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# STRUCTURE AND MECHANICAL PROPERTIES OF AIMg4.5 AND AIMg4.5Mn WIRES EXTRUDED BY KoBo METHOD

## STRUKTURA I WŁASNOŚCI MECHANICZNE DRUTÓW AIMg4.5 I AIMg4.5Mn OTRZYMANYCH METODĄ KoBo

The influence of the number of extrusion steps in KoBo method (at the same total extrusion ratio of  $\lambda = 100$ ) on structure, mechanical properties and work hardening characteristics of AlMg4.5 and AlMg4.5Mn (AA5083) alloys was investigated. It was found that one-step extrusion leads to the formation of recrystallised structure of the material, while the use of two-step extrusion yields a fibrous structure of a "mixed" type, i.e. containing areas where the intensive recovery effects are associated with partially recrystallised structure. As a consequence, the strength properties of the latter extrudate are much higher in both as extruded state and after the subsequent cold rolling. In all cases, the tensile stress-strain curves of the extrudates show the flow stress serrations that are typical for the Portevin – LeChatelier (P-L) effect. In a few tensile tests, the P-L effect was preceded by the plastic flow instability being typical for the occurrence of Lüders bands. Both AlMg4.5 and AlMg4.5Mn extruded wires show a monotonic increase of the work hardening that results from the following cold deformation in the groove rolling.

Keywords: aluminium alloys, extrusion, KoBo method, structure, mechanical properties, work hardening

W pracy badano wpływ liczby operacji wyciskania metodą KoBo (z identycznym sumarycznym stopniem przerobu  $\lambda$  = 100) na strukturę, własności mechaniczne i charakterystyki umocnieniowe drutów ze stopów AlMg4,5 i AlMg4.5Mn (AA5083). Stwierdzono, że wyciskanie jednooperacyjne prowadzi w przypadku obu stopów do formowania struktury typowej dla materiałów zrekrystalizowanych, podczas gdy zastosowanie dwuoperacyjnego wyciskania skutkuje utworzeniem struktury włóknistej o charakterze "mieszanym", tzn. zawierającej zarówno obszary, w których dominowały procesy intensywnego zdrowienia, jak i rekrystalizacji. W konsekwencji własności wytrzymałościowe tych ostatnich są zdecydowanie wyższe zarówno po wyciskaniu, jak i po późniejszym walcowaniu na zimno. We wszystkich przypadkach na krzywych rozciągania drutów obserwowano skokowe oscylacje naprężenia, charakterystyczne dla efektu Portevin – LeChatelier (P-L). W nielicznych próbach rozciągania efekt P-L poprzedzała niestateczność płynięcia plastycznego typowa dla występowania pasma Lüdersa. Charakterystyki umocnienia wywołane procesem walcowania wykazywały przebieg monotonicznie rosnący.

## 1. Introduction

The KoBo extrusion method, used in experiments, consists in the extrusion with reversibly oscillating die. The method allows "cold" extrusion, (i.e. without the need of billet preheating), that usually results in high strength properties of extrudates, often unavailable by the conventional extrusion process [1, 2]. The effect of KoBo extrusion parameters on the mechanical properties of aluminium was analysed in detail in paper [3]. It has been shown that only in the case of extrusion of the billet with a low initial temperature, carried out at a low rate and/or at a low die oscillation frequency, it is possible to manufacture products with a dynamically recovered fibrous structure. Such extrudate is characterised by relatively high strength properties, which are stable up to ~473 K and usually accompanied by elevated electrical resistance values. These features of material properties have been used as essential arguments in a discussion on the presence of overbalance

concentration of point defects in KoBo extrudates (probably in the form of clusters). The above mentioned defects may arise as a result of severe plastic deformation induced in the condition of the change of deformation paths during the extrusion combined with reversible torsion of the material [3]. Moreover, it was found that the spontaneous rise of billet temperature occurring during the extrusion process (even up to  $\sim$ 573 K [4]) does not significantly change the structure of the extrudate and to a lesser extent, (i.e. by not more than 20%), reduces the strength properties in the end part of the product. The increase of initial temperature of the billet above ~493 K, as well as an increase of the extrusion rate and, particularly, an increase of the die oscillation frequency, can stimulate the structure-forming processes leading to a significant decrease of the mechanical properties. It is commonly believed that aluminium and its alloys belong to the group of metals undergoing only dynamic recovery while the material is deformed [5]. However, at high enough strain, the process of intensive

