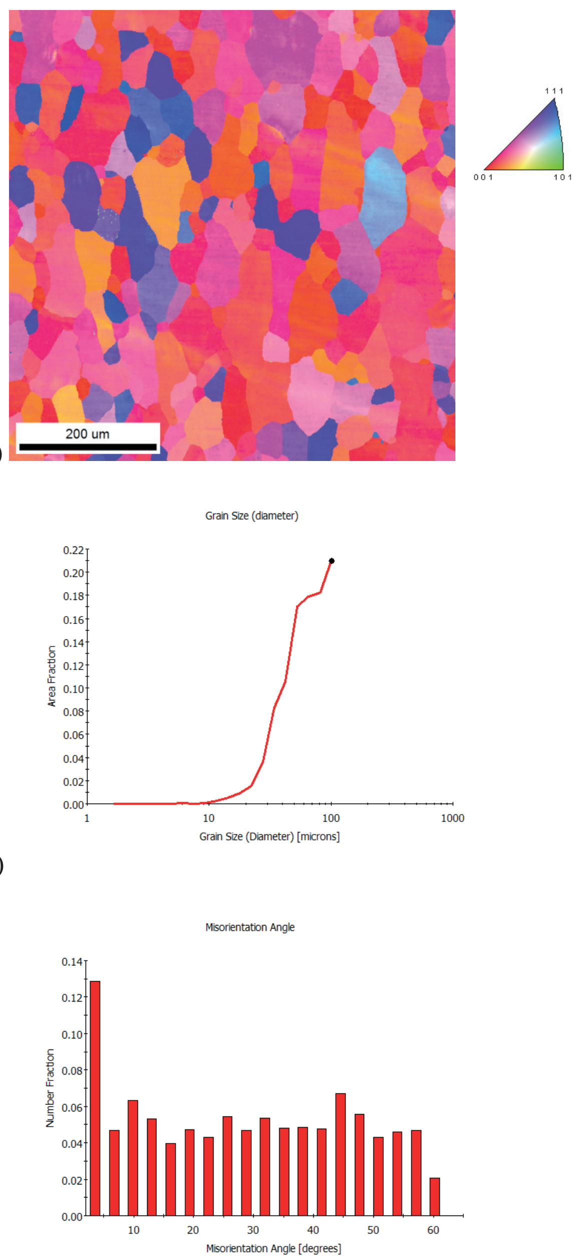
Al	Fe	Si	Zn	Cu	Mn	Mg	Ti
99,5	0,30	0,07	0,005	0,004	<0,002	0,008	0,008

Microhardness tests were performed using Innovatest 400 Series Model 423a microhardness tester using 4.9N of force. Hardness was tested on transverse cross-sections of samples. For each of ECAP processed material the series of 27 indentations were performed. At each cross-section indentations were aligned in three mutually and to sample walls parallel rows. In case of KoBo processed samples, five indentations were done on each sample's cross-section of diameter Φ = 4 mm. Static tensile strength was performed using tensile strength device MTS Criterion Model 43 applying head movement rate of 0,5 mm/min. For ECAP processed materials, tensile samples of diameter Φ = 4 mm and of length equal to Lo – 20mm were used. In each case two samples were tested. For KoBo processed materials tensile samples were of dimensions Φ = 3 mm and length Lo = 20 mm. EBSD measurements were performed using high-resolution SEM FEI Quanta 3D FEGSEM equipped with EDAX TSL EBSD system. Crystallographic orientation maps for ECAP after 2, 4 , 6, 8 and 10 passes and KoBo processed samples were done in planes perpendicular and parallel to extrusion direction. For orientation statistical analysis TSL OIM ANALYSIS were used with standard procedure for map cleaning and step size adequate to the microstructure refinement ranges from 1 μm to 100 nm.

