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TEXTURE AND MICROSTRUCTURE OF PURE (6N) AND COMMERCIALLY PURE ALUMINUM AFTER DEFORMATION BY EXTRUSION WITH FORWARD-BACKWARD ROTATING DIE (KOBO)

Pure aluminium (6N) and commercially pure aluminium (99.7) was deformed by KOBO method. Microstructure and texture of both materials after deformation was analyzed by means of scanning and transmission electron microscopy. Advanced methods of crystallographic orientations measurements like Electron Backscatter Diffraction – EBSD (SEM) and microdiffraction (TEM) was used. Grain size distribution and misorientation between grains in cross and longitudinal sections of the samples were analyzed. Differences in size and homogeneity of the grains were observed in both materials. Pure aluminium was characterized by larger grain size in both sections of extruded material. Whereas commercially pure aluminium reveals smaller grain size and more homogeneous and stable microstructure.

Keywords: pure aluminium, KoBo, texture, EBSD, Orientation Microscopy

1. Introduction

Methods of severe plastic deformation (SPD) such as equal channel angular pressing ECAP [3,4] high pressure torsion HPT [5,6] or accumulative roll bonding ARB [7] are widely used in order to obtain improvement in mechanical properties by grain refinement [1,2] according to Hall-Petch strengthening mechanism. Advantage of these methods is maintaining unchanged initial shape and size. However most of typical SPD methods needs more than one step to obtain satisfactory results of grain refinement. Interesting technique providing also grain refinement only in one step is KoBo method. This method involves extrusion of the metallic sample in the forward-backward rotating die [8,9]. Rotation of the die during extrusion led to change in a stress state of the metallic material which provide better properties of materials [10] deformed by this technique. Because of five parameters which are used in this process it is very important to assure detailed microstructural characterization of materials after KoBo and combined it with obtained properties. Presented research are focused mainly on microstructural evolution in Al(6N) and AA1070 aluminium alloys processed by KoBo.

2. Experimental procedures

The investigated material was Al99,9999% and Al99,7% received as bars (initial diameter 35 mm and 40 mm respectively), after hot extrusion. In case of Al(6N) the initial microstructure was homogeneous and average grain size was about 35 μm, in AA1070 the initial microstructure was comprised of larger grain size with characteristic substructure (Fig.2). The bars were extruded to a rod with the diameter of 4mm by KOBO method. The Al(6N) was extruded at the rate of 0.1 mm/s, with the die oscillation by the angle of ±8° and the frequency of 1 Hz. The AA1070 was extruded at the same rate and angle of oscillation, but at the frequency of 5 Hz. Samples used for EBSD measurements were grinded on sandpapers, finishing on 5000 gradation, and polished on diamond paste, finishing on 1 μm. Furthermore, the surfaces of the samples were electropolished. Samples for TEM experiments were thinned by electro-polisher, using solution of nitric acid and methanol. The microstructure of the extruded specimens were researched in the longitudinal and transverse cross sections (Fig.1a,b) using Electron Backscatter Diffraction (EBSD) in the scanning electron microscope (SEM) FEI Quanta 3D FEGSEM, and microdiffraction in the transmission electron microscope(TEM) FEI Tecnai G20. The analysis of orientation maps was performed with the OIM ANALYSIS 6.2 software by TSL EDAX in SEM and TEM system developed in IMMS PAS and OML Skawina. From orientation maps were calculated parameters like grain size, misorientation distribution and texture. Texture was presented in the form of the pole figures (001),(111),(101) projected on the specimens planes (1-2) and (1-3) (Fig.1a).
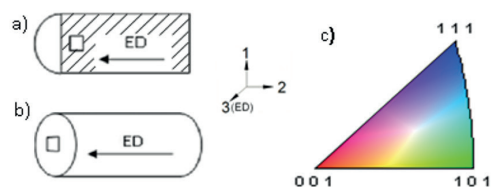

Fig. 1. Indication of sample preparation from longitudinal (1-3) a) and transverse (1-2) b) sections in the extruded billet with marked extrusion direction 3 (ED). c) Inverse pole figure (IPF) for the axis ED

