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THE MICROSTRUCTURE AND PROPERTIES OF HYDROSTATICALLY EXTRUDED POLYCRYSTALLINE ALUMINIUM Al99.5

MIKROSTRUKTURA I WŁASNOŚCI WYCISKANEGO HYDROSTATYCZNIE POLIKRYSTALICZNEGO ALUMINIUM Al99.5

The changes of Al99.5 microstructure and properties deformed by hydrostatic extrusion process in the conditions of constant strain rate ($1.35\times10^{2}s^{-1}$) and variable deformation (ϕ = 1.44÷2.85) were investigated. The samples were investigated by means both optical (LM) and transmission electron microscopy (TEM). The size of subgrain was statistically measured by mean chord. To establish the influence of hydrostatic extrusion on the properties of the polycrystalline aluminium Al99.5, the microhardness was measured and the tensile test was performed.

The bands and shear bands were the characteristic feature of the sample microstructure. TEM micrographs show equiaxed subgrains with small density of dislocations inside. The measured subgrain size was placed in the range of d = 550÷650 nm. The mechanical properties of polycrystalline aluminium Al99.5 keep almost the same level in the investigated range of deformations. It was found that after the initial deformation microhardness and yield stress nearly twice increase in comparison to the initial state. The greatest increase of properties was observed after deformation ϕ = 1.44. Then the mechanical properties stabilize.

Keywords: aluminium, microstructure, properties, subgrain size, hydrostatic extrusion

W pracy przedstawiono wyniki badań mikrostruktury i własności wyciskanego hydrostatycznie polikrystalicznego aluminium Al99.5. Proces wyciskania hydrostatycznego był realizowany w warunkach stałej prędkości odkształcenia wynoszącej $1,35\times10^{2}s^{-1}$ w zakresie odkształceń rzeczywistych ϕ = 1,44÷2,85.

Tak odkształcone próbki poddano obserwacjom przy zastosowaniu mikroskopu świetlnego (MO) oraz transmisyjnego mikroskopu elektronowego (TEM), zmierzono mikrotwardość oraz przeprowadzono próbę jednoosiowego rozciągania. Dodatkowo stosując parametr średniej cięciwy dokonano statystycznej analizy utworzonych w mikrostrukturze podziarn.

Charakterystyczną cechą wyciskanego hydrostatycznie aluminium są licznie występujące pasma i pasma ścinania, które widoczne są zarówno na przekrojach wzdłużnych, jak i poprzecznych próbek. Wyniki uzyskane przy zastosowaniu transmisyjnego mikroskopu elektronowego wykazały występowanie struktury podziarnowej. Obserwowane podziarna mają niemal równoosiowy kształt. Średnia zmierzona wielkość podziarna mieści się w zakresie d = 550÷650nm. Wyznaczone własności mechaniczne wskazują na ponad dwukrotny wzrost w odniesieniu do stanu wyjściowego przed odkształceniem.

Przeprowadzone badania dowodzą, że polikrystaliczne aluminium Al99,5 odkształcane przy stałej prędkości odkształcenia wynoszącej $1,35\times10^{2}s^{-1}$ w zakresie odkształceń rzeczywistych ϕ = 1,44÷2,85 nie wykazuje istotnych zmian zarówno w mikrostrukturze, jak i w poziomie umocnienia.

1. Introduction

Hydrostatic extrusion process (HE) is one of the Severe Plastic Deformation Method (SPD) which makes possible microstructure refinement to the submicron or even nanometric grain size. From the technological point of view HE gives the possibility to obtain bulk materials in a variety of forms, for example wires, tubes and others.