3. Results

3.1. Microstructure

Microscopic observations were performed on both surfaces: parallel and perpendicular to extrusion direction. Annealed material was characterized with equiaxed microstructure of average grain size approx. 33,4 μm, Fig. 1. After second ECAP cycle one can observe strongly elongated structure of original grains which were fragmented partially. In relation to annealed sample the reduction of average grain size occurred. Additionally,increase of the fraction of low-angle grain boundaries was noticed. Primary grains structure disappears after four ECAP passes, Fig. 3. In consequent cycles one can notice systematic decrease of average grain size and relevant increase of fraction of high-angle grain boundaries, Fig. 4. In KoBo processed material the microstructure is strongly elongated in direction of extrusion, Fig. 5. In both planes longitudinal and transverse to extrusion direction, significant microstructure refinement was observed. Directions were described as: ED -Extrusion Directio, ND - Normal Direction, perpendicular to the upper face of the sample and TD - Transverse Direction, perpendicular to the side face of the sample).



a) b) c)

Fig. 1. Microstructure of the sample after the annealing process (tranverse to extrusion direction) : a) EBSD map of the microstructure, b) statistical variation of grain size, c) histogram of the variation of disorientation angle

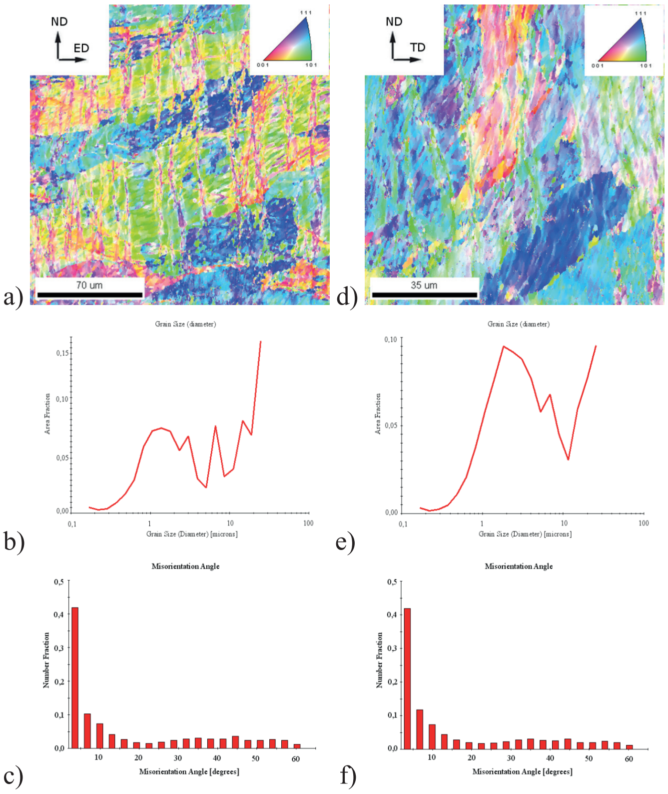


Fig. 2. Microstructure of the sample processed to two passes of ECAP: a, d) EBSD map of the microstructure, b, e) statistical variation of grain size, c, f) histogram of the variation of disorientation angle, a-c) transverse to the extrusion direction, d-f) normal to the extrusion direction

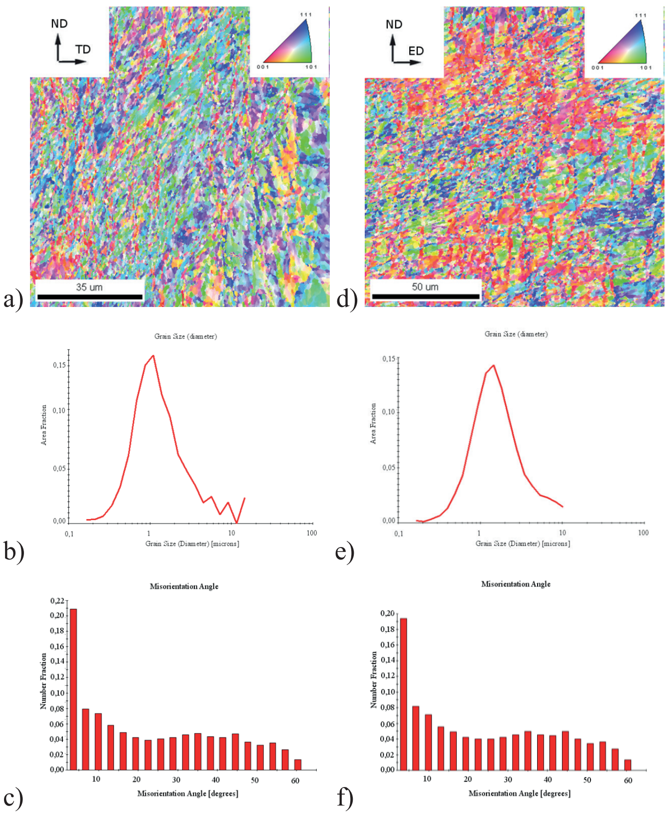


Fig. 3. Microstructure of the sample processed to four passes of ECAP, a, d) EBSD map of the microstructure, b, e) statistical variation of grain size, c, f) histogram of the variation of disorientation angle, a-c) transverse to the extrusion direction, d-f) normal to the extrusion direction

