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EFFECTS OF EXTRUSION PARAMETERS BY KoBo METHOD ON THE MECHANICAL PROPERTIES AND MICROSTRUCTURE OF ALUMINUM

WPŁYW PARAMETRÓW WYCISKANIA METODĄ KoBo NA WŁASNOŚCI MECHANICZNE I STRUKTURĘ ALUMINIUM

Commercial purity aluminum was extruded by means of KoBo method at varied processing parameters. Received extrudates, with different mechanical and electrical properties and work hardening behavior, were obtained. It was found, that some conditions of KoBo extrusion process such as low initial billet temperature, low extrusion rate and low frequency of oscillating die lead to extremely high strength and high electrical resistivity of the material. The absence of work hardening (up to 40% strain) during subsequent groove rolling is also a specific feature of received materials. It was suggested that mentioned features are related to the development of overbalance concentration of point defects (clusters) generated during the extrusion process. During following cold rolling of the extrudate, mentioned defects annihilate at gliding dislocations and make the dislocation climbing and their rearrangement easier. Therefore, until the exhaustion of this mechanism, the hardening of material during cold rolling is very limited. Following increase of the material strengthening at higher rolling strains point to the return of the material to its typical behavior observed for cold deformed aluminum produced by conventional hot extrusion.

Keywords: aluminum, KoBo extrusion, electrical resistivity, work hardening

Aluminium o czystości handlowej poddano wyciskaniu metodą KoBo przy zastosowaniu zmiennych parametrów procesu, w wyniku czego uzyskano prasówki o zróżnicowanych własnościach mechanicznych i elektrycznych oraz odmiennych charakterystykach umocnieniowych. Stwierdzono, że w przypadku procesu wyciskania wsadu o niskiej temperaturze początkowej, prowadzonego z małą prędkością i przy niskiej częstotliwości oscylacji matrycy, prasówka wykazuje nadzwyczaj wysokie własności wytrzymałościowe, wysoki opór elektryczny oraz brak umocnienia odkształceniowego podczas walcowania (w zakresie do 40%), co związano z obecnością w materiale ponadrównowagowej koncentracji defektów punktowych (klasterów), wygenerowanych w procesie wyciskania. Podczas walcowania defekty te ulegają anihilacji na przemieszczających się dyslokacjach, ułatwiając ich wspinanie oraz przegrupowanie. W konsekwencji, dopóki mechanizm ten nie ulegnie wyczerpaniu, umocnienie odkształceniowe takiego materiału podczas walcowania ma ograniczony charakter, a typowe efekty umocnieniowe, podobne do tych dla aluminium wyciskanego konwencjonalnie na gorąco, występują przy większych odkształceniach.

1. Introduction

Extrusion of metallic materials by means of KoBo method can be performed at lower temperature than commonly used conventional hot extrusion methods [1, 2]. The advantage of the method results from the specific die oscillation, that causes the effective reduction of processing energy, increasing the processing efficiency [3, 4]. Significant benefits for industrial production are related to the product quality, quantity, mass of individual product, especially to receiving specific mechanical properties, of hardly deformable alloys [5, 6]. Korbel and Bochniak [7, 8] concluded that the plastic flow at KoBo method is controlled by the generation of Frenkel defects and estimate their overbalance concentration rising up to 10^{-8}. On the basis of TEM observations they suggested that at least part of strain induced point defects may create stable clusters in extrudate, which result in increasing strength of the material.

Experiments performed on pure zinc and aluminum [9, 10] revealed that the mechanical properties of KoBo extruded wire varied along its length that depends on the die oscillation angle and the extrusion rate to oscillation frequency ratio. Extruded material was characterized by the high strength properties, low dislocation density and a conventional grain size. It was found that in spite of strain induced spontaneous increase of deformation temperature from the initial 293 K at the beginning of extrusion up to estimated value of ~570 K at the end of the processing, mechanical properties of received extrudate did not change significantly along the wire length. Samples machined from the initial part of as extruded wire, i.e. processed at low temperature, and from the end of the wire (high temperature) were characterized by high value of ultimate tensile stress ~200 MPa and ~180 MPa, respectively. Moreover, annealing experiments performed on KoBo extruded samples revealed unexpectedly high thermal stability of mechanical properties up to ~473 K. It was suggested that

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mentioned above effects are related to high thermal stability of point defect clusters produced due to specific intense deformation at KoBo extrusion [9, 10].