The possibility of grain refinement depends among others on the: kind of the initial material, amount of deformation, strain rate, value of the stacking fault energy or existence of second phases. Aluminium is the material with high value of the stacking fault energy (SFE), therefore is less susceptible for grain size reduction because of the easy structural recovering, preventing the formation of nanostructures. Application of severe plastic deformation methods (SPD) or deformation with high strain rates, for example by hydrostatic extrusion, gives possibility of microstructure refinement to the submicron size in the aluminium and other metals and alloys [1-15]. The mechanical properties of the ultrafine grained (UFG)

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TABLE 1

Chemical composition of the Al99.5 (Al1050) [% weight]

Cu	Mg	Mn	Si	Fe	Zn	Ti	Al
0.05 max	0.05max	0.05 max	0.25 max	0.40 max	0.07 max	0.05 max	99.5 min

TABLE 2

Parameters of the hydrostatic extrusion process

No	d_o [mm]	d_f [mm]	Extrusion pressure [MPa]	Linear velocity [cm/s]	True strain	Reduction [%]	Strain rate [s^{-1}]
1	10	4.87	251	39.4	1.44	76.3	1.35×10^2
2	13.3	4.87	302	39.4	1.81	83.6	
3	15	4.86	393	39.5	2.25	89.5	
4	20.13	4.85	524	39.7	2.85	94.2	

material differ in comparison to coarse grained (CG) counterpart. Fine grained materials have new and even extraordinary properties such as high strength, low temperature superplasticity, high toughness, specific defect structures, high internal stress and often change of phase composition [1-14].

The aim of the presented study is to establish the influence of the deformation from the range of ϕ = 1.44 to ϕ = 2.85 exerted under the conditions of the constant strain rate $1.35\times10^2 s^{-1}$ on the evolution of microstructure and properties in polycrystalline aluminium Al99.5 deformed by hydrostatic extrusion.

2. Experimental procedure

The investigations were carried out on the polycrystalline aluminium Al99.5 (Table 1).

Before the deformation the samples were annealed in the temperature 500°C during 2 hours. The samples having a mean grain size of 100 μm were subjected hydrostatic extrusion at the constant strain rate $1.35\times10^2 s^{-1}$ in order to attain true strains between 1.44 and 2.85. Although the process was realized at room temperature, sample heating induces by the high strain rates was possible. Therefore the samples were water cooled at the exit of the die in order to minimize the effect of temperature on properties and microstructure. Detailed information's about deformation process are presented in the Table 2.

After the deformation, the samples were investigated by means both optical and transmission electron microscopy JEM 2010 ARP. The investigations by means of optical microscopy were performed on the longitudinal and transverse sections of the samples. The samples were cut out and then mechanically ground and polished using diamonds pastes and colloidal suspension of SiO_2. The used technique for showing microstructure and deformation effects, was etching in the Barker reagent. The composition of the Barker reagent was: 1.8 cm^3 HBF_4+ 100 ml H_2O. The thin foils for TEM observations were cut out from longitudinal section of the samples and prepared applying the standard technique of electrolytic polishing using Struers apparatus. The micrographs obtained from TEM observations were used for the calculations of the statistical size of the subgrains observed in the microstructure. This was determined by mean chord method.

To establish how hydrostatic extrusion influences the properties of the polycrystalline aluminium Al99.5, the microhardness was measured and the tensile test was performed. The microhardness measurements were performed on the polished longitudinal sections of the samples perpendicularly to the sample axis. The applied load was 100 g. The tensile test was performed in the room temperature with constants strain rate of the order of $10^{-3}s^{-1}$.

3. Results and discussion

In order to avoid misunderstanding definitions of "bands" and "shear bands" for the need of the article should be introduced. Thus, the word "a band" concerns a band which is limited to a single grain only, whereas "shear bands" may proceed at the considerable distance and cross grain boundaries and sometimes forms distinct jogs at the intersected boundaries.

The micrographs obtained by using optical microscope are presented in Figures 1-4. Characteristic features was bands and shear bands occurrence which were observed both in the transverse (Fig.1a, 2a, 3a, 4a) and longitudinal section of the samples (Fig.1b, 2b, 3b, 4b).

The observed bands and shear bands are seen both as a thick lines (Fig. 3b, 4b) and having dozens micrometers in thickness and propagate to great distances (Fig. 1a, b, 2a, 3a, 4a). The observed bands and shear bands have diversified inclination to the extrusion direction. Also characteristic are "serrate" grain boundaries, what is particularly seen in Figures 2b and 3b. This phenomenon can prove shear bands propagation across grain boundaries.

