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Mechanical properties and microstructure of ultrafine grained commercial purity aluminium prepared by cryo-hydrostatic extrusion

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ABSTRACT

CP 99.5% aluminium was processed by cryo-hydrostatic extrusion with true strains up to 3.4 in one pass. The aim was to refine its microstructure and improve its mechanical properties. The properties of the thus processed material were compared with those obtained after the same process but run at room temperature. Cooling the billet with liquid nitrogen combined with water cooling of the extruded wire enabled suppressing partially the dynamic and static structural processes. The grain size was reduced to ~400 nm in the cryo-extrusion, and to ~450 nm in the room-extrusion. In the cryo-extrusion the increase of the yield strength to 168 MPa and the hardness to 56*HV0.2* with the respective reduction of the elongation to fracture to 13.6% were obtained. The cryo-cooling effectiveness and the influence of the adiabatic heat generated during the plastic processing on the structure, mechanical properties, hardness, and tensile impact toughness just after hydrostatic extrusion, an also after the post deformation annealing are discussed. In view of the intensive adiabatic heating amounting to $0.57T_m$ no special improvement of the mechanical properties after the post-deformation annealing was observed. The cryo-cooling became effective at the true strain $\varepsilon > 2$, where the extrusion pressures clearly differed from the room-extrusion pressures and the defect density substantially increased. After the cryo-hydrostatic extrusion the mechanical properties were comparable to the highest values reported in the literature for cryo-rolling but, since they were obtained in a single deformation step and with twice as large subgrains, the ductility of the extruded aluminium was higher. During the cryo-hydrostatic extrusion conducted at high strains the reduction in ductility of the aluminium is hindered and thanks to the beneficial role of the hydrostatic stress active in the material the structural and mechanical effects which occur during severe plastic deformation are enhanced.

1. Introduction

It has been commonly proved that cryo-deformation employed to severe plastic deformation (SPD) materials is a potential technology for the application to the large-scale industrial production of nanostructured materials. Various techniques are used for introducing large plastic strains into bulk aluminium and aluminium alloys, such as cryo-rolling of 99.999%, AA6061, AA7050 [1–3], cryo-groove pressing [4], cryo-multidirectional forging AA6061 [5], high pressure torsion, and cryo-tensile AA6061 [6], cryo-milling followed by CIP and extrusion AA5083 [7], cryo-rolling and stretching AA2195 [8], homogenization and deep cryogenic treatment AA3104 [9]. Because of its experimental simplicity and the accessibility of the necessary equipment the most popular technique is the cryo-rolling process. Both, pure aluminium and aluminium alloys have been processed by this method. The literature reports concerning aluminium include asymmetric cryo-rolling Al 1050 [10], ECAP, SMAT, cold rolling and cryo rolling of ultra-high purity Al (99.9999) [1], cryo-rolling of 99.6% Al and AA2024 [11,12] and CP aluminium Al 99.5 either 99.6% [13–16]. The aluminium alloys include such alloys as AA6061 [2,17,18], Al–4Zn–2Mg alloy [19], AA5083 [15,21], AA2024 [11], Al–4.4%Cu alloy [21], AA2195 [8], AA6063 [18,22,23], AA5052 [24], AA7050 [3], and AA2219 [25]. The refinement of the microstructure in light metals gives a significant improvement in their strength-to-weight ratio, which is observed especially in aluminium alloys [16,26]. Cryo-rolling applied to commercially pure aluminium refines its structure to the ultrafine-grained (UFG) scale and also increases its tensile strength and ductility [15]. This is attributed to the formation of a bimodal structure, built of a smaller fraction of large equiaxial grains surrounded by fine grains. Moreover, deformation conducted at a cryogenic temperature followed by low-temperature

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annealing may improve considerably not only the strength of the material but also its ductility. This effect was also demonstrated in pure Al and Al alloys [20,27], such as e.g. the Al-Mg alloy [28], 6063 alloy [23] and many other alloys [15].

In the past years, the hydrostatic extrusion (HE) has been successfully used for inducing large plastic strains in bulk metals and generating UFG and nano structures [29,30]. The awareness of the advantages of hydrostatic extrusion in applying it as an efficient SPD technique is constantly increasing [31–33], but no literature reports are available concerning the combination of HE with cryogenic deformation. Hydrostatic extrusion conducted at room temperature permits refining the structure more strongly than the conventional plastic deformation treatments (e.g., rolling, extrusion, drawing, forging, or certain SPD processes, such as ECAP, CEC or ARD [30]). In the conventional deformation processes the deformation per pass cannot be large since the deformation strengthening may result in material cracking.

The high hydrostatic pressure engaged in HE, which increases the metal ductility [34], plays a very beneficial role in cryo-treatments in which the solids become more brittle compared to those subjected to room temperature deformation. On the other hand, the three-axial compressive stress state induced during the hydrostatic extrusion preserves the material cohesion even at much greater reductions per pass. The high hydrostatic pressure applied during the HE deformation is expected to prevent the disadvantageous increase of the brittleness which is characteristic of metals at low temperatures. This is why the present authors propose a combination of SPD with the HE conducted at a cryogenic temperature, which they expect will give a more advantageous compromise between the strength and the ductility of the material. This could be achieved thanks to the increased mobility of dislocations and, at the same time, their increased density.