The research work were undertaken to get more information on features of the aluminum processed by means of KoBo extrusion with respect to the effect of specific extrusion parameters and following cold rolling on the structure and mechanical properties of KoBo extruded materials.

2. Experimental

Hot extruded commercial rods of aluminum 99.7% purity (AA1070) with a diameter of 40 mm were used for the experiment. Billets, 55 mm length, were cut from as delivered rods and extruded by means of KoBo method using 1 MN press and extrusion ratio $\lambda = 100$. Wires, 4 mm in diameter were received. Initial temperature of the billet 293 K, 373 K, 493 K and 673 K and constant extrusion rate, i.e. velocity of the punch displacement v = 0.1 mm/s and 0.5 mm/s were applied. Processing was carried out using the die oscillation angle ±8° at the oscillation frequency f = 3 Hz, 5 Hz and 8 Hz. Both the die and the extrudate were water cooled during processing. As extruded wires were then cold rolled in grooves (square / square scheme) up to the cross-section reduction ratio ~90%. Mechanical properties of both as extruded and cold rolled aluminum rods were tested by means of Zwick / Roell Z050 testing machine using a tensile test performed at strain rate of $8\times10^{-3}s^{-1}$. Some of the results were presented in an earlier work [11]. Light microscopy, transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM) were used to examine the material structure. Samples for light microscopy observations were cut along extrusion axis and prepared using electrolytic polishing and anodizing in Barker's reagent. Thin foils for TEM / STEM observations were thinned mechanically and plates of ~0.2 mm in thickness were finally electropolished using Struers Tenupol electrolytic device. For comparison purposes, structure of cold rolled aluminum samples deformed with cross-section reduction corresponding to the deformation value of tested wires was also observed. Samples 300 mm in length were cut from the wire to test electrical resistivity of both as extruded and cold rolled wires. The measurements were performed at ambient temperature using Resistomat 2304 device.

3. Results and analysis

3.1. Aluminum extrudate

The effect of extrusion parameters, i.e. initial temperature of the billet, extrusion rate and frequency of die oscillation, on mechanical properties of aluminum extrudates are shown in Fig. 1a,b. The properties were measured for the beginning (Fig. 1a) and the ending part of extruded wire (Fig. 1b) to estimate the effect of increasing deformation temperature that can be expected from the intense processing of the material. Tensile tests revealed diversification of mechanical properties along the length of KoBo extruded wire, that depends on initial temperature of the billet, extrusion rate and die oscillation frequency. Both the yield stress (YS) and ultimate tensile stress (UTS) value were reduced from the beginning to the ending part of the extrudate that was accompanied by the increased tensile elongation. However, differences between mechanical properties of initial and ending part of the extrudate do not exceeds ca. 35%.

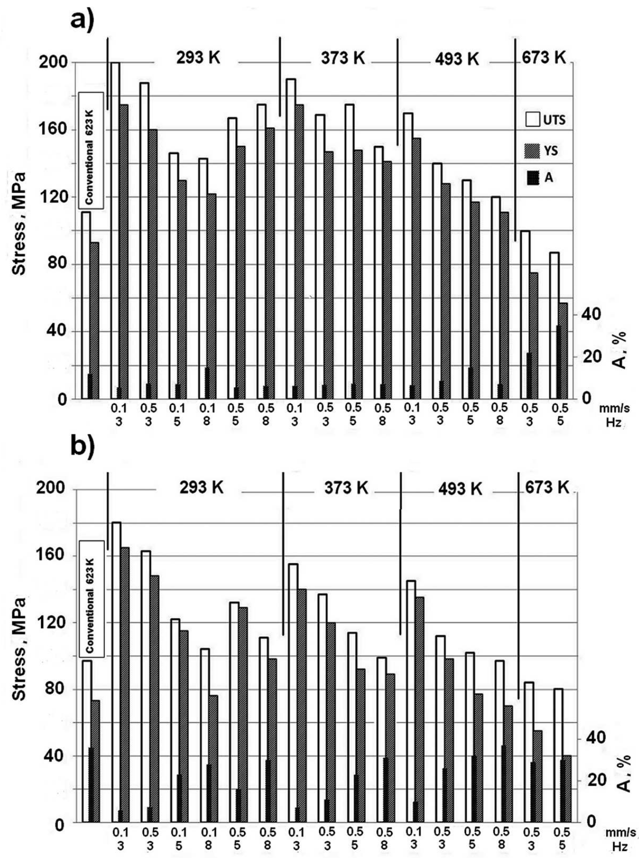

Fig. 1. Mechanical properties of aluminum wire extruded by means of KoBo method: (a) beginning part of the extrudate, (b) ending part of the extrudate. Initial temperature of extruded billet, velocity of the punch displacement, oscillation frequency is marked in the figure