The microstructures, with the described features, observed by using optical microscope were typical in the whole investigated range of deformations.

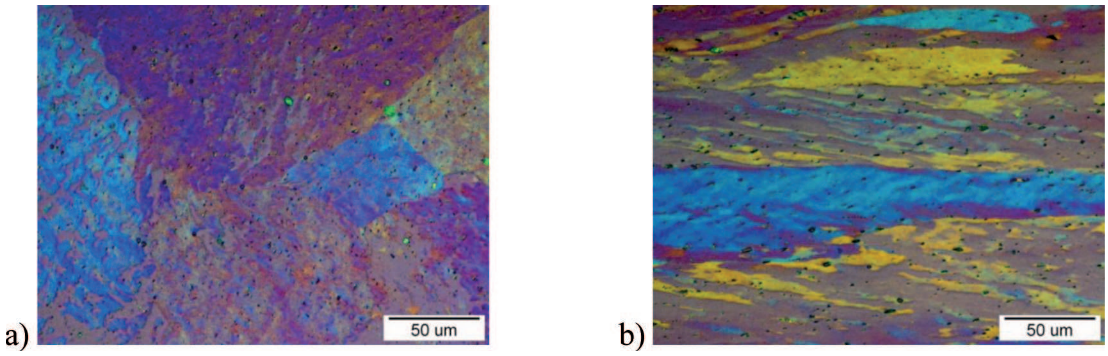

Fig. 1. Microstructure of extruded aluminium Al99.5 to the deformation $\phi = 1.44$; a) transverse section of the sample, b) longitudinal section of the sample

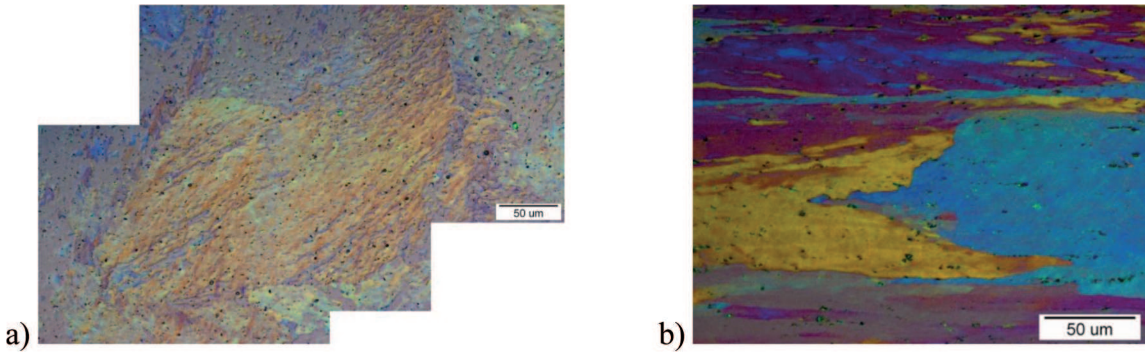

Fig. 2. Microstructure of extruded aluminium Al99.5 to the deformation $\phi = 1.81$; a) transverse section of the sample, b) longitudinal section of the sample