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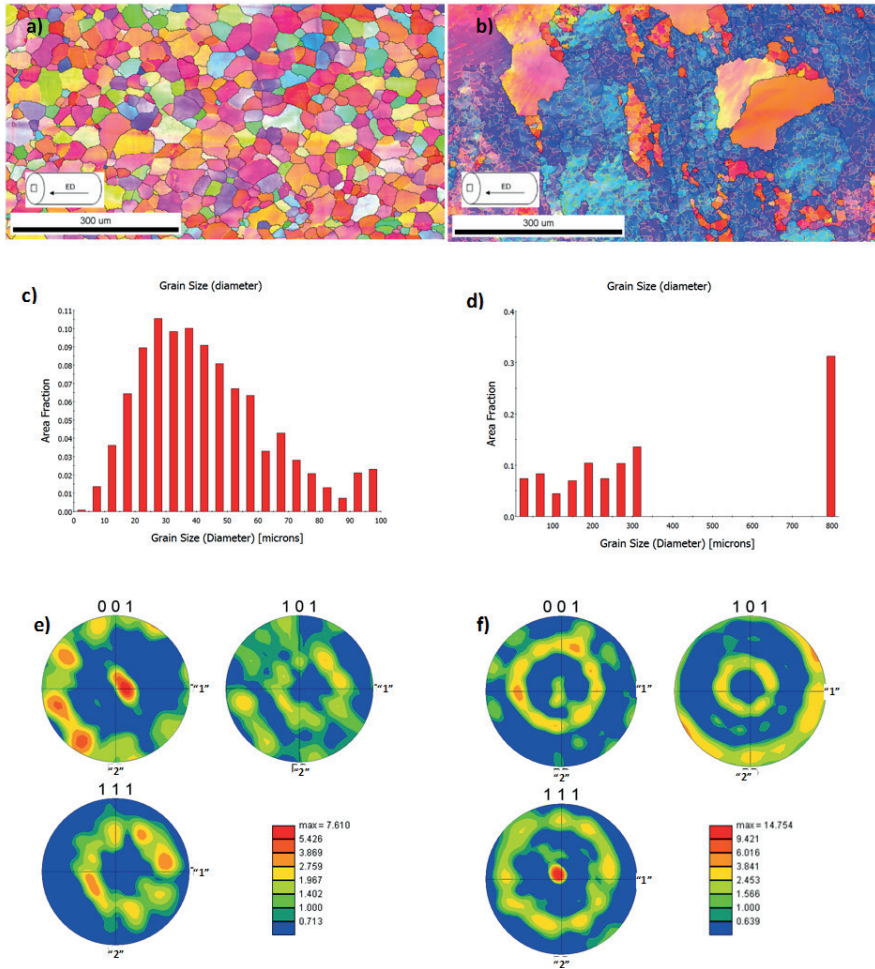
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Fig. 2. Orientation maps, grain size distribution (dark lines corresponds to high angle grain boundaries HAGB and yellow to low angle grain boundaries LAGB) and pole figures from cross section of the bars of Al(6N) (a, c, e) and AA1070 (b, d, f) initial for KoBo treatment
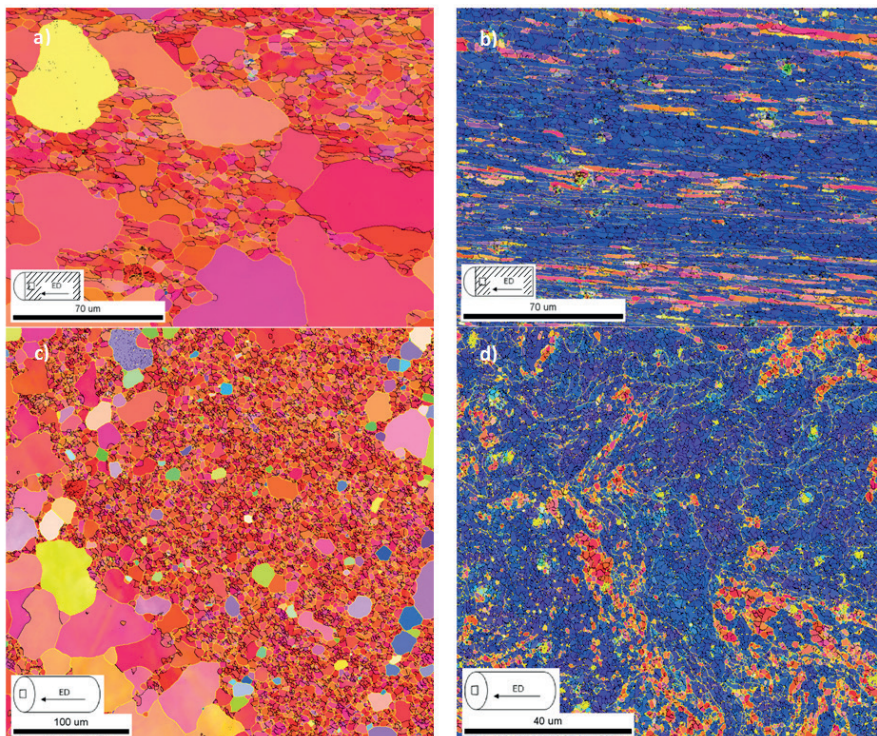