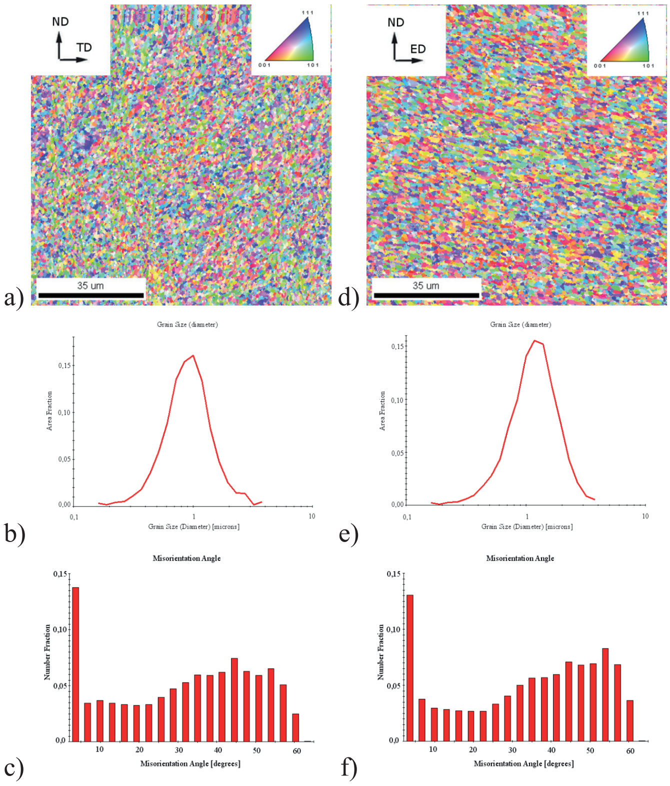


Fig. 4. Microstructure of the sample processed to ten passes of ECAP, a, d) EBSD map of the microstructure, b, e) statistical variation of grain size, c, f) histogram of the variation of misorientation angle, a-c) transverse to the extrusion direction, d-f) normal to the extrusion direction

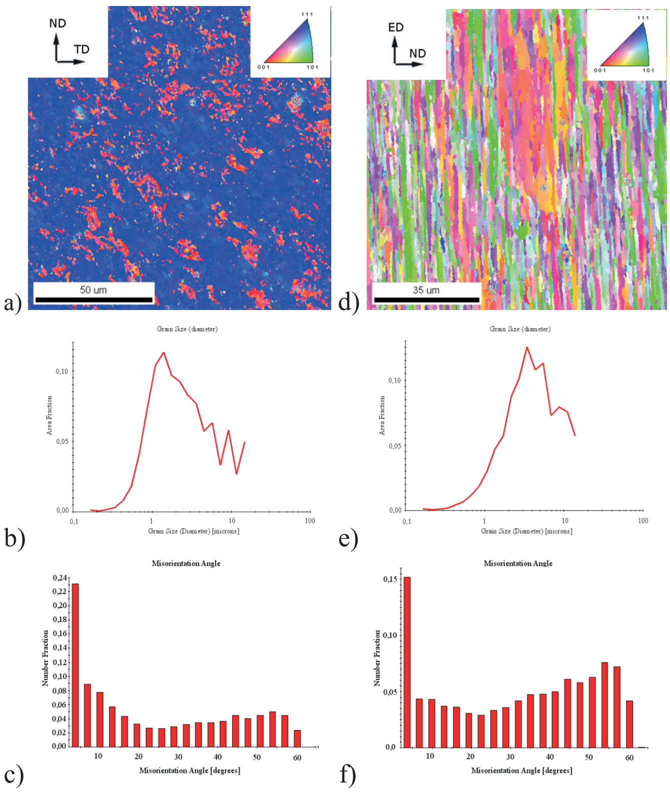


Fig. 5. Microstructure of the sample processed of KoBo a, d) EBSD map of the microstructure, b, e) statistical variation of grain size, c, f) histogram of the variation of disorientation angle, a-c) transverse to the extrusion direction, d-f) normal to the extrusion direction