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dynamic recovery leads to the formation of wide-angle boundaries and, as a consequence, the observed structure becomes composed of equiaxed grains, which are typical for the dynamic recrystallization processes [6]. It can be concluded that, especially under the conditions of severe plastic deformation induced by KoBo extrusion, temperature of the shaped product can rise enough to generate some structural changes indicating the occurrence of dynamic recrystallization.

The experiment described in [7, 8] has shown the structural and mechanical effects that prove the limitation of the spontaneous increase of the billet temperature if the KoBo extrusion is realised in more than one step. The extrusion process carried out with a much lower deformation intensity allows obtaining the strength properties higher by at least 10% for the products extruded with similar value of the total extrusion ratio.

The aim of this study was to answer the question whether a similar effect of the increase of strength properties can be obtained in the case of hardly deformable aluminium alloys of an Al-Mg type, in which the high-temperature deformation promotes the formation of the dynamically recovered and/or dynamically recrystallised structures [9-12].

# 2. Experimental

The investigations were carried out on two alloys of the following composition (in wt%): AlMg4.5% and AlMg4,5%Mn0.85% (AA5083), which further in this study were denoted as AlMg4.5 and AlMg4.5Mn, respectively. Billets of Ø40×60 mm used for KoBo extrusion of AlMg4.5Mn wires were machined from hot extruded rods of Ø40 mm diameter. The AlMg4.5 alloy billet was composed of hot-extruded flat bars 40×14 mm in cross-section dimensions, from which rings of Ø40×14 mm were turned and put in a stack (package) about 60 mm high. The billets of both types were extruded by KoBo method using a hydraulic press operating under maximum pressure of 1 MN. The punch rate was 0.5 mm/s, the frequency of die oscillations was 5 Hz, and the oscillation angle was  $\pm 8^{\circ}$ . Both die and the extrudate were intensively cooled with water. The initial billet temperature was 293 K. The extrusion was carried out using extrusion ratio  $\lambda = 100$ (true strain  $\varepsilon_t = 4,6$ ) for one-step extrusion ( $\emptyset 40 \rightarrow \emptyset 4$ ) and  $\lambda_1 = 11$  ( $\varepsilon_t = 2,4$ ) and  $\lambda_2 = 9$  ( $\varepsilon_t = 2,2$ ) for two-step extrusion of the wires ( $\emptyset 40 \rightarrow \emptyset 12 \rightarrow \emptyset 4$ ). It is worth stressing that the corresponding effective true strain is much higher than the values mentioned above, if an additional non-dilatational strain induced by oscillatory shearing of the extruded material is taken into consideration.

The obtained wires were sampled in the beginning and end section of the product (designated as B and E, respectively). Since in industrial practice the extrudates are usually subjected to further processing at room temperature, such as cold rolling or drawing, the additional experiments were performed on the wires cold rolled using shape milling in a square/square scheme. The purpose of this test was to obtain information on the possibilities of further plastic forming of the extrudates as well as to examine the effect of extrusion mode i.e. one-step or two-step extrusion on the work hardening of the material. The wires in both as-extruded state and after additional cold rolling were tested by means of a static tensile test using a Zwick / Roell Z050 tensile testing machine operating at a strain rate of  $8 \cdot 10^{-3} \text{ s}^{-1}$ . The sample gauge length and diameter were 50 mm and 4 mm, respectively. The following properties were determined: yield strength (YS), ultimate tensile strength (UTS) and total elongation (A).

To evaluate the thermal stability of the structure and mechanical properties of the obtained wires, tests of one-hour annealing at various temperatures in the range of up to 773 K were performed. Hardness of the samples was measured with a Shimadzu HMV device. For comparison purposes, hardness tests were also performed for samples cut out from the billet, where the samples were next cold rolled to a 50% of strain.