Fig. 3. Microstructure of Al(6N) (a, c) and AA1070 (b, d) after KoBo extrusion. Example EBSD maps in longitudinal cross-sections (a, b) and transverse (c, d) cross-sections. The images color corresponds to extrusion direction ED from inverse pole figure shown in Fig.1c. Dark lines corresponds to HAGB and yellow to LAGB
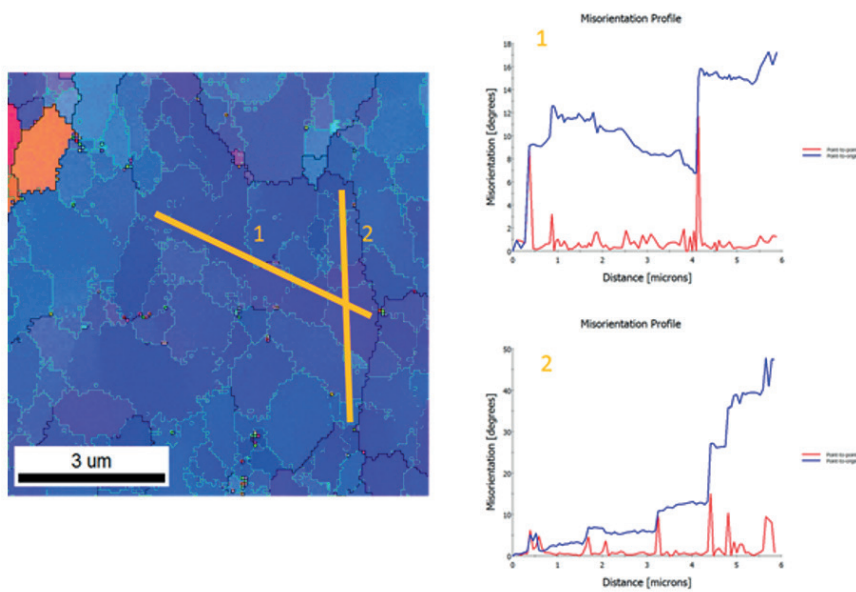
FiG. 4. Orientation map of AA1070 after KoBo extrusion in transverse cross-section with marked misorientation profile (1,2). The images color corresponds to extrusion direction ED from inverse pole figure shown in Fig.1c. Dark lines corresponds to HAGB and yellow to LAGB
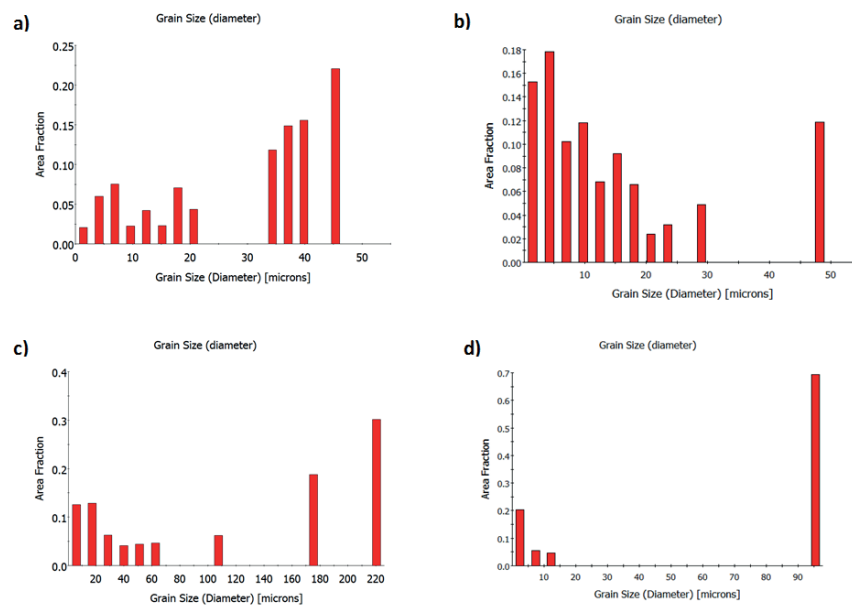

