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AA6013 aluminium alloy deformed by forward-backward rotating die (KoBo): Microstructure and mechanical properties control by changing the die oscillation frequency

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ABSTRACT

The precipitation hardened AA 6013 aluminium alloy was extruded by means of the KoBo extrusion method with an oscillating die. In the presented paper, the influence of two different die rotation frequencies on the microstructure and mechanical properties after KoBo deformation and during post-deformation ageing process is discussed. Initial materials were conventionally hot extruded rods. The extrudates were heat treated to T6 condition and subsequently deformed using the KoBo method, at two frequencies: 2.5 and 8 Hz. The obtained samples were characterised by static tensile tests, scanning and transmission electron microscopy (SEM and TEM), and differential scanning calorimetry (DSC). Electron back scattered diffraction (EBSD) in SEM was applied in order to verify grain size, density of boundaries and local texture. TEM and DSC were used to analyse the influence of plastic deformation on the precipitation process. The results revealed that die oscillation frequency has a great influence on the behaviour of materials during ageing.

1. Introduction

The influence of severe plastic deformation (SPD) on microstructure and mechanical properties of aluminium alloys has recently been studied intensively (Avtokratova et al., 2016). The KoBo extrusion method represents a new approach to this subject. The KoBo device is a press with a forward-backward rotating die, enabling extrusion of ingots under conditions of constant destabilisation of their microstructure (Bochniak et al., 2005). Each of the five process parameters (temperature, extrusion ratio, extrusion rate, angle and frequency of oscillations) can exert influence on mechanical properties and microstructure. This influence should be considered in order to obtain desirable results (Pieła et al., 2013).

The great advantage of the process compared to classical SPD and extrusion methods is to obtain the product in a single step (Bogucki et al., 2015), at room temperature and with properly reduced cross sections, even for hardly deformable materials (Korbel and Bochniak, 2004). It is believed that, during deformation, a new mechanism of plastic formation called visco-plastic flow occurs (Korbel et al., 2011). The axial-radial flow typical for the extrusion process is replaced by layer-like radial flow. Researchers emphasise that, during plastic flow, the relation between stress and strain rate is linear, where the viscosity coefficient is the proportionality factor. The proposed mechanism is sensitive to the occurrence of point defects, especially vacancy and self-interstitial ones. In the materials processed to high strains by reversible, monotonic extrusion, the generation of defects may lead to an overbalanced state, greatly exceeding equilibrium values. High contention of point defects affects the viscosity coefficient (Kawasaki et al., 2011). Radial flow dominates in the deformation zone, while the zone shape is similar to a cylinder with a base diameter equal to the diameter of the batch. Therefore, during the KoBo extrusion dead zone, a region where material does not flow, is not apparent, as has been confirmed by modelling of the process (Maciejewski and Mróz, 2008). The KoBo method has in recent years been applied to various materials, especially those such as zinc (Kawałko et al., 2016) and titanium (Sztwiertnia et al., 2015), which are difficult to deform, but also for pure aluminium (Bieda et al., 2016) and its alloys (Korbel et al., 2015). This work deals with the influence of the KoBo method on the precipitation processes in

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6013 aluminium alloy. The AA6013 alloy belongs to the 6XXX series, with magnesium and silicon addition. In comparison to other alloys of this series, AA6013 contains more copper, which enhances strength properties (Nafsin and Rashed, 2013). AA6013 exhibits superior mechanical properties, density and low production costs (Carbonini et al., 1997). Alloys of the 6XXX series belong to precipitate hardening materials. Strengthening is obtained by heat treatment and a subsequent ageing process that ensures elevation of properties.

Much attention has recently been devoted to researching the influence of plastic deformation after heat treatment on the post-deformation precipitation process. However, this issue has still not been examined sufficiently for 6XXX alloys. It is claimed that Q' and β" particles could be formed on dislocations, as the preferred sites of nucleation. Therefore, the density of dislocations is a significant parameter that has great influence on further heterogeneous nucleation. Change of shape by plastic deformation is always connected with increase of defects. Thus, the number of preferable places for nucleation also increases incrementally, as long as the deformation temperature is below the temperature of microstructure recovery processes. On the other hand, with raised temperature the dynamic precipitation process and/or acceleration of new nucleation sites in-post deformation heat treatment can take place (Nageswara et al., 2014). Properly chosen deformation and heat treatment temperature could provide preferable mechanical properties (Wu et al., 2014). Unfortunately, the temperature of the KoBo process cannot be measured directly in the deformation zone because of the complicated construction of the press. Therefore, evaluation must be focused on material response after deformation.

This work is focused on the influence of one KoBo extrusion parameter, die oscillation frequency, on the microstructure and precipitate kinetic and mechanical properties of AA6013 aluminium alloy. In the literature are references dealing with the specific behaviour of AA7075 aluminium alloy capable of being age-hardened. In the described case, the process resulted in a stable material, insensitive to post-deformation ageing and typically capable of precipitate hardening by a change of extrusion parameters (Korbel et al., 2015). Thus, it was decided to verify the influence of this same parameter of KoBo deformation on behaviour in the 6XXX aluminium series, and to examine the cause of such changes.

