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Extruded Hot Compacted Alumix 431 Powder - Mechanical Properties and Microstructure

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Abstract:

Extrusion at 450°C of hot compacted premixed aluminium alloy powder 7075 resulted in full density and banded microstructure with the average transverse grain size of 13.6 μm after T6 heat treatment. It increased mechanical properties to yield and tensile stresses of 457 and 492 MPa, respectively and elongation 6.4 %. These meet the requirements of the ASTM standard ASM for 7075T6 conventionally manufactured alloy of similar chemical composition. The respective microstructures were compared and also to that of previously reported extruded fully dense 7075 Al which was not heat treated, and so possessed lower mechanical properties, yield and tensile stresses 254 and 402 MPa, respectively and elongation 10.4 %.

Keywords*: Alumix 431D; Hot extrusion; Microstructure; Mechanical Properties.*

1. Introduction

To attain properties of Powder Metallurgy aluminium alloys matching those of cast counterparts, processing modifications include super solidus liquid phase sintering [1-9], cold and warm compaction [10], mechanical alloying [11], extrusion of compacts [12-15] and minor compositional modifications [16-17]. The oxide layer on the powder surface limits the diffusion processes during sintering. It is facilitated when, during compaction, oxide layers are cracked and fragmented. Microstructural development during conventional processing takes place during de-lubrication, sintering and also during heat treatment [4]. Schaffer and Huo [1] investigated the influence of the composition on the sintering mechanisms and the resultant tensile properties of aluminium alloys and found that even for the alloy with the greatest ability to age harden, Alumix 431, Al-5.5Zn-2.5Mg-0.5Cu, yield and tensile strengths did not match those of cast 7075, of similar chemical composition, probably because full density was not achieved.

Cold pressing Alumix 431 powder, followed by pre-sintering and extrusion, with the ratio of 25:1, at 425°C, resulted in full density and fine, recrystallized microstructure with little evidence of banding, characteristic of extruded material that had not been pre-sintered [12]. The extrusion process significantly enhanced hardness, attributed partly to densification, and partly to the work hardening effect associated with the extrusion process. The yield and tensile strengths were 307 and 450 MPa, respectively, 8.9 % and hardness 94 HV10 [12], superior to the strengths of the material that had not undergone pre-sintering. The differences between the microstructures and hardness of the master alloy and the Al particles were proposed to account for the development of banded structures during the extrusion process.

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The present work concerns also the processing of this Al alloy by compaction and hot extrusion of a pre-mixed powder, but the powder was warm pressed at 460°C and extruded with the ratio 13.7:1 at 450°C, followed by the T6 heat treatment. For comparison, the microstructure of a forged squeeze cast and heat treated 7075 alloy, of similar chemical composition to the PM alloy, was also examined.

2. Materials and Experimental Procedures

2.1. Materials

2.1.1. Hot Compaction

Premix Al-5.5Zn-2.5Mg-0.5Cu aluminum alloy powder, Ecka Alumix 431 [18], granules were used to produce the specimens for the tests. The morphology of the granules and EDAX microanalysis results are shown in Fig. 1. The particle contents in size are 200-250 μm: 1 %, 160-200 μm: 3.4 %, 160-63 μm: 57.4 %, 63-100 μm: 31.2 %, 45-63 μm: 17.2 %, < 45 μm: 21 %. Preforms had 37.8 mm diameter, height 12.6 mm and weight of 40.17 g. The powder was poured into the die, heated to 460°C and held for 2 min, at 60 MPa pressure, resulting in the product density being 2.71 g/cm^3.