Fig. 5. Grain size (diameter) in function of area fraction for Al(6N) (a,c) and AA1070 (b,d) measured from longitudinal cross-sections (a, b) and transverse (c, d) cross-sections
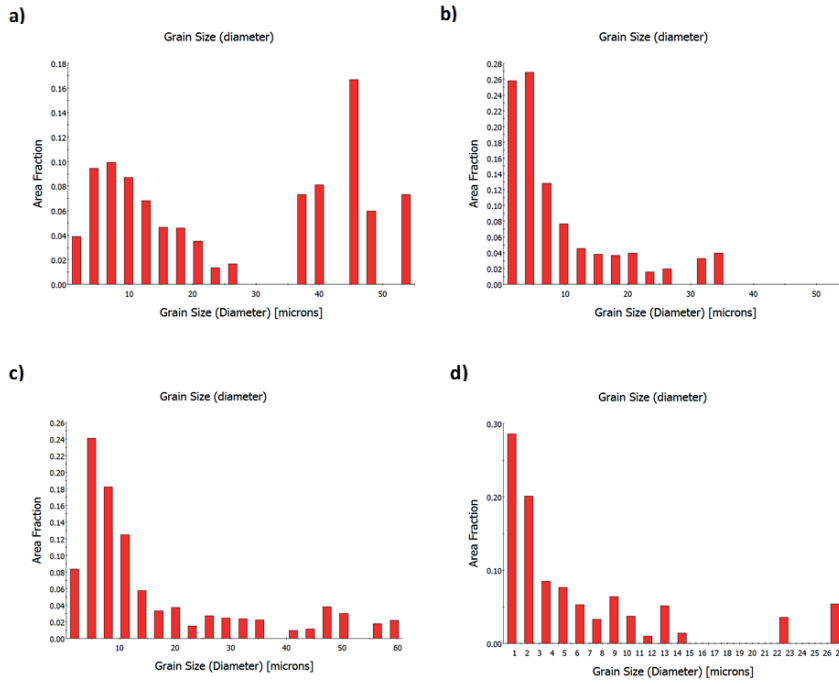
Fig. 6. Subgrain size distribution(diameter) in function of area fraction for Al(6N) (a,c) and AA1070 (b,d) measured from longitudinal cross-sections (a, b) and transverse (c, d) cross-sections
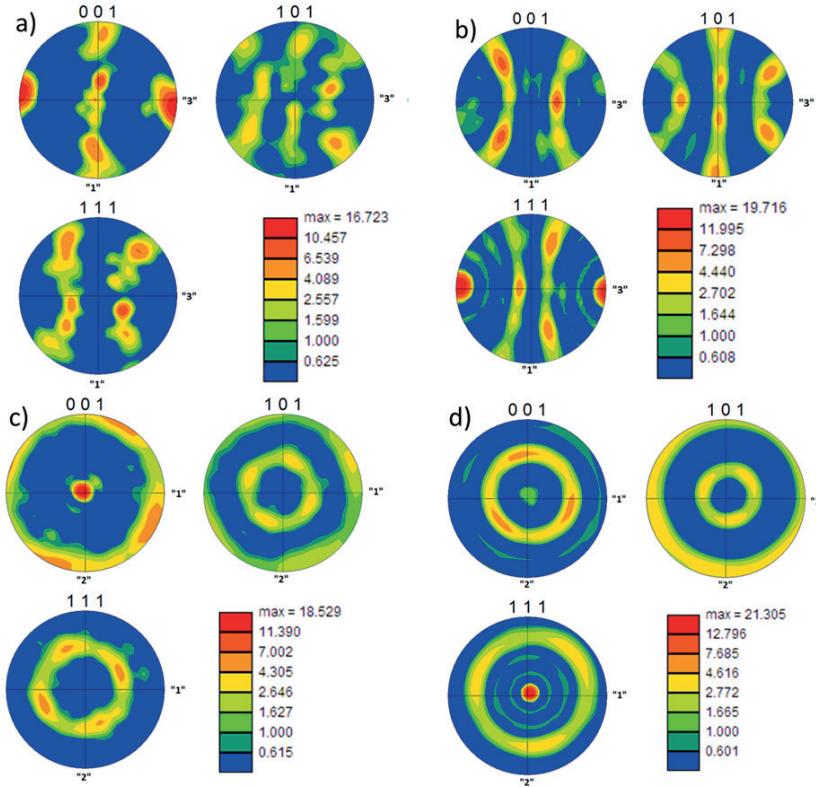