2. Experimental procedure

The AA6013 aluminium alloy was received as conventionally extruded rods with diameter of $\phi = 40$ mm. The scheme of the experimental procedure is presented in Fig. 1. Rods were heat treated before deformation. They were supersaturated at 530 °C for 1 h with quenching to water, followed by artificial ageing at a temperature of 165 °C for 2 h. The heat-treated batch was deformed by the KoBo method (Fig. 2). The KoBo process was performed under the following conditions: punch speed = 0.1 mm/s, die rotation angle = ± 8°, initial process temperature = 24 °C, extrusion ratio $\lambda = 100$ (which means that the diameter of the rods after the KoBo process was equal to $\phi = 4$ mm). In this work, the authors focused on impact of die rotation frequency on microstructure and material properties after deformation and during post-deformation ageing process. Two frequencies, 2.5 and 8 Hz were applied. Samples for investigation were taken from the end parts of the received rods. Static tensile tests were performed using a ZwickRoell Z050 machine, at a constant deformation speed of 8×10^3 1/s. The samples had following dimensions: base length $L_0 = 50$ mm, base diameter $\varphi = 4$ mm. For each research step, three samples were prepared. An FEI Quanta 3D FEG scanning electron microscope equipped with the EDAX OIM TSL EBSD system was used for microstructural characterisations. The EBSD measurements were performed with accelerating voltage of 20 kV and step size of 150 nm. The TSL OIM Data Analysis 7.2 program was used for investigating collected sets of EBSD patterns. For calculations, only grains with image quality higher than 5×10^5 were taken into account. This procedure filtered badly indexed points appearing on orientation maps, and gave information about grain sizes, density of boundaries and texture. Grain size was calculated as a diameter of a circle of the same area as the measured grain. The presented values are the weighted average, where weight is grain area. The density of boundaries was calculated as a grain boundary line divided by the area of a map. Intensity of ⟨111⟩ fibre texture was determined from (111) pole figures. Samples for SEM were ground on sandpaper up to 5 000 grit and polished with diamond suspension liquids up to 1 μm. Finally, samples were electro-polished using a solution of 10% per chloric acid in ethanol, to obtain perfect flat surface. A Tecnai G2 F20 transmission electron microscope was used for microstructure observations and analysis of precipitations. Samples for TEM were prepared on a twin jet electro polisher using a solution of nitric acid and methanol at −22 °C. Differential scanning calorimetry (DSC) was performed on a DuPont 910 calorimeter. Samples were cleaned with an ultrasonic wave pool before examination. Specimens were heated from 20 °C to 600 °C, with a heating rate 20 °C/min, under an argon atmosphere. Two cycles of heating were performed for each sample.

3. Results

3.1. Mechanical properties

The mechanical properties of the initial material and after applying KoBo with different parameters of extrusion appear in Fig. 3. In general, extrusion with added torsion motion enforcing permanent change in the deformation path led to improved strength properties. Die rotation frequency at 2.5 Hz raised tensile yield strength by about 40 MPa at the expense of plasticity. After 2 h of ageing at 165 °C, the mechanical properties of the material had stabilised at similar levels. Completely different behaviour was observed for material after applying KoBo at a die rotation frequency of 8 Hz. Acceleration in the periodicity of the deformation path led to the improvement of all mechanical parameters. The material preserved the tendency to precipitation strengthening. Ageing at 165 °C for 2 h resulted in maximum strength properties. It is evident that a change in deformation path rate can be considered as a crucial parameter affecting material properties.

3.2. Microstructure

Observations of microstructure were performed to explain discrepancies between the ageing behaviour of deformed material at two different die rotation frequencies. The samples were studied immediately after KoBo extrusion, and then after the subsequent ageing process for which maximum values of strength were obtained. Materials in such states were analysed in order to correlate microstructure with mechanical properties. EBSD maps were collected from transverse and longitudinal cross sections of the rods. Fig. 4 presents an example of EBSD maps for material subjected to KoBo at a die oscillation frequency of 2.5 Hz. The crystallographic direction ⟨111⟩ in the grains is mainly parallel to the ED (85% of the total). However, grains with a ⟨001⟩ direction parallel to the ED are also visible (15% of the total). Grains preserved their decidedly elongated shape. EBSD maps for material subjected to the KoBo procedure with faster change of deformation path are presented in Fig. 5. The proportion of grains with ⟨111⟩ and ⟨001⟩ crystallographic direction parallel to the ED was 75% to 25%, respectively. Fig. 6 illustrates the average grain size of the materials. Deformation led to great microstructure refinement. Reduction of grain size was obtained after the KoBo process, at both die rotation frequencies. However, higher refinement was received for more intense changes of the deformation path. In general, grains after the KoBo process were from 4 to 10 times smaller. Fig. 7 shows the density of the boundaries. Samples deformed by KoBo at 2.5 Hz exhibited the highest amount of HAGBs, but also high dislocation of LAGBs. The subsequent