Structure examinations were carried out under a Nikon optical microscope on both transverse and longitudinal sections of the billet, and on the longitudinal sections of the obtained wires. The metallographic specimens were mechanically grinded and polished and were next treated by electropolishing in 80% ethanol and 20% perchloric acid solution combined with anodic oxidation in Barker's reagent. TEM studies were performed using a JEM2010 microscope with an accelerating voltage of 200 kV, equipped with a scanning transmission electron microscopy device (STEM).

### 3. Results and analysis

# 3.1. Structure of billet

Microstructure of billets used in the KoBo extrusion process is shown in Fig. 1. The structure of the AlMg4.5 billet is characterised by equiaxial recrystallised grains of an average size of ~45  $\mu$ m (Fig. 1a,b). On the other hand, the "fibrous" structure of AlMg4.5Mn billet is typical for the dynamically recovered material deformed at high temperature, but without the visible symptoms of recrystallisation (Fig. 1c,d).



Fig. 1. The microstructure of alloy billet: a) AlMg4.5 – cross-section;
b) AlMg4.5 – longitudinal section;
c) AlMg4.5Mn – cross-section;
d) AlMg4.5Mn – longitudinal section

## 3.2. Mechanical properties of alloys after extrusion

The stress-strain curves received for as extruded wires from the AlMg4.5 and AlMg4.5Mn alloys are shown in Fig. 2a and Fig. 2b, respectively. In each case, characteristic serrations on the plastic flow curves point to a non-uniform deformation of tested material, that is typical for the Portevin – LeChatelier effect. Some attention deserve significant variations in the image of this effect as regards both the number and nature of load oscillations, but it is difficult to reasonably attribute it to the type of billet used or to the applied mode of extrusion.

The beginning sections of the AlMg4.5 wire (B) and the end sections (E) of the AlMg4.5Mn wire obtained in a two-step extrusion are characterised by slightly higher frequency of the flow stress serration – the phenomenon is particularly well-visible in Fig. 2b (curve 4). On the other hand, the largest amplitude of the flow stress oscillations (reaching up to 20 MPa, i.e. 6% of initial stress level) was observed in the beginning (B) sections of AlMg4.5 alloy and in the end (E) sections of AlMg4.5Mn alloy extruded in a two-step processing of the wire (Fig. 2, curves 3 and 4, respectively). In some samples of wires extruded in a one-step processing, the presence of Lüders phenomenon was also reported (Fig. 2, curves 1 and 2). The last effect – despite the similarity of grain size – is in contrast with the behaviour of AlMg4.5 samples deformed by tension as mentioned in [13].

Mechanical properties of as extruded wires are shown in Fig. 3. It is easy to note that what makes the difference is, first of all, the number of extrusion operations, followed by the alloy chemical composition and a section of the wire where the sample has been taken. Wires extruded by one-step processing from AlMg4.5 alloy reach the yield strength of ~140 MPa, the tensile strength of ~280 MPa, and elongation of nearly 40%. The AlMg4.5Mn alloy extruded in a similar manner has nearly the same value of yield strength, the tensile strength UTS higher by 20 MPa, and the elongation A reaching a value of 28% in the beginning section of the wire and rising to 40% at its end. The two-step extrusion leads to a noticeable increase in the strength of the wires. The increase in YS is much higher than in UTS (by about 30% and 10%, respectively). The highest mechanical properties were reached by the beginning section of the wire made from an AlMg4.5Mn alloy (YS ~280 MPa, UTS ~370 MPa), with a slightly lower elongation of A  $\sim$ 20%. (Fig. 3). It is worth to note that the addition of Mn has practically no influence on the as extruded mechanical properties of the tested wires.