Analysis of grains size change of ECAP process revealed systematic decrease of average grains size along with increasing ECAP passes in both planes: longitudinal and transverse to extrusion direction, Fig. 6. There were observed small differences in average grain size between the longitudinal and transverse directions. Relevant grains size reduction was observed after second ECAP pass. Original grain size equal to 65.9 µm in annealed sample underwent decrease down to level of 9.1 µm in transverse to extrusion direction and down to 7.6 µm in longitudinal to extrusion direction. Further increase in ECAP passes number resulted in slight but systematic refinement of grains size.. The greatest refinement was result of 10 consequent ECAP passes and was equal to 1 µm and 1.27 µm respectively in transverse and longitudinal to extrusion direction .

Significant grain refinement was obtained in the samples after the process KoBo. Average grains size was equal 3.73 µm in transverse and 5.07 µm in longitudinal to extrusion direction. These grains' sizes are close to those obtained in sample subjected to four ECAP passes.

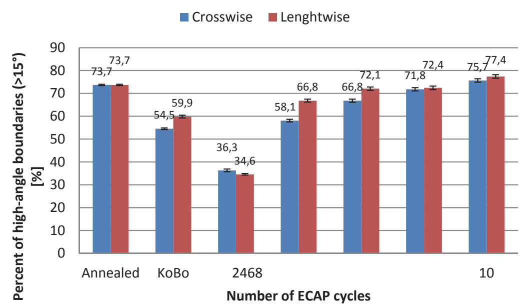


Fig 6. Variation of percent of high-angle boundaries of samples in the annealed condition and after KoBo and ECAP processing

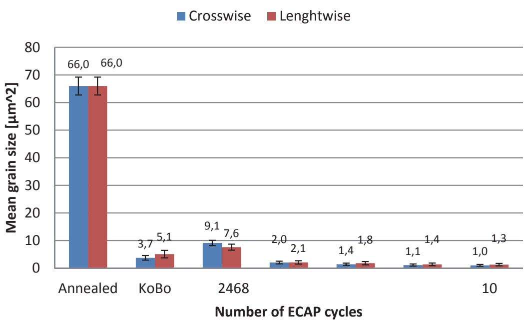


Fig 7. Variation of mean grain size of samples in the annealed condition and after KoBo and ECAP processing

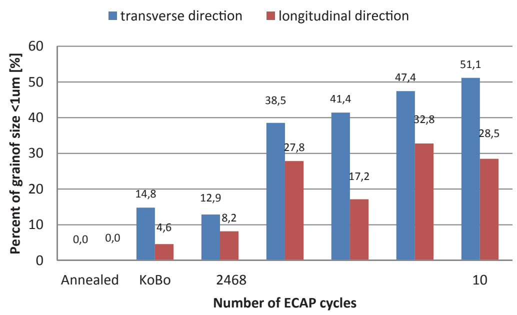


Fig 8. Variations of percent of grain size less than 1µm of samples in the annealed condition and after KoBo and ECAP processing

Along with decrease of average grain size one can observe increase of fraction of high-angle brain boundaries, Fig. 7. The highest fraction was achieved in samples processed 10 times using ECAP and it was equal to 75.66% in transverse and 77.40% in longitudinal plains to extrusion direction. For KoBo process this fraction was equal 54.52% in transverse direction and 72.43% in longitudinal direction. Fraction of grains of size below 1 µm in longitudinal direction changes irregularly The highest fraction of grains of size smaller than 1 µm was observed in sample ECAP processed 8-times and it was equal 32.78%. Different trend was observed when analyzing transverse direction, along with ECAP passes increase the increase of fine grains (<1 µm) fraction was observed. For sample processed with ECAP 10-times the fraction was equal to 51.14%. For KoBo technique worth noticing in low fraction of grains of size < 1 µm which was equal to 4,59% in longitudinal direction and 14,8% in transverse direction.