Relatively high influence on the mechanical properties of aluminum wires was found to be related to applied processing parameters. In particular, initial temperature of the billet and both extrusion rate and the die oscillation frequency were found to affect the mechanical properties of extruded wires. For example, the highest YS ≈ 175 MPa and UTS ≈ 200 MPa were observed for the beginning part of the 'cold' extruded wire. It should be stressed that the initial part of the wire extruded at 0.1 mm/s and low die oscillation frequency 3 Hz was obtained from the billet having initial temperature of 293 K (Fig. 1a). The high strength properties of wire are accompanied by relatively low elongation value that does not exceed 5-7%. Reduction of strength properties due to increasing preheating temperature of the billet up to 373 K was negligible and relatively high values of YS ≈ 155 MPa and UTS ≈ 175 MPa were observed even for the initial part of the wire extruded at preheating temperature 493 K (Fig. 1a). If the die oscillation frequency remain unchanged (f = 3 Hz) and extrusion rate is increased to 0,5 mm/s, resulted YS and UTS values are reduced ~20 % with respect to the 'cold' extruded material (Fig. 1a,b). Relatively low mechanical properties are received for the wires 'cold' extruded at v = 0.1 mm/s and f = 5 Hz and 8 Hz. As mentioned formerly, the temperature of billet increases during process due to the deformation heating effect. Therefore, the ending part of all extrudates is characterized by lower mechanical properties and higher tension elongation values (A ≈ 30%), which become similar to these received for conventional hot extruded (673 K) aluminum products (YS = 40÷45 MPa, UTS = 80 MPa, A ≈ 30 %).

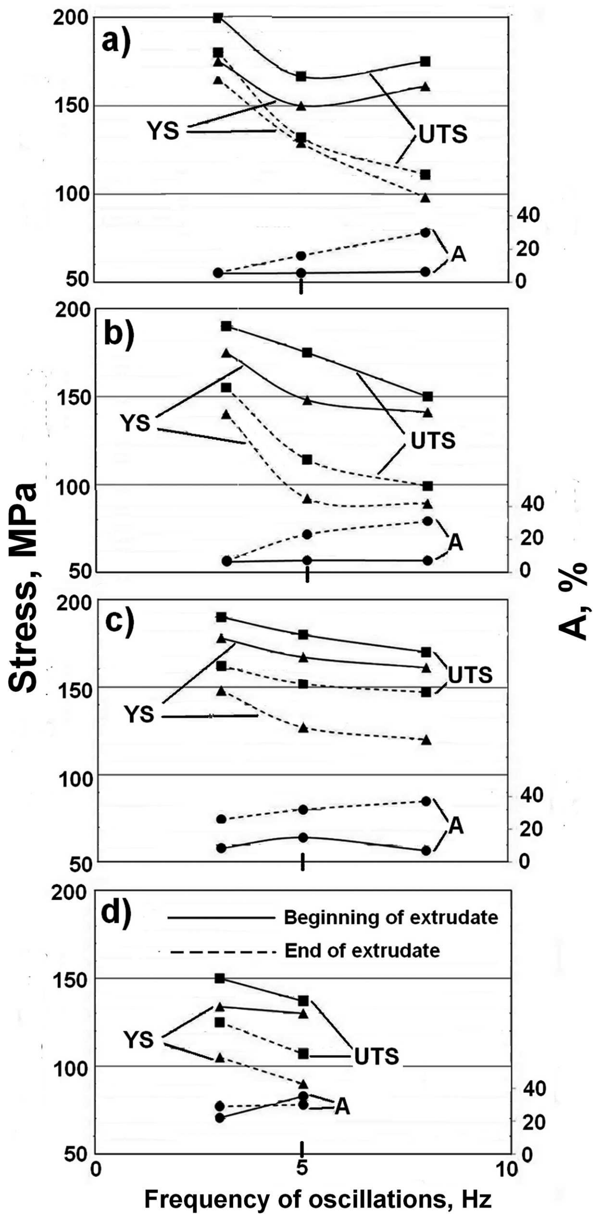

Fig. 2. Effect of die oscillation frequency on mechanical properties of aluminum wire extruded by KoBo method. Following initial temperature of billets and extrusion rate were used: (a) T = 293 K, v = 0.1 mm/s; (b) T = 373 K, v = 0.1 mm/s; (c) T = 493 K, v = 0.5 mm/s; (d) T = 673 K, v = 0.5 mm/s