Fig. 2. Tensile stress-strain curves: a) AlMg4.5 wire; b) AlMg4.5Mn wire; the extrusion mode and the location of the sample taken from as extruded wire are marked in the legend



Fig. 3. Mechanical properties of AlMg4.5 and AlMg4.5Mn wires extruded by KoBo method. B, E – samples selected from the beginning and end part of as-extruded wire, respectively

### 3.3. The structure of wires

The results of optical microscopic observations are documented in Fig. 4. It is evident that, regardless of the alloy type, the structure of all wires extruded in a one-step processing is composed of equiaxed grains of the size of about 25  $\mu$ m (AlMg4.5 alloy) and about 40  $\mu$ m (AlMg4.5Mn alloy) (Fig. 4a,c). The structure of two-step extruded wires can be described as "mixed", i.e. having the characteristics of a fibrous structure typical of the deformed material. An important element of such structure is the presence of bands composed of very fine equiaxed grains (areas indicated by arrows in Fig. 4b,d). Such differences in the structure may result from a non-uniform (laminar) flow of materials during the extrusion process, which is the reason why these materials respond in a different way to the dynamic processes of structure renewal (combined effects of both dynamic recovery and local recrystallisation of the material).



Fig. 4. Microstructure of AlMg4.5 (a, b) and AlMg4.5Mn (c, d) wires extruded by KoBo method in one-step processing (a, c) and two-step processing (b, d)

Figs. 5 and 6 show the examples of STEM and TEM structure micrographs, which further document the effect of the number of extrusion operations on the structure of the material. An average subgrain size of about 6  $\mu$ m and very

low dislocation density was observed in AlMg4.5Mn wires extruded in a one-step processing. In some subgrains one can observe the elevated dislocation density in the subgrain interior (Figs. 5a and 6a-c). Both the low dislocation density and well-arranged dislocations in subgrains prove the dynamic recovery and recrystallisation processes occurring simultaneously during the extrusion in different parts of the material. Unlike the material extruded in one operation, the structure of the two-step extruded wires is characterised by the development of much smaller subgrains, less than 1  $\mu$ m in size. Moreover, owing to a high degree of the dislocation ordering in subgrain boundaries, it can be concluded that an intensive process of the dynamic recovery has been the dominant structure restoration mechanism (Figs. 5b and 6d-f).



Fig. 5. STEM microstructures of AlMg4.5Mn wire extruded by KoBo method in: a) one-step processing; b) two-step processing



Fig. 6. TEM microstructures of AlMg4.5Mn wire extruded by KoBo method in one-step processing (a-c) and two-step processing (d-f). Bright field image (a, c, d, e) and dark field image (b, f) microstructures. Were received using operating vector  $\bar{g} < 111 >$ , as marked in the figure

# 3.4. Thermal stability of mechanical properties

Significant variations in the structure and mechanical properties of AlMg4.5 and AlMg4.5Mn wires, depending on the number of extrusion operations, raise some questions with

regard to their stability at elevated temperatures. The results of experiments on the influence of annealing temperature on hardness of alloys, tested in both the as extruded state and after the additional cold rolling to a 50% of strain, are shown in Fig. 7. All one-step extruded wires are characterised by relatively low hardness value (75-85HV), which is only slightly reduced after high-temperature annealing (Fig. 7a). Wires with high hardness, extruded in a two-step processing, undergo softening due to static recovery and recrystallisation processes, that become much more effective during annealing above 473 K (Fig. 7b). The course of hardness vs. annealing temperature curves is close to similar annealing characteristics obtained in a material subjected to additional deformation by cold rolling (Fig. 7c). It is easy to note that, compared with the AlMg4.5 alloy, hardness of AlMg4.5Mn samples remains higher by about 10 units, which may result from the generally known effect of Mn addition on combined solution and dispersion hardening (Al<sub>6</sub>Mn precipitates) [14, 15].



Fig. 7. Annealing temperature vs. hardness of the AlMg4.5 and AlMg4.5Mn alloys extruded by KoBo method in: a) one-step processing; b) two-step processing; c) samples deformed with strain 50% using groove rolling at room temperature; the extrusion mode and the location of the sample taken from as-extruded wire are marked in the legend

#### 3.5. Work hardening of cold rolled wires

The work hardening characteristics of the examined AlMg4.5 and AlMg4.5Mn wires cold rolled in grooves are shown in Figs. 8 and 9, respectively. There is a monotonic increase of strength and efficient reduction of the elongation value (A), that is typical of the early stage of work hardening. The diagrams show that differences in the mechanical properties of wires extruded in a one-step or two-step processing observed in the as-extruded state are preserved also during successive cold rolling passes. Consequently, wires extruded in a two-step processing, rolled up to 90% of strain, achieve YS ~420 MPa, UTS ~530 MPa and A ~2%, which gives results similar to those stated in [16] for the cold rolled AlMg4.5 alloy.