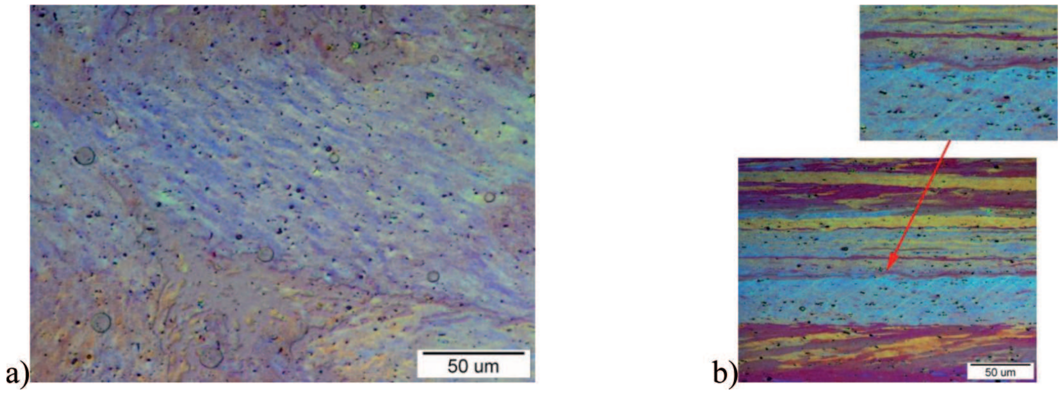

Fig. 3. Microstructure of extruded aluminium Al99.5 to the deformation $\phi = 2.25$; a) transverse section of the sample, b) longitudinal section of the sample

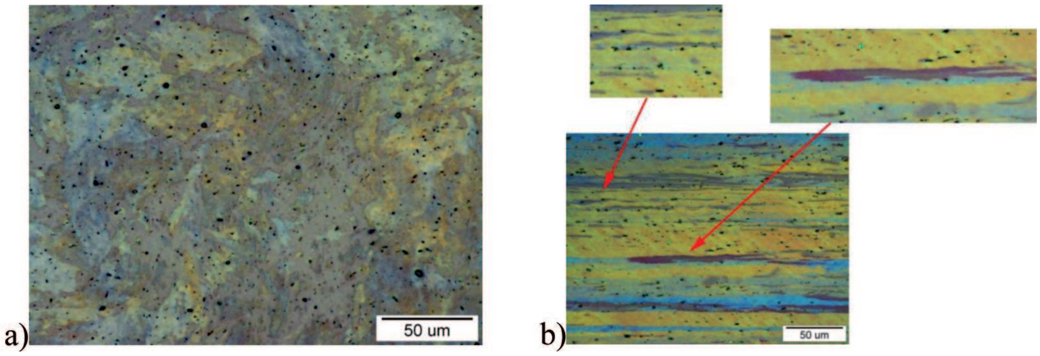

Fig. 4. Microstructure of extruded aluminium Al99.5 to the deformation ϕ = 2.85; a) transverse section of the sample, b) longitudinal section of the sample