The obtained results lead to the conclusion that the higher frequency of die oscillation result in lower strengthening of aluminum extrudate (Fig. 2). It can be caused by the increase of deformation temperature due to more deformation (torsion) energy supplied to the metal and dissipated in a form of heat. It should also be considered, how the frequency of die oscillation affects the number of twists in deformation zone of the extruded billet. It was reported that the size of active torsion zone in the billet is ~2 mm in thickness [7]. Therefore, if the extrusion ratio $\lambda = 100$, extrusion rate v = 0.5 mm/s and the die frequency f = 3 Hz are taken into consideration, there should be 12 cycles of torsion in 2 mm zone of the plastic flow. If the process is carried at v = 0.1 mm/s and f = 8 Hz twist cycles in the deformation zone should reach even 160 cycles. Calculation results for all parameters used in the experiment are given in Table 1 and it seems to correspond very well to the reduction of strength properties caused by the increased temperature and the deformation energy supplied into the material.

TABLE 1

Number of twists (±8°) in 2 mm thick deformation zone for the billet extruded at given extrusion rate and frequency of die oscillation

Die oscillation frequency, Hz	Extrusion rate, mm/s			
	0.1	0.3	0.5	1
3	60	20	12	6
5	100	33	20	10
8	160	53	32	16

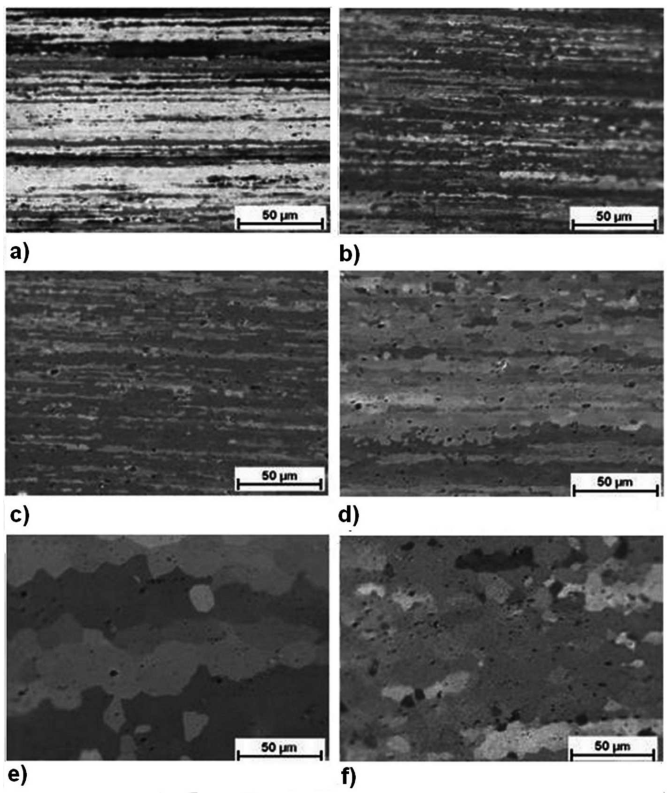

Fig. 3. Microstructures of the ends of aluminum wires extruded by means of KoBo method at different processing conditions: (a) T = 293 K, v = 0.5 mm/s, f = 8 Hz; (b) T = 293 K, v = 0.1 mm/s, f = 8 Hz; (c) T = 373 K, v = 0.5 mm/s, f = 8 Hz; (d) T = 493 K, v = 0.5 mm/s, f = 8 Hz; (e) T = 673 K, v = 0.5 mm/s, f = 8 Hz; (f) T = 673 K, v = 0.1 mm/s, f = 8 Hz (T, v, f – initial temperature of billet, extrusion rate and frequency of die oscillation, respectively)