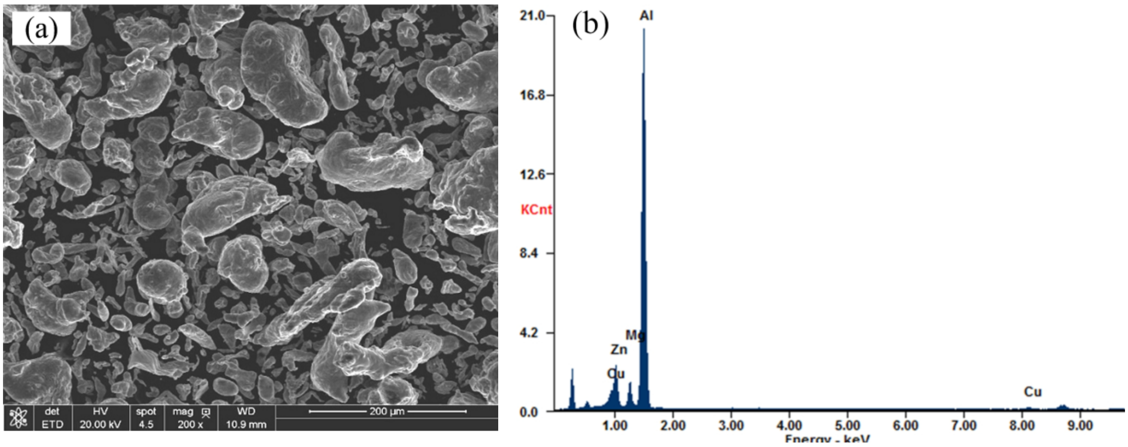

Fig. 1. Morphology Alumix granules (a) and EDAX microanalysis (b).

2.1.2. Extrusion

The preform after heating at 450°C for 5 minutes was extruded with a 13.7 ratio. The extruded rods had 10.1 mm diameter and 115 mm length. Hot extrusion increased the density of the material from 2.71 to 2.77 g/cm^3. Some materials underwent T6 heat treatment at 475°C for 1h and ageing at 125°C for 24 h. The liquid phase also appears during heating for extrusion to 450°C and heat treatment T6.

The simulated distribution of strain and stress is presented in Fig. 2. The calculated equivalent stresses were 18.9-24.3 MPa in the conical and calibrating part and strains were respectively 2.97-3.76 and 0.6-4.55 along extruded sample (die angle of 120º, 450ºC and the ratio 13.32). The strain values and their distribution in the extruded material are the results of the material flowing in the calibrating part of the die. The simulated values are higher on the side surface of the extruded product than on its axis. This has an influence on the development of microstructures during hot extrusion.

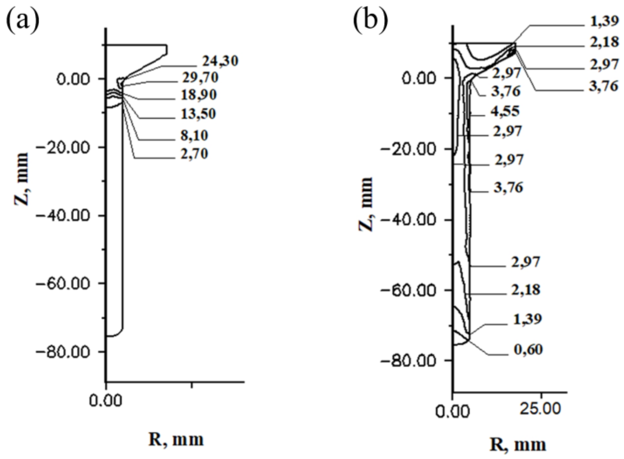

Fig. 2. Distribution of the equivalent stress (a) and strain (b) in extruded sample, extrusion ratio 13.32.

2.2. Mechanical properties

2.2.1 Hardness

Hardness HV1 was measured on cross and longitudinal sections of the sample three times on a Zwick device.

2.2.2 Tensile and Compression Testing

Gauge length of tensile specimens was 35 mm and diameter 6 mm. Compression testing was on specimens 10 mm diameter and 12 mm in height with surfaces lubricated with graphite grease. Testing was on a Zwick Roell 250 machine at 5 mm/min in tension and 7 mm/min in compression.