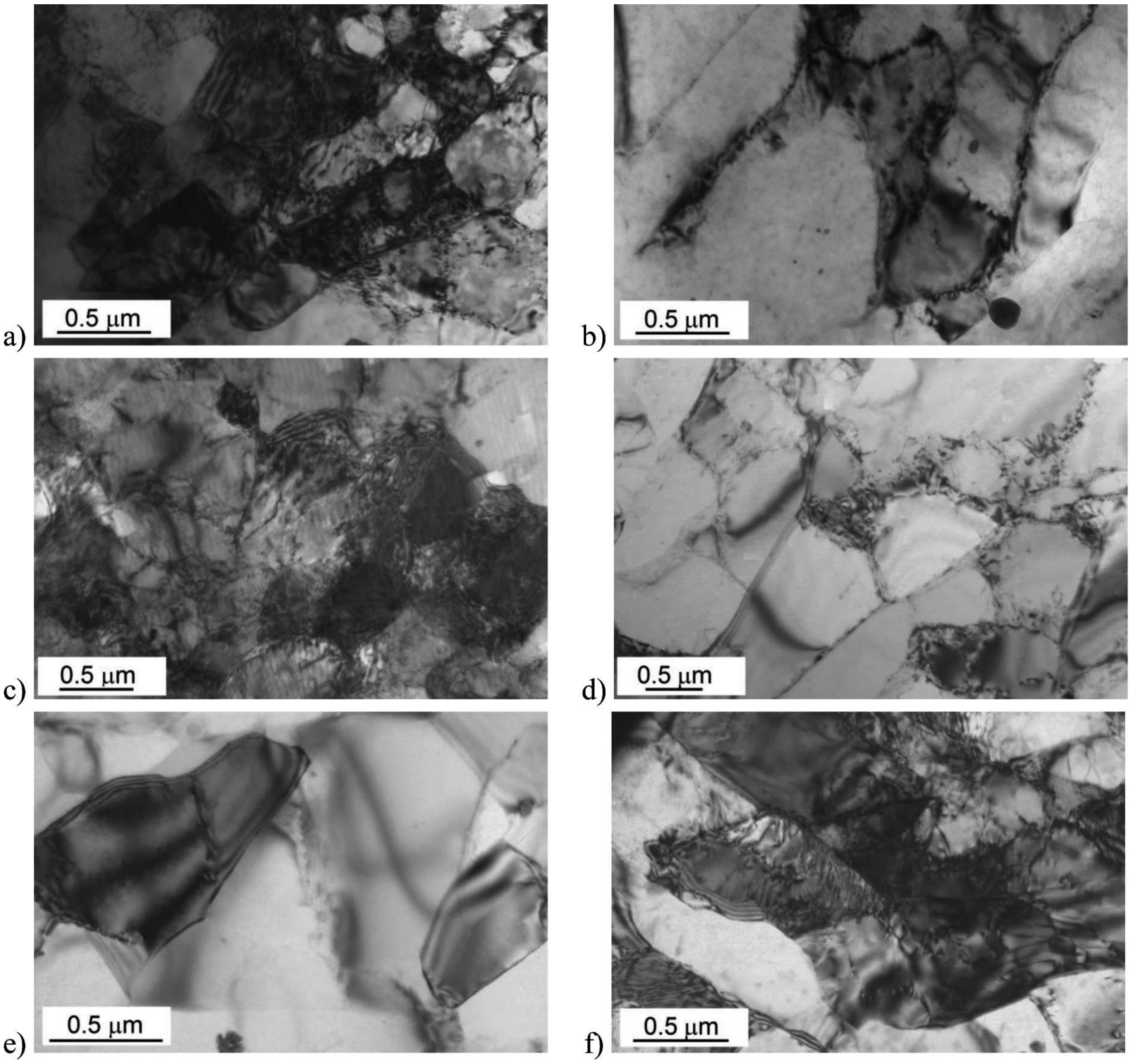

Fig. 5. Characteristic dislocation microstructures of hydrostatic extruded aluminium Al99.5; a, b) ϕ = 1.44, c, d) ϕ = 2.25, e, f) ϕ = 2.81

Detailed information about influence of the deformation exerted in the conditions of constant strain rate on the polycrystalline aluminium Al99.5 provided data of the microstructure observation performed by using transmission electron microscopy presented in Figs 5a-f and the subgrain size measurements (Fig. 6, 7).

Characteristic feature was occurrence of equiaxed subgrains in microstructure. It was observed that some of subgrains have dislocations inside (Fig. 5a, c, f) and some of them are free of dislocations (Fig. 5b, d, e). The existence of dislocations inside depended on the changing of the position and inclination of thin foil, however some area didn't exhibit dislocations independently to the foil position.

The next characteristic feature of microstructure was microbands. The observed microbands were built from elongated subgrains (Fig. 5a, b). In some of them the occurrence of small subgrains was found (Fig. 5a).

The subgrain and microbands boundaries show small amount of dislocation inside. The arrangement of dislocation inside subgrain is especially presented in Figure 5f. The existence of the free of dislocation subgrains indicates on the intense structural renewal processes development, which probably take place during or directly after deformation. Similar microstructure was observed in the aluminium deformed by equal channel angular extrusion (ECAE) process and reported by P.L.Sun and all [14, 15]. The microstructure was built from equiaxed grains with a submicron size. Most of the grains in as deformed structure were free of dislocations.