The effect of the billet temperature on the material structure, revealed by optical microscopy, is illustrated in Fig. 3. The process carried out at low initial temperature of the billet promotes the development of fibrous structure. Increasing billet temperature result in receiving of partially or completely recrystallized structure of as extruded wire. However, a very important observation is that regardless of the analyzed variant of deformation, it should be stressed that all used extrusion parameters foster creation of highly recovered subgrains 2-5 μm in size containing negligible number of dislocations in their interior (Fig. 4a). It is commonly accepted that the effect of grain size (or subgrain size) on strength properties of aluminum is very low, i.e. Hall-Petch relation is very week [12]. Therefore, so large differences on observed strength properties of aluminum KoBo-extruded at varied extrusion parameters cannot be ascribed to the received subgrain/grain size (Figs. 1 and 2, Table 2). It seems reasonable to search for some additional structural processes, which may affect the final strength of KoBo extruded material.

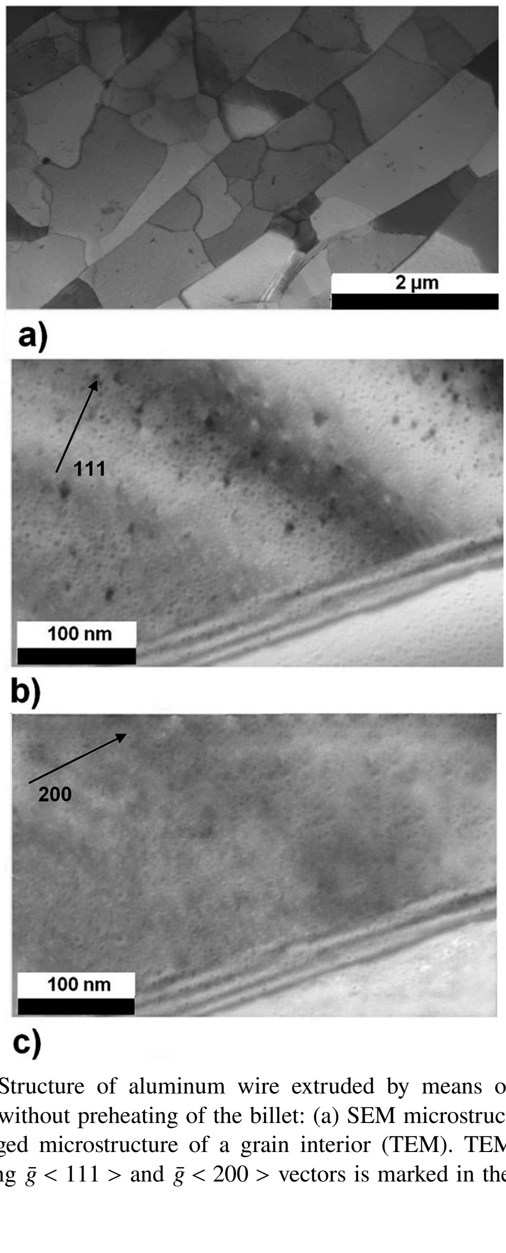

Fig. 4. Structure of aluminum wire extruded by means of KoBo method without preheating of the billet: (a) SEM microstructure; (b, c) enlarged microstructure of a grain interior (TEM). TEM image (operating $\bar{g}$ < 111 > and $\bar{g}$ < 200 > vectors is marked in the figure)

TABLE 2

Strength properties and electrical resistivity of aluminum wires manufactured using different processes

Processing of the material		YS / UTS MPa	Resistivity nΩm
Conventionally extruded		111 / 128	28.6
Conventionally extruded and drawn to Ø 2.2 mm		151 / 188	28.8
Conventionally extruded, drawn to Ø 2.2 mm and recrystallized at 673 K / 2 h		32 / 87	27.8
Conventionally extruded, drawn to Ø 2.2 mm, recrystallized at 873 K / 0.5 h and cooled in cold water		20 / 59	29.1
KoBo extruded; initial temperature of a billet 293 K	beginning of extrudate	123 / 180	29.2
	end of extrudate	89 / 120	29.0
KoBo extruded; initial temperature of a billet 493 K	beginning of extrudate	90 / 110	28.5
	end of extrudate	76 / 100	28.7
KoBo extruded; initial temperature of billet 673 K	beginning of extrudate	64 / 100	28.6
	end of extrudate	63 / 100	28.4