Fig. 7. Pole figures for Al(6N) (a, c) and AA1070 (b, c) measured from longitudinal cross-sections (a, b) and transverse (c, d) cross-sections
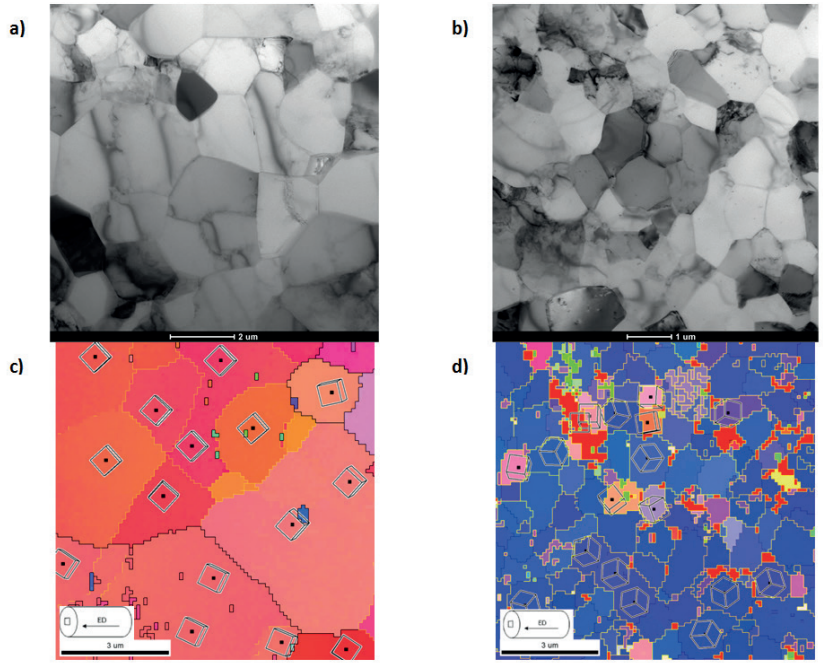
Fig. 8. Bright field images (a,b) and corresponding IPF maps (c,d) of Al(6N) (a, c) and AA1070 (b, d) after KoBo extrusion. Example TEM maps from transverse cross-sections with grains colors correspond to extrusion direction ED from inverse pole figure shown in Fig.1c and with marked cubes visualizing orientation of particular grains. Dark lines corresponds to HAGB and yellow to LAGB
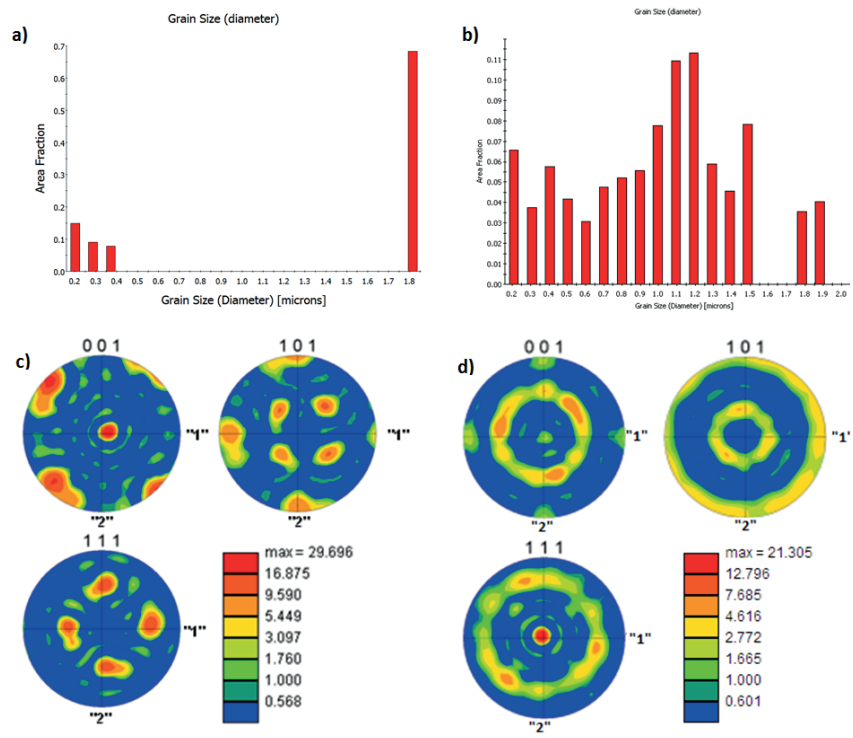