Presented in Fig. 6 subgrain measurements didn't show considerable differences in the size in the range of HE deformations exerted with constant strain rate $1.35\times10^{2}\mathrm{s}^{-1}$. The smallest subgrain size was measured in the sample deformed to the strain $\phi = 2.85$ and achieved the level of 546 nm. In the case of the samples deformed to strains $\phi = 1.44$ and $\phi = 2.25$, the measured subgrain size was 20% higher and placed in the range of 620÷650 nm.

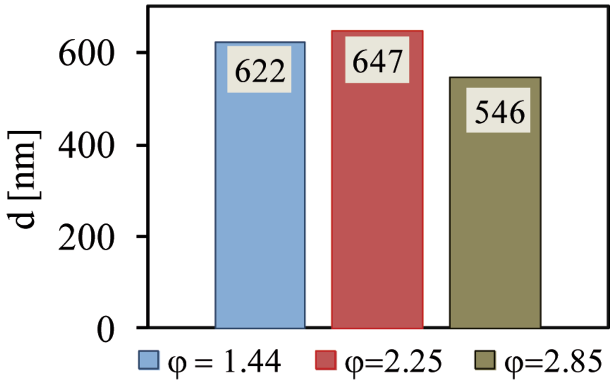

Fig. 6. The mean subgrain size as a function of deformation

The distributions of the mean subgrain size presented in Fig. 7 shows the increase of deformation causes reduction of subgrain population from the range 1÷1.4 μm. Both in the case of the samples deformed to the deformation $\phi = 1.44$ and higher ($\phi = 2.25$ and $\phi = 2.81$) most representative group of the measured subgrains were observed in the range of 300÷800 nm. The population of the measured subgrains from this range was about 80%.

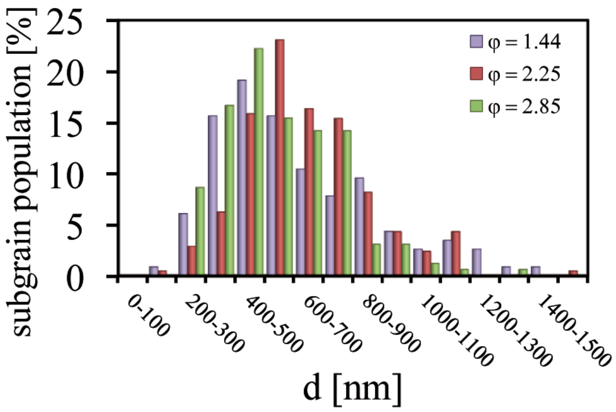

Fig. 7. The influence of the deformation on the subgrain size in the polycrystalline aluminium Al99.5

The influence of subgrain size on the mechanical properties was tested in microhardness and yield stress measurement. The results obtained from microhardness measurements are presented in Fig. 8, whereas results of the tensile test in Fig. 9. Additionally in Fig. 10 the distribution of the microhardness on the samples section is presented.

The nearly double increase of microhardness after deformation $\phi = 1.44$ was found in comparison to the initial state, from 23 HV0.1 to 39HV0.1. Above this strain only a slight variations in microhardness were noticed. After the deformation $\phi = 2.25$ the microhardness achieved the highest level, average 42 HV0.1 (Fig. 8).

The distribution of the microhardness along the samples sections is rather homogeneous. The highest spread of results was found after the deformation $\phi = 1.44$. On the edge of the sample, values of the microhardness are lower than in the central part of the sample. The differences between edge and central part of the samples don't beyond of 10%. Inversely is after deformation $\phi = 2.25$, the edge of the sample is harder than central part. Diversified distribution of the microhardness is probably causing by diversification of sample hardening.