TEM observations performed at high magnification revealed very fine spots in the grain interior as shown in Fig. 4b. The spots were observed in the image created < 111 > $\bar{g}$ vector diffraction conditions and < 200 > $\bar{g}$ and did not occur outside of the specific diffraction conditions (Fig. 4c). Density of the spots is especially high in wires with high value of UTS. Similar nanometer-in-size spots were observed at some irradiated metallic materials and have been ascribed to the clusters of point defects (i.e., vacancies and atoms in interstitial position) and / or very fine dislocation loops [13, 14]. Mentioned crystalline structure imperfections may contribute some hardening of the material. It was found that several times increase in the yield stress value can be obtained due to rapid quenching of aluminum single crystals and the maximum yield stress was observed not instantly after quenching, but after some ageing at room temperature [15]. Assumption that the spots can be related to point defects was positively verified by the elecrical resistivity measurements of investigated aluminum (Table 2). The lowest electrical resistivity value was found for conventionally extruded and cold drawn aluminum wires, annealed at 673 K / 2 h. UTS and the electrical resistivity value for as recrystallized and slowly cooled material was 87 MPa and 27.8 nΩ m, respectively. Water quenching from 873 K raised excess of vacancies resulting in an increase in resistivity to 29.1 nΩ m. Relatively high resistivity values, observed for KoBo extruded wires having highly recovered or recrystallized structure, can be also ascribed to high density of point defects, which are produced due to especially intensive processing of the material (Table 2).

3.2. Work hardening behavior of KoBo extruded aluminum wires

KoBo extruded wires were cold rolled using grove rolling mill. Effect of cold rolling reduction on a tensile strength and elongation, with respect to the preliminary extrusion conditions, are shown in Fig. 5. Analysis of received results leads to two conclusions: (1) the lower YS and UTS of received extrudate, the higher the work hardening due to the rolling strain and (2) the highest strength parameters for as extruded material are maintain a high value during cold rolling. For example, cold rolling of material extruded at T = 493 K, v = 0.1 mm/s, f = 3 Hz, result in an increase of YS and UTS from 175 MPa and 200 MPa, respectively, to YS = 190 MPa and UTS = 230 MPa for cold rolled wire with strain ε = 90%.

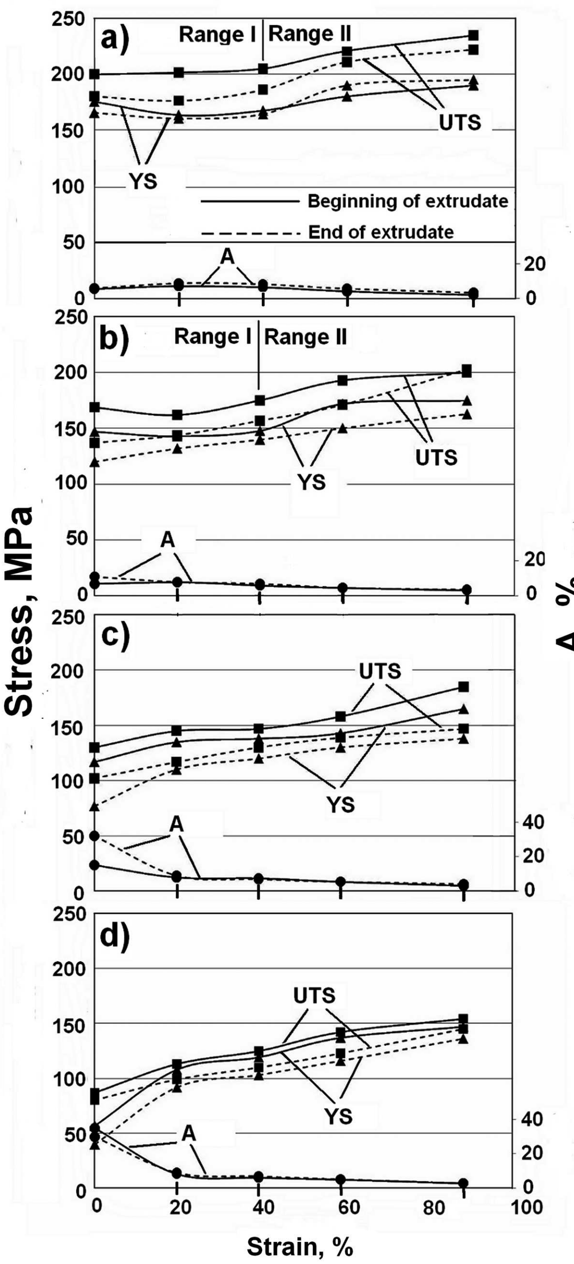

Fig. 5. Work hardening curves of groove rolled aluminum wires extruded by means of KoBo method at following processing parameters: (a) T = 293 K, v = 0.1 mm/s, f = 3 Hz; (b) T = 373 K, v = 0.5 mm/s, f = 3 Hz; (c) T = 493 K, v = 0.5 mm/s, f = 5 Hz; (d) T = 673 K, v = 0.5 mm/s, f = 5 Hz