3. Results

3.1 Metallography

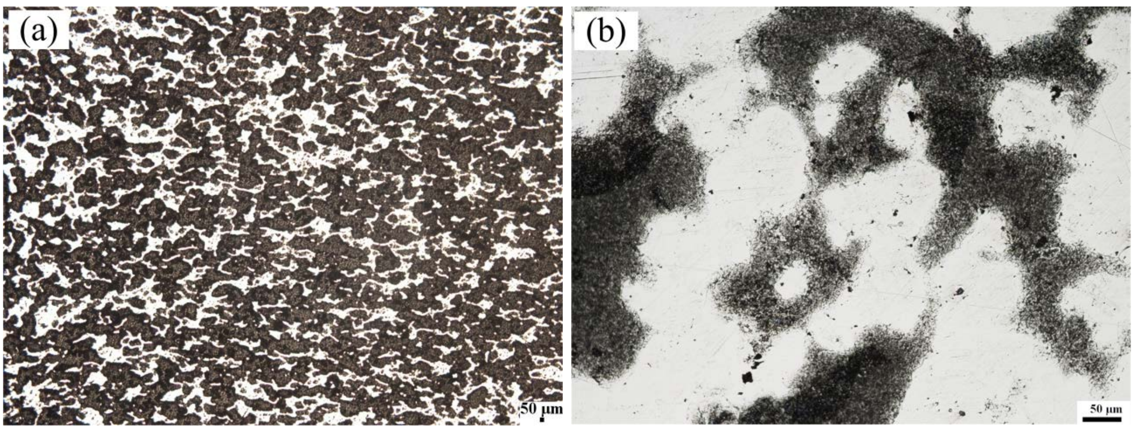

Fig. 3. Microstruture of the hot compacted specimen. Magnification: (a) 25X, (b) 200X.

Specimens for microstructural investigations were cut on their longitudinal and cross-sections. For these, Leica DM4000M light microscope and Hitachi-3500N scanning electron microscope were used. The microstructure of a hot compacted specimen is shown in Fig. 3, seen are poorly visible boundaries of the grain from a few to 100 μm in size, fine nanometricgrey particles at grain boundaries and within the grains, as well as dark pores.

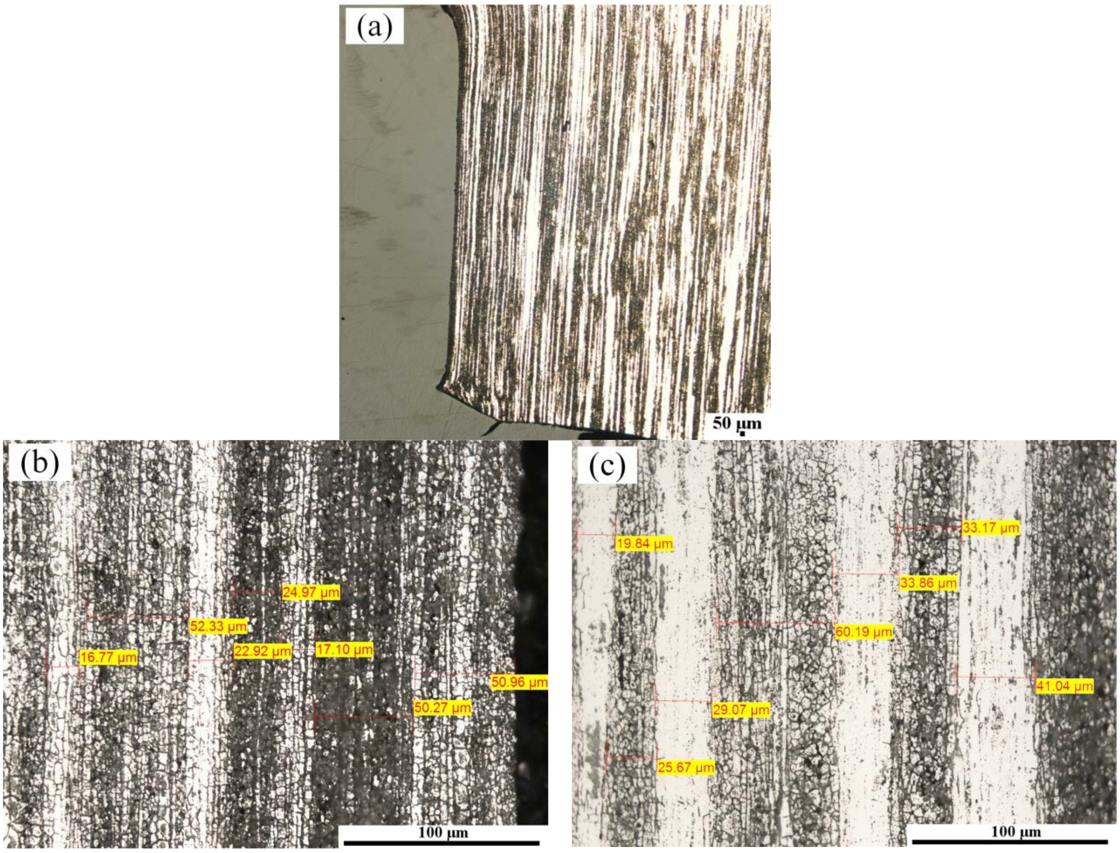

Fig. 4. Microstructure material in the initial stage of the extrusion observed on a longitudinal section at magnification 25X (a), near the outer surface (b), and (c) in the middle of the sample (magnification 500X).