Fig. 8. Effect of cold rolling on mechanical properties of AlMg4.5 wires; the extrusion mode and the location of the sample taken from as-extruded wire are marked in the legend

It can be concluded that, regardless of the type of alloy used and the mode of extrusion, wires subjected to rolling to 90% deformation show an increase in YS and UTS by more than 50% and 35% with respect to as-extruded materials (Figs. 8 and 9). Differences in the strength properties observed in as-extruded wire between the beginning and end section of the extrudate are gradually disappearing under the cold rolling with the strain above  $\sim 70\%$ .



Fig. 9. Effect of cold rolling on mechanical properties of AlMg4.5Mn wires; the extrusion mode and the location of the sample taken from as extruded wire are marked in the legend

#### 4. Discussion

The results of studies of the AlMg4.5 and AlMg4.5Mn alloys extruded in a one-step or two-step processing by KoBo method show that it is possible to form different type of structure and, consequently, make products with different mechanical properties. Experiments performed on hardly deformable AlMg4.5 and AlMg4.5Mn alloys subjected to the "cold" extrusion (the initial billet temperature of 293 K) demonstrate the possibility of the formation of varied structures, which depend on a number of extrusion results in the material structure that is typical for materials undergoing dynamic recrystallisation, being responsible for the equiaxed shape of grains/subgrains. In the two-step extrusion process, structure

of tested materials is mostly formed by the intensive process of dynamic recovery. However, colonies of very fine grains along extrusion direction point evidently to the development of dynamic recrystallisation, which accompanies the effective dynamic recovery of extruded alloys. With the given parameters of extrusion, i.e. the extrusion rate of 0.5 mm/s, the die oscillation frequency of 5 Hz, the oscillation angle of  $\pm 8^{\circ}$ and the total extrusion ratio  $\lambda = 100$ , the alloys subjected to one-step extrusion have low strength properties and relatively high ductility (Figs. 3 and 4a,c). The use of a two-step extrusion has considerably increased the strength properties (YS and UTS by 30% and 10%, respectively) with the accompanying reduction of the elongation by about 8%. The above mentioned effects result from the formation of a fibrous structure composed of the areas arranged in layers, characteristic of the structural changes induced by dynamic recovery and locally occurring recrystallisation (Figs. 3 and 4b,d).

It was reported that the reduction in extrusion rate and in the die oscillation frequency provides a structure formed solely by the dynamic recovery. Obtained in this way relatively high strength properties may be due also to an additional effect, which is the formation of excess point defects, as suggested in [3]. However, the TEM structure examinations do not confirm the presence of these structural features, even at high magnifications and under selected conditions of the diffraction contrast (Fig. 6). TEM images of the AlMg4.5Mn structure of wires extruded in a one-step processing reveal a low dislocation density inside the grains (Fig. 6a,b) and incidentally observed clusters of much smaller subgrains as shown in Fig. 6c. The structure variations presumably result from the intensive dynamic recovery (fine subgrains) and grain growth by recrystallisation (colonies of coarse equiaxial grains). The density of dislocations in the same material but extruded in a two-step processing (Fig. 6d-f) is significantly higher, which corresponds well with relatively high strength of the material (Fig. 3). A large number of the subgrain boundaries formed by densely arranged and evenly distributed dislocations (Fig. 6f) proves the occurrence of high-intensity dynamic recovery with very limited recrystallisation, operating both in dynamic conditions (in the zone of plastic flow during extrusion) and static conditions (after deformation).