The most important observation is that varied KoBo extrusion parameters leads to the fundamental differences in the work hardening characteristics shown in Fig. 5. Despite some exceptions to the rule, one can generally state that the wires extruded from billets with low initial temperature ($\leqslant$ 493 K at v = 0.1 and 0.5 mm/s) exhibit two-range work hardening curves (Fig. 5a,b) independently of a die oscillation frequency. The first hardening range (Range I, $\varepsilon = 0 \div 40\%$), does not reflects the typical cold strain hardening curve for aluminum. Range I correspond to the lack of strengthening processes (even small increase in plasticity is observed) and a weak work hardening appears for $\varepsilon > 40\%$ (Range II in Fig. 5). Two-range of work hardening behavior of high strength aluminum KoBo extrudate can be discussed on the basis of TEM observation results. Highly recovered substructure and a very low dislocation density is typical for the aluminum wire extruded by KoBo method at T = 373 K, v = 0.1 mm/s, f = 3 Hz as well as for the end part of the wire additionally cold rolled to the end of Range I (ε = 40%) (Fig. 6a,b). At larger strains, corresponding to Range II, both highly recovered subboundaries and dislocation tangles were observed (Fig. 6c,d). For comparison, structure of conventionally hot extruded and cold rolled wires, is shown in Fig. 7a,b and 7c,d, respectively. The observed (work hardening) behavior of rolled KoBo extruded aluminum wires is strictly connected with the presence of overbalance of point defects in tested materials [7].

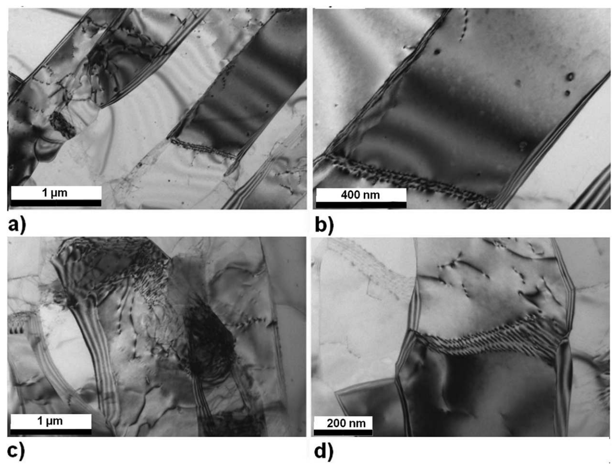

Fig. 6. TEM microstructures of the beginning of aluminum wire extruded by means of KoBo method (T = 373 K, v = 0.1 mm/s, f = 3 Hz), then cold rolled with cross-section reduction of 40% (a and b) and 70% (c) and (d). (The end of Range I and Range II marked in Fig. 5b, respectively)

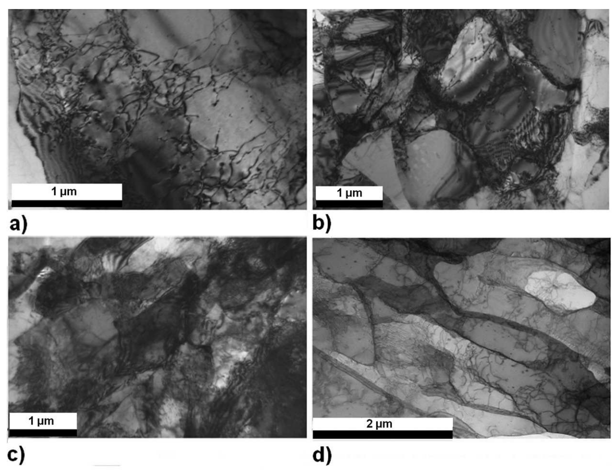

Fig. 7. Microstructure of conventionally hot extruded aluminum wire subjected to rolling with cross-section reduction of 40% (a) and (b) and 70% (c) and (d)

Due to increased density of point defects in rolled aluminum the selfdiffusion processes are very fast within Range I that result in intense recovery process and consequence in lack of work hardening effects. As the excess of point defects is reduced during rolling in Range I due to their absorption at dislocations and grain boundaries, strength properties at the Range II start again to increase with strain. If assume that the point defects density rises due to cumulated torsion / extrusion deformation at KoBo method, it seems reasonable to expect that following cold rolling of as extruded material result in strain hardening characteristics, which are different from these received for conventionally hot extruded aluminum.