3.2. Mechanical properties

Microhardness of AA1050 alloy subjected to annealing process was fluctuated around 20 HV. After one ECAP pass the hardness increased significantly to level of 48.3 HV. The highest microhardness was achieved for sample subjected to four ECAP passes and was equal to 52.5 HV. Further increase of plastic deformation did not cause further increase of microhardness. Up to 10 ECAP passes any small fluctuation of microhardness was observed around the value of 51 HV, Fig. 9. In annealed state the material was characterized with low mechanical properties i.e. YS = 30 MPa, UTS = 59 MPa and very high elongation EL = 39.8%. After first ECAP pass resulted in dramatic rise of YS and UTS which were equal to 133 MPa and 141 MPa, respectively, at the expense of elongation which dropped down to 11.2%, Fig. 10 and Fig 11. Further increase of plastic deformation resulted in systematic increase of both mechanical properties and ductility. After 7 ECAP passes mechanical properties stabilize and do not change significantly anymore. The highest mechanical properties was observed for sample processed with ECAP 9-times and were equal to YS = 162 MPa, UTS = 181 MPa and EL = 20,9%. Investigated material subjected to KoBo process reached following mechanical properties: YS = 142 MPa, UTS = 162 MPa which was comparable with 4 passes of ECAP, however ductility was lower and equal to EL = 6.8 %.

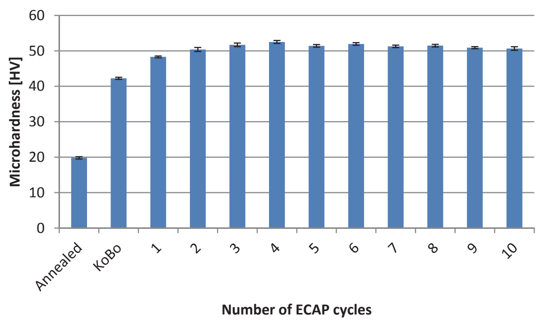


Fig 9. Vickers microhardness for samples in the annealed condition and after KoBo and ECAP processing

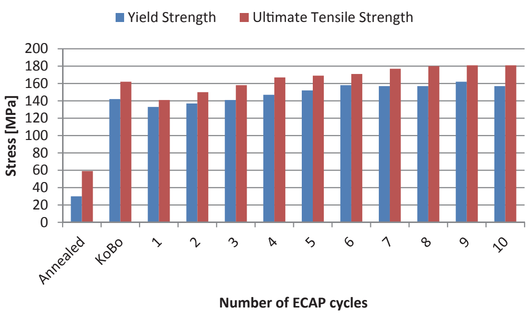


Fig. 10. Variation of yield strength and ultimate tensile strength for samples in the annealed condition and after KoBo and ECAP processing

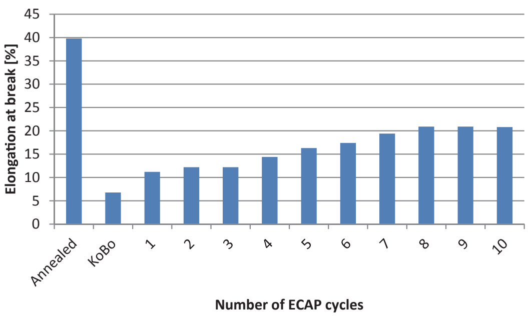


Fig. 11. Change of elongation for samples in the annealed condition and after KoBo and ECAP processing

4. Discussion

Obtained results for AA1050 subjected to ECAP process are similar to those presented in other papers [3] for round shaped samples. After the initial passes the fragmentation processes of primary grains were observed. Increase of dislocations density caused by severe plastic deformation leads to relevant rise of mechanical properties accompanied with drop of ductility in comparison to annealed material. After four passes of ECAP the primary structure disappears and grains become equiaxial. This is the consequence of dislocations density increase at dislocation tangles of cellular substructure what is characteristic feature for materials with high stacking fault energy. This effects in increase of disorientation angle in cellular substructure and in increase of mechanical properties. Observed systematic increase of strength results from grains refinement and from increase of fraction of high-angle grain boundaries. Microstructure refinement leads to increase of volumetric fraction of grain boundaries and therefore also to increase of number of barriers on dislocation way. Observed increase of ductility cannot be explained by microstructure refinement. It may be concluded that observed increase of ductility for ECAP processed samples over 4 passes may be caused by phenomena of dynamic recovery. As some investigations results suggest [8] during shearing process of sample passing through ECAP die the local increase of temperature may occur up to temperature of 50°C. This combined with high density of dislocations probably leads to activation of phenomena of structure recovery and to decrease of dislocations density. Also microhardness measurements results suggest such explanation due to their stabilization over fourth ECAP pass at level of approx 51 HV.