Systematic studies of the effect of KoBo extrusion parameters on the properties of "cold" extruded aluminium showed that the reduction in extrusion rate and die oscillation frequency to 0.1 mm/s and 3 Hz, respectively, resulted in an increase of YS and UTS by about 15% compared to the material which was extruded at a relatively higher rate (0.5 mm/s and 5 Hz) [3]. Unfortunately, maintaining the same conditions during the extrusion of the AlMg4.5 and AlMg4,5Mn alloys was not possible as it exceeded the available press capacity. But apart from this aspect of the experiment, it is worth paying attention to some of the results presented, in particular to the flow characteristics of tested AlMg4.5 and AlMg4.5Mn extrudates. First of all, rather surprising may seem the fact that the P-L effect has occurred in each of the extruded AlMg4.5 and AlMg4.5Mn wires despite significant differences in their structure. The Lüders effect occurred only in the wires obtained in one-step extrusion process, i.e. in the material with low value of YS having a structure formed by dynamic recrystallisation (Fig. 2). On the other hand, Lüders effect was not observed in the AlMg4.5 alloy, which was cold deformed and subjected to recrystallising annealing [13]. In the case of aluminium, Lüders effect was also observed to occur in extrudates whose structure was formed under the conditions that induced very intensive dynamic recovery [17], but the products did not exhibit particularly high strength properties.

Another aspect, which requires some comments, is related with the work hardening characteristics of the tested AlMg4.5 and AlMg4.5Mn alloys resulting from the hardening by rolling (Figs. 8 and 9). In each case, the strength properties (YS, UTS) monotonically increase with increased rolling strain, preserving the initial differences observed in as extruded state. Consequently, in the one-step extruded wires, the values obtained for YS and UTS are ~360 MPa and ~480 MPa, respectively, whereas in the two-step extruded wires these values may reach the level of even YS ~400 MPa and UTS ~530 MPa (Figs. 8 and 9). Thus, the work hardening characteristics described above are significantly different from those observed in KoBo extruded aluminium wires [7]. The aluminium wires with high mechanical properties, cold deformed by the following cold rolling, exhibit in the initial range, i.e. up to ~40% of strain, total lack of the hardening effect that sometimes even enters the work softening range. These effects can be attributed to the presence of excess point defects (probably clusters) formed during extrusion by KoBo, leading next to the localisation of deformation and intensification of recovery effects in shear bands [3, 7].

The third aspect relates to the formation of a mixed structure, partly polygonised and partly dynamically recrystallised in the case of AlMg4.5 and AlMg4.5Mn alloys processed in two-step extrusion by KoBo method. With respect to commonly manufactured products, the resulting materials exhibit relatively high thermal stability of both the structure and properties. Practically no changes in these properties were observed up to about 473 K (Fig. 7). This effect, characteristic also for other products extruded by KoBo method, can be used in the development of a technology for the manufacture of products that are designed to operate at elevated temperatures for example, up to ~473 K in case of the tested materials.

# 5. Conclusions

- 1. High extrusion ratio,  $\lambda = 100$ , used in one-step extrusion of tested alloys promotes the formation of uniaxial grains that result from the dynamic recrystallisation rather than from the recovery process. At the same total extrusion ratio, two-step extrusion leads to formation of "mixed" fibrous type structure. With respect to one-step extrusion, relatively high strength and reduced plasticity of the extrudate were received. Moreover, the properties were found to be stable for samples annealed up to 473 K.
- 2. In contrast to the high effect of varied KoBo procedures on the material structure (i.e. one step or two-step extrusion), the received structure of wires was not dependent on the different initial structure of used billets. The last effect can be ascribed to very high effective processing of the KoBo extruded materials.
- 3. Regardless of the type of structure formed in KoBo-extruded AlMg4.5 and AlMg4.5Mn wires each ex-

trudate exhibits the tendency to heterogeneous deformation typical for the Portevin – LeChatelier effect, while serrated flow stress curves are observed. Non-uniform plastic flow in the form of Lüders band was additionally observed in only one-step extruded wires.

4. Experiments performed on wires cold rolled after KoBo extrusion revealed that the initial difference in the materials strength (YS and UTS), obtained in one-step and two-step extrusion, persist up to the cross-section reduction by cold rolling equivalent to 90% of strain.

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