Fig. 9. Grain size (diameter) in function of area fraction (a, b) and pole figures (c, d) for Al(6N) (a, c, e) and AA1070 (b, d, f) measured from transverse cross-sections

Methods of orientation mapping technique is used for quantitative and qualitative analysis of microstructure parameters such as: grain size distribution, grain boundaries and misorientation distribution and local textures. Orientation Microscopy (OM) methods in SEM called EBSD system is now popular and common used for this application. In presented investigation additional information from TEM was used. OM in TEM are not so commonly used, there exist only few systems[11-17], most of them not commercially available. In this research OM in TEM was performed on TECNAI G20 in OML Skawina. This system was built lately on the same concept as in IMMS PAS [11] using for investigation GATAN camera ORIUS SC200 and DM scripts for control microscope and KIKSpot software [18] for analysis and indexing diffraction patterns. For statistical analysis OIM Analysis 6.2 software was used in order to obtain good comparison with SEM maps.

3. Results

On the basis of Inverse Pole Figures (IPF) orientation maps microstructure of both investigated materials was characterized. SEM/ EBSD investigation of Al(6N) showed microstructure characteristic for extruded metals (Fig.3). On the longitudinal section Fig3 visible grains are elongated in ED direction , contrary in transverse cross section they are almost equiaxial. Moreover can be observed typical for KoBo extrusion bimodal distribution of the grain size in transverse and longitudinal cross section. Furthermore especially for AA1070 for transverse cross section was observed substructure with not continuous structure of high angle grain boundaries (fig 4) which can be observed as gradually changes orientation from point to point cumulated into large misorientations. It made difficult proper analysis of grains distribution in TSL OIM analysis. As it was presented in fig. 5 grain sizes were over-
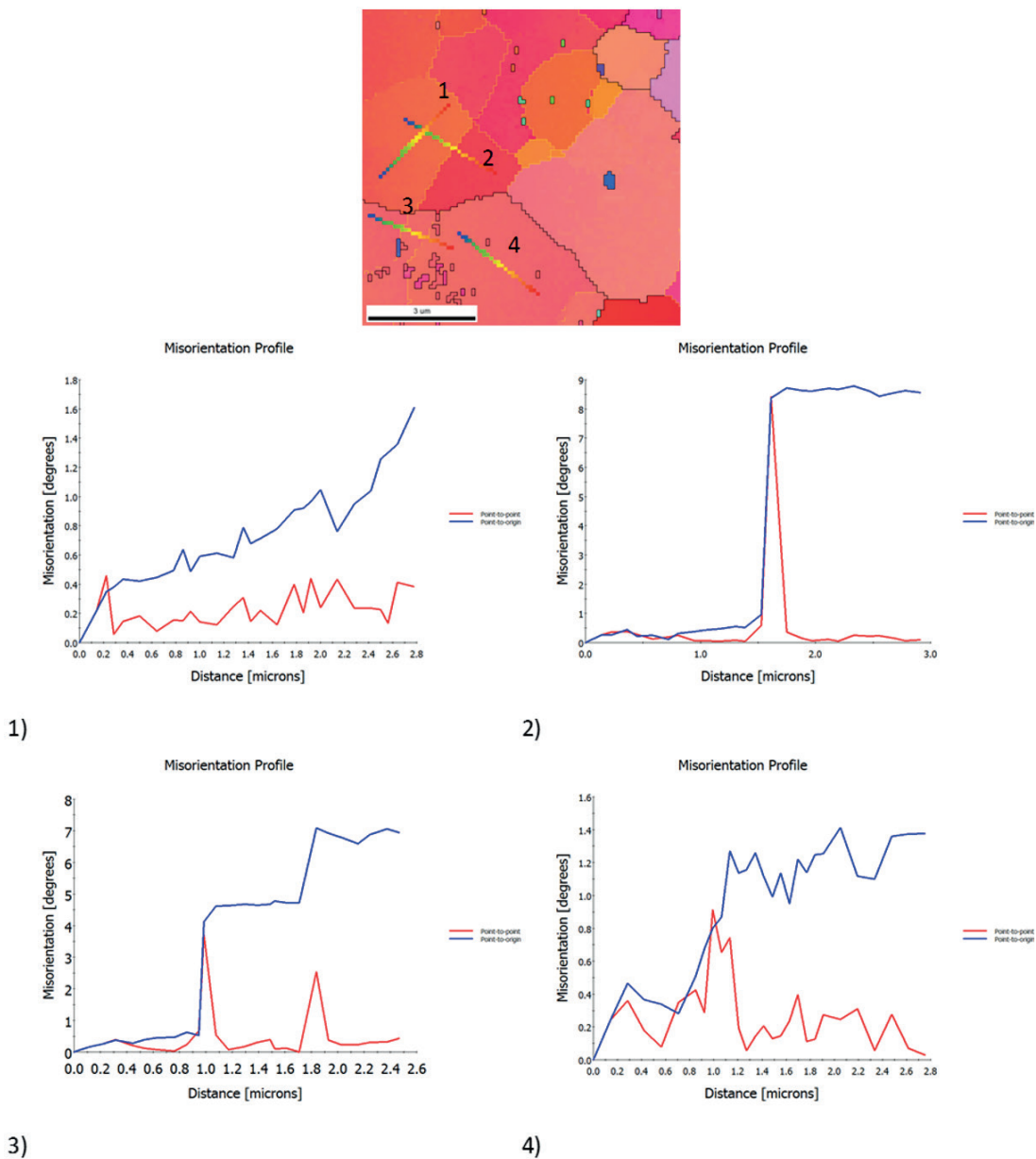