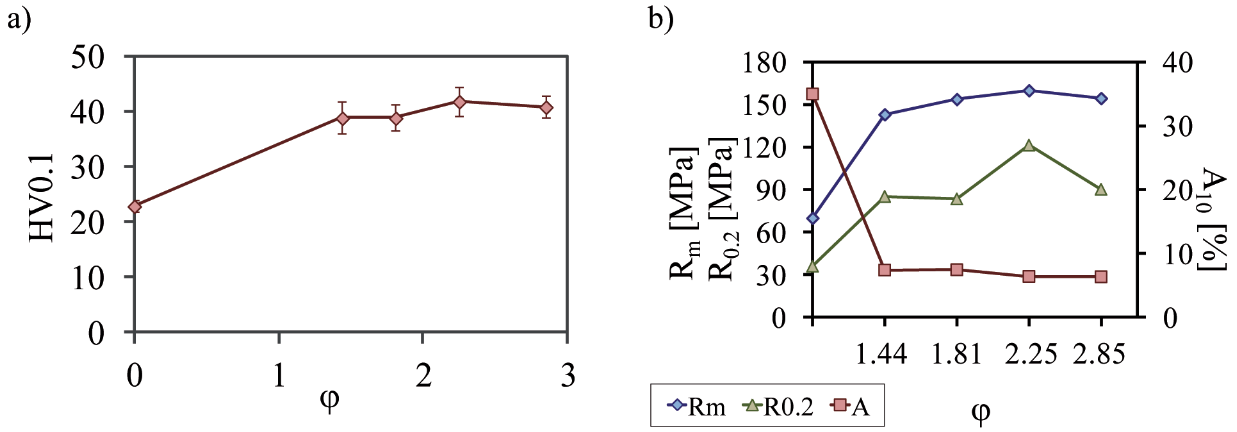

Fig. 8. The influence of the hydrostatic extrussion on the mechanical properties of the polycrystalline aluminium Al99.5; a) microhardness measurements, b) tensile test results