Interesting results were obtained for sample subjected to KoBo process. One KoBo cycle allow to achieve properties comparable to material subjected to 4 ECAP passes. It is evidence of much higher intensity of plastic deformation processes in material processed using KoBo technique. Average grain size was similar to grain size in ECAP processed sample subjected to four passes. Interesting is considerably lower elongation EL which is significantly lower comparing to ECAP. The main causes are probably strongly elongated microstructure in direction of material flowing which was not subjected to fragmentation as well as linked with it, low fraction of grains of size lower than 1 µm.

5. Conclusions

- Severe plastic deformation of samples using ECAP and KoBo methods leads to microstructure refinement and obtaining good combination of mechanical properties and ductility.
- Mechanical properties obtained in KoBo processed material are similar to values obtained in ECAP processed material subjected to 4 passes.
- Average grain size obtained in KoBo processed sample is comparable with value obtained in ECAP processed material subjected to 4 passes.
- Both ECAP and KoBo methods allows to achieve over fourfold increase of mechanical properties of AA1050 in comparison to annealed material.
- Systematic increase of ductility combined with stabilization of mechanical properties for higher degrees of deformation in ECAP method is most probably result of dynamic recovery which progress is linked with high accumulation of plastic deformation.

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REFERENCES

[1] R.Z. Valiev, T.G. Langdon, Prog. Mater. Sci. **51**, 881 (2006).
[2] R.Z. Valiev, R.K. Islamgaliev, I.V. Alexander: Prog. Mater. Sci. 45 (2000) 103–184.
[3] E.A. El-Danaf, M.S. Soliman, A.A. Almajid, M.M. El-Rayes, Mater. Sci. Eng. A 458 (2007) 226-234.
[4] V.M. Segal, Mater. Sci. Eng. A197, 157 (1995).
[5] B.Q. Han, D. Matejczyk, F. Zhou, Z. Zhang, C. Bampton, E.J. Lavernia, Metall. Mater. Trans. 35A (2004) 947–950.
[6] K. Bryła, J. Dutkiewicz, L.L. Rokhlin, L. Lityńska-Dobrzańska, K Mroczka, P. Kurtyka, Arch. Metall. Mater. 58.

(2013) 481-487,
[7] J. Kusnierz, J. Bogucka, Arch. Metall. Mater. 48, 173 (2003).
[8] R. Kocich, M. Greger, M. Kursa, I. Szurman, A. Machackova, Mater. Sci. Eng. A 527 (2010) 6386-6392.
[9] M. Kulczyk, J. Skiba, W. Pachla, Arch. Metall. Mater. 59 (2014) 163-165
[10] S. Rusz, L. Cizek, M. Salajka, S. Tylsar, J. Kedron, V. Michenka, T. Donic, E. Hadasik, M. Klos, Arch. Metall. Mater. **59**, 359-364 (2014).
[11] R.Z. Valiev, A.V. Sergueeva, A.K. Mukherjee, Scr. Mater. **49**, 669 (2003).
[12] Z. Horita, T.G. Langdon, Mater. Sci. Eng. **A 410-411**, 422 (2005).
[13] D. Orlov, Y. Beygelzimer, S. Synkov, V. Varyukhin, N. Tsuji, Z. Horita, Mater. Trans. 50 (2009) 96-100.
[14] Sh. Ranjbar Bahadori, S.A.A. Akbari Mousavi, Mater. Sci. Eng. A **528** (2011) 6527-6534.
[15] J. Bogucka, Arch. Metall. Mater. 59 (2014) 127-131
[16] J. Bogucka, H. Paul, M. Bieda, T. Baudin, Solid State Phenom, 186 (2012) 112-115.
[17] M. Richert, B. Leszczynska, Arch. Metall. Mater. 53, 721 (2008).
[18] W. Bochniak, K. Marszowski, A. Korbel, Journal of Materials Processing Technology **169**, 44-53 (2005).
[19] K. Sztwiertnia, J. Kawałko, M. Bieda, K. Berent, Arch. Metall. Mater. **58** 157-161 (2013).

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