In the present research, aluminium of commercial purity is hydrostatically extruded at cryogenic and room temperatures. It is anticipated that the grain sizes of pure Al can be more effectively reduced when the deformation is carried out at high strain rates and at cryogenic temperatures. The study includes examinations of the influence of the cryogenic extrusion strain and the post-deformation annealing on the microstructure and mechanical properties of the material. The examinations were performed within a wide true strain range using the static and impact tension tests.

2. Experimental

The starting material was commercial 99.5% purity aluminium (AA1050 according to ASTM B491) in the form of a round bar 50 mm in diameter. First, the bar was hydrostatically extruded to rods with diameters between 5 and 16 mm. The material thus obtained was subjected to cryo hydrostatic extrusion to produce 3 mm diameter wires. Prior to the deformation, the billets were annealed at 623 K for 1 h in order to diminish the disadvantageous effects of the mechanical processing and to produce homogeneous coarse grains. The average grain size of the as-annealed samples was 36 μm. As shown by the static tensile test, the starting material had the ultimate tensile strength UTS =76 MPa, the yield stress YS =51 MPa, the elongation to fracture ε_f =36%, and the uniform elongation ε_u =17%. The hardness after annealing was 29*HV0.2*.

The hydrostatic extrusion was performed in a Unipress' Hydrostatic Extrusion Press with operating pressure of 2 GPa and the die angle of 2α =45°. The extruded products were cooled at the die exit using running cold water. Two extrusion series were conducted at the same parameters: one using non-cooled billets loaded at room temperature, further referred to as the 'room-water' or 'room-extruded' material and the other with cryo-cooled billets, which, before extrusion, were immersed in a liquid nitrogen bath, further referred to as 'LN_2-water' or 'cryo-extruded'. The 'room' billets were lubricated with wax and the cryo-cooled billets were non-lubricated. Occasionally, the same billets were extruded without water cooling, and accordingly named as 'room-room' and 'LN_2-room'.

After loading the billet, the working chamber was filled with a pressure transmitting medium overcooled to −30 °C and the hydrostatic extrusion was immediately started. The time interval between the loading of the billet and the initiation of the extrusion process did not exceed 10 s. The properties of the cryo-extruded material was compared to those of the reference sample extruded at room temperature. To let the liquid nitrogen cooling conditions stabilize the billet was dipped for ~45 min before the deformation was started and the loading time was possibly minimized. In order to ensure the same water cooling conditions at the die exit the extruded wires were always 3 mm in diameter. The extrusion true strain ε ranged between 0.9 and 3.4, where the true strain is given by the natural logarithm of the extrusion ratio R defined as the ratio of the cross section surface areas of the material before and after the extrusion. The billets were hydrostatically extruded in one pass with the extrusion ratio R ranging between 2.5 and 29 to obtain a round 3 mm wire. The linear extrusion speed amounted to ~250–300 mm s^{-1}, which corresponds to the strain rate range between 1.4 and $1.7\times10^2\ s^{-1}$.

In order to maintain the efficient grain refinement during the deformation it is important to reduce the thermally activated structural process which proceeds during the extrusion, i.e. to reduce the adiabatic heating generated within the working zone of the extrusion die. The adiabatic heating effect is characteristic of the SPD processes such as hydrostatic extrusion, and originates from the mechanical work done during the deformation. In the case of hydrostatic extrusion this work is proportional to the extrusion pressure which is a measure of the work of deformation per unit volume of the treated material, and it is proportional to extrusion pressure and inversely proportional to the specific heat and the density of the material and also depends on the portion of plastic work which is converted into heat during the deformation [29]. In the case of hydrostatically extruded aluminium, the estimated temperature rise ΔT due to the adiabatic heating, assuming that 95% of the work is converted into heat ranges from 60 to 220 °C, see the dashed line in Fig. 1.

Fig. 1 also shows the experimental effectiveness of cooling the billet by liquid nitrogen compared to the effectiveness achieved without cryo-cooling, i.e.: when the billet is loaded into the extrusion chamber at room temperature. The temperature was measured directly at the contact of a Pt-PtRh thermocouple with the extruded wire a few seconds after the extrusion process is completed, and thus the results are underestimated. It can be seen that, with the cryo cooling the product after deformation is colder by ~100 °C. This difference is smaller at low strains, thanks to the weak adiabatic heating effect, and

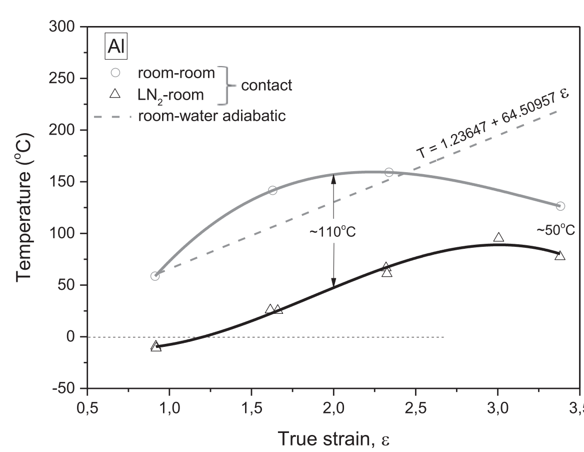

Fig. 1. Comparison of the temperatures measured at the contact with the Al wires after hydrostatic extrusion without and with the billet cryo cooling. *Note*: the dashed line represents the temperature evaluated according to adiabatic heating based on the mechanical work of deformation.