4. Discussion

TEM observations performed on aluminum wires extruded by KoBo method from the billets with low initial temperature revealed highly recovered subboundaries and specific fine-modulated structure in subgrain interiors (Fig. 4). Fine dotted contrast was found to appear at $< 111 > \bar{g}$ and $< 200 > \bar{g}$ vectors diffraction conditions (Fig. 4b). As discussed previously, specific diffraction contrast at numerous subgrains interiors (Fig. 4b) suggest the development of point defects (clusters) induced by heavy deformation of KoBo extruded material (Table 2). The overbalanced density of point defects may be responsible for accelerated recovery at initial cold rolling passes and reduction of the strain hardening effect through the Range I marked in Fig. 5a,b. Contribution of point defects to diffusion controlled structural processes, including plastic flow of metals and alloys, has been widely discussed for over sixty years and still take an interest of researchers. It is well known, that the equilibrium concentration of point defects in the metallic materials is relatively small. Its maximum value occurs close to the melting point temperature and is estimated to the value of $\sim 10^{-4}$ for vacancies and a few orders of magnitude less for atoms at interstitial positions. It is related to the value of energy needed for the generation of mentioned point defects that can be estimated as about 1 eV for vacancies and at least three times higher for interstitial atoms [16, 17].

It is known, that overbalance concentration of point defects, both vacancies and own interstitial atoms (Frenkel defects) can be also attained if a high energy bombardment of the material using protons or neutrons is applied. TEM observations confirmed the development of large amounts of point defects in the form of condensates (clusters), which play a meaningful role in strengthening of materials [18]. Experiments performed on materials heavy deformed by means of severe plastic deformation methods demonstrate similar behavior as mentioned above for irradiated materials [18, 19]. However, in this case, the deformation characteristics are directly linked by the authors with observed efficient of grain size reduction rather than point defects even if their high concentration can be expected highly deformed materials. The common denominator of both types of experiments may be, however, that a significant part of the atoms is located in the boundary areas, which from the crystalline structure point of view can be considered as extremely defected areas and thus with high concentration of point defects.

Arguments considered above are important for understanding relatively high intensity of the recovery process at KoBo extruded aluminum and related effect of post-extrusion cold rolling on mechanical properties. Effect of point defects development due to severe deformation of KoBo extruded materials and their contribution to the hardening was formerly discussed by Korbel and Bochniak [7]. However, the problem is practically not raised in concurrent literature on the extrusion method. Therefore, described above structural and mechanical effects of KoBo extrusion had to be considered with respect to the wide review of literature data presented above. Nevertheless, further careful structural analyses are necessary to explain the origin of very fine contrast dots in KoBo extruded wires and specific hardening characteristics for cold rolled extrudates.

5. Summary

Application of KoBo method to the processing of aluminum billets allows receiving a wide spectrum of mechanical properties at varied electrical resistivity of as extruded wires that can be controlled using extrusion parameters such as the billet initial temperature, cross-section reduction ratio, extrusion velocity and the die oscillation frequency. At particular extrusion conditions, in spite of intense recovery of the material at low deformation temperature, very high strength properties are received. High strength properties of aluminum, accompanied by low tension elongation values and relatively high electrical resistivity of extrudates were received if low extrusion rate and a die frequency parameters and the billet initial temperature 293 – 493 K was used. Moreover, following cold rolling of the extrudate results in the lack of strain hardening of the material up to strain $\varepsilon = 40\%$.

It is worth stressing that aluminum extrudates received at higher preheating temperatures and higher values of the frequency of die oscillation, demonstrates similar behavior at following processing (groove rolling) as it is observed for conventionally hot extruded wires. In particular, the materials are characterized by relatively low electrical resistivity, low strength properties and increased tension elongation values.

Acknowledgements

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