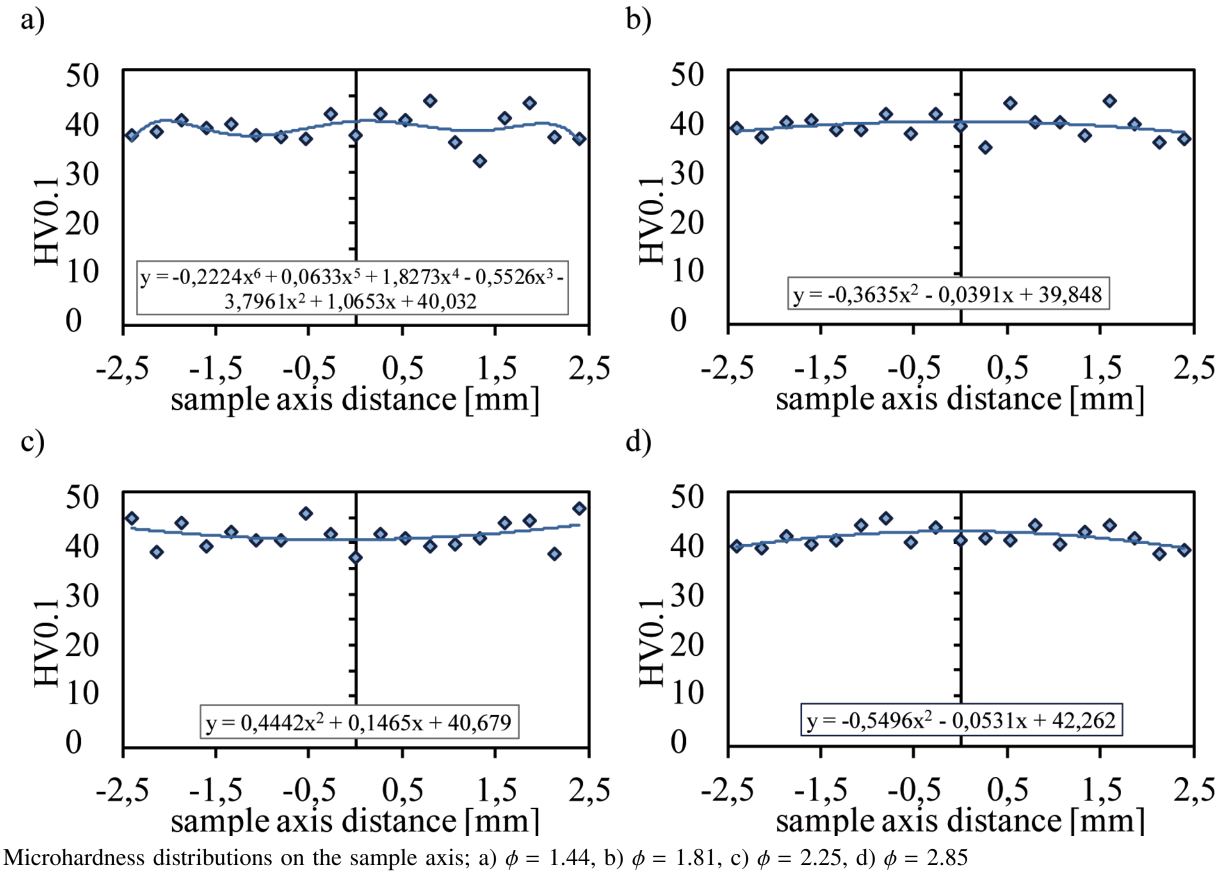

Fig. 9. Microhardness distributions on the sample axis; a) ϕ = 1.44, b) ϕ = 1.81, c) ϕ = 2.25, d) ϕ = 2.85

The results obtained from the tensile test indicate considerable hardening of the polycrystalline aluminium Al99.5 after hydrostatic extrusion process. However, after achieve of the some value of properties, in the investigated range of deformation (ϕ = 1.44÷2.85) its keeps almost the stable level. Exception to this is value of the $R_{0.2}$ for the sample deformed to the deformation ϕ = 2.25, which is 30% higher than in the other samples (Fig. 8b). With the comparison to the initial state, R_m and $R_{0.2}$ increase over two times. The elongation decreases five times.

The highest level of the mechanical properties in the sample deformed to the deformation ϕ = 2.25 and simultaneous the highest measured subgrain size can prove, that in this sample the density of the dislocations is higher than in other samples and that the work hardening predominate the structural renewal processes.

Two stage hardening indicate that the Al99.5 achieved an equilibrium state between strain hardening and microstructure renewal and that the increase of deformation exerted in the conditions of constant strain rate didn't influence on the properties level in the HE deformation conditions.

In Table 3 the yield stress and the mean subgrain size as a function of the deformation are presented.

TABLE 3
Yield stress and the mean subgrain size of hydrostatically extruded Al99.5

ϕ	**d [nm]**	**$R_{0.2}$ [MPa]**
1.44	622	85.2
2.25	647	121.4
2.85	546	90.3

Presented data show highest value of the $R_{0.2}$ for the sample with the highest subgrain size (d = 627 nm) and about 35% lower value of the $R_{0.2}$ for the sample with the subgrain size of d = 546 nm. This phenomenon can suggest the inversion of the Hall-Petch relation and is often observed in the materials with nanometric or ultrafine grain size.

4. Conclusions

1. Hydrostatic extrusion process can be an effective method for microstructure refinement to the submicron size in the polycrystalline aluminium Al99.5. The subgrain size of Al99.5, deformed by HE in the strain range $\phi = 1.44 \div 2.85$, is placed in the range d = 540÷620 nm.
2. The bands limited to single grains and shear bands proceed at the considerable distance and crossing grain boundaries are characteristic feature of HE microstructures.
3. The microhardness measurements and the value of properties of Al99.5 obtained from the tensile test show two stages course. The greatest increase in microhardness and R_m was observed after the deformation $\phi = 1.44$. The microhardness increased from the initial state 23 HV0.1 to 39 HV0.1 and R_m from 70 MPa to 143 MPa, adequately. Above the deformation $\phi = 1.44$ the level of properties stabilized and only a slight variations in microhardness and R_m were noticed.
4. The presented data show that the Al99.5 achieved an equilibrium state between strain hardening and microstructure renewal within the investigated range of HE strains $\phi = 1.44 \div 2.85$, exerted at the constant strain rate ($1.35 \times 10^2 s^{-1}$), which manifested by properties stabilization.

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