Fig. 10. Orientation map from TEM with marked misorientation profiles (1,2,3,4) across the grains

estimated. For further analysis subgrain structure was taking into account which were presented in fig. 6.

In longitudinal cross section was observed two fraction of the grain size above and under 20 μm respectively 45 and 55%, for transverse cross section similar fraction can be distinguished, but in general the grains are smaller and the ratio of grains above and under 30 μm is 17:3, and 50 % of the grains have diameter less than 10 μm. Misorientation distribution reveals that fraction of LAGBs and HAGBs are respectively 40 and 60% for longitudinal section and 48 and 52% for transverse direction. From pole figures was observed that after KoBo deformation material possesses strong texture {001} on ED (3) direction reassemble texture of initial extruded bars.

EBSD SEM investigations reveals that in AA 1070 can be observed large refinement of the microstructure comparing to initial one. Subgrains structure was observed especially in transverse cross section. In longitudinal cross section subgrains under 10 μm was about 70% and in transverse cross section subgrains under 5 μm was about 58 %. Simultaneously the amount of HAGBs increased and for transverse cross section it reached even 82%. The grains were strongly elongated in ED (Fig. 3b). EBSD measurement of AA1070 shown strongly elongated grains along ED and the HAGBs were mainly parallel to ED. For AA1070 was obtained strong {111} texture on ED (fig.6b, d) and it was also similar to initial one.

In order to compare and confirm investigation from SEM, TEM maps was measured for thin foil from respectively Al6N and AA 1070.

Presenting maps 8 c, d showed that grains in Al 6N after KoBo was larger than in AA1070. In case of AA 1070 on orientation map dominate HAGBs which can be explained by higher accuracy of orientation determination in TEM and higher magnification of investigations then in SEM EBSD (fig.3). Additional information was obtained during observation of orientation gradient between particular grain. This observation showed that between neighboring grains accumulated LAGBs transformed in HAGBs (fig.10).

The local texture for Al 6N and Al 1070 also confirmed previous investigation from SEM and reassemble initial one (fig. 9e,f).

4. Discussion

Examination of Al(6N) and AA1070 shown quite visible differences in microstructure between those materials. Microstructure of Al(6N) consisted of bimodal distribution of grain size suggested local recovery process during KoBo deformation method. It is well known that during KoBo, temperature of extrusion is above room temperature that could provide enough energy to start process. Experiments about static recrystallization performed for Al(5N) deformed by torsion at -196°C shown, that for deformed pure aluminum static recrystallization could occur in room temperatures[19]. Recrystallization of high purity aluminum was also observed after ECAP processing [20]. Pure aluminum is material with high stacking fault energy so it is susceptible for dynamic recrystallization, but recent research started describing discontinuous dynamic recrystallization as a main mechanism that occur in pure aluminum processed by SPD[20,21]. In present research even if dynamic recrystallization occurred, it was incomplete, what is suggested by two peaks of grain size. Grain size diameter larger then 45 μm may also suggest that abnormal grain growth was an effect of secondary recrystallization. Al(6N) after KoBo tends to develop cube texture {001} shown on IPF maps from longitudinal and cross sections. The mechanism is accepted for rolling [22], and after approximation shear to composition tension and compression in ECAP [20,23]. Microstructure of AA1070 was different, grains were smaller and elongated along ED, without strong visible signs of renovation of structure. Obtained texture {111} was also observed for face-centered cubic metals, especially after ARB in close to surface region of specimens, the same like {001} texture [24,25]. Furthermore, in central part of samples {112} and {4 4 11} orientations were observed [24]. Thanks to observation in TEM it was possible to detailed observation of changes inside grains of both deformed materials. Results for AA 1070 confirm reduction of grain size and existence of HAGB's between them. Moreover in TEM it was possible to observe internal small changes of orientation cumulated along the grains into high angles grain boundaries in Al(6N) which confirmed that grains are not fully recrystallized after KoBo deformation.

5. Conclusions

Observation confirm that only small addition of alloying elements lead to significant changes in deformed microstructure especially in grain size. The KoBo processing of pure and commercial pure aluminum led to obtain two different materials in terms of microstructural aspects.

Kobo process for pure aluminum causes dynamical recrystallization. These phenomena effect was texture {001} along extrusion direction.

Commercial pure aluminum performed by KoBo extrusion was not recrystallized. Grains were fiber-like and the main texture along extrusion direction was {111}.

REFERENCES

[1] Hall E.O., *Proc. Phys. Soc. London*, **64**, 747-753 (1951).

[2] Petch N.J., *J. Iron Steel Inst. London*, **173**, 25-28 (1953).

[3] Segal V.M., Reznikov V.I., Drobyshevkij A.E., Kopylov V.I., Russian Metallurgy **1**, 99-105 (1981).

[4] R. Bogucki, K. Sulikowska, M. Bieda, P. Ostachowski, K. Sztwiertnia, Arch. Metall. Mater. **60**, 3063-3068 (2015).

[5] P. Bazarnik, Y. Huang, M. Lewandowska, T. G. Langdon, Materials Science and Engineering A **626**, 9-15 (2015).

[6] A.P. Zhilyaev, T.G. Langdon, Progress in Materials Science **53**, 893-979 (2008).

[7] Saito Y., Utsunomiya H., Tsuji N. and Sakai T., Acta Materiallia **47**, 579–583 (1999).

[8] A. Korbel , W. Bochniak , 1998, U.S. Patent 5.737.959, 2000 European Patent 0.711.210.

[9] A. Korbel , W. Bochniak , P. Ostachowski, Błaż L., Metall. Mater. Trans. A **42**, 2881-2897 (2011).

[10] K. Sztwiertnia, J. Kawałko, M. Bieda, K.Berent, Arch Metall Mater **58** ,157-161 (2013).

[11] M. Bieda, Sol St Phen **186**, 53-57 (2012).

[12] M. Bieda, K. Sztwiertnia, A.Korneva, T. Czeppe, Solid State Phenom. **163**, 13-18 (2010).

[13] J. Bogucka, H. Paul, M. Bieda, T. Baudin, Solid State Phenom 186, 112-115 (2012).

[14] A. Góral, J. Jura, E. Bouzy, M. Bieda, L. Litynska, Arch. Metall. Mater. **51**, 565-568 (2006).

[15] J.J. Fundenberger, A.Morawiec, E. Bouzy, J.S. Lecomte Ultramicroscopy 96, 127-137 (2003).

[16] E.F. Rauch, L. Dupuy, Arch. Metall. Mater. 50, 87-99 (2005).

[17] E. F. Rauch, M. Véron, J. Portillo, D. Bultreys, Y. Maniette, S. Nicolopoulos Microscopy Anal. **93**, S5 (2008).

[18] KIKSPOT www.crystorient.com

[19] F. Haessner, in Chandra T, editor. Recrystallization 90. Warrendale: TMS; 511 (1990).

[20] W. Skrotzki., N. Scheerbaum , C.-G. Oertel Brokmeier, H.-G., S. Suwas S., L.S. To´th Acta Mater. **55**, 2211–2218 (2007).

[21] F. Montheillet, L. Coze, J. Phys Status Solidi A, **51**, 189 (2002).

[22] F.J. Humphreys, M. Hatherly, Recrystallization and related annealing phenomena. Oxford: Elsevier; 2004.

[23] L.S. Toth Adv Eng Mater **5**, 308-316 (2003).

[24] H.W Kim., S.B. Kang, N. Tsuji, Y.Min Amino, Metall Mater Trans A **36**, 3151-3163 (2005).

[25] C.P. Heason, P.B., Prangnell Mat. Scie. For. **408-412**, 733-738 (2002).

[26] S. Li, F. Sun, H. Li, Acta Mater. **58**, 1317–1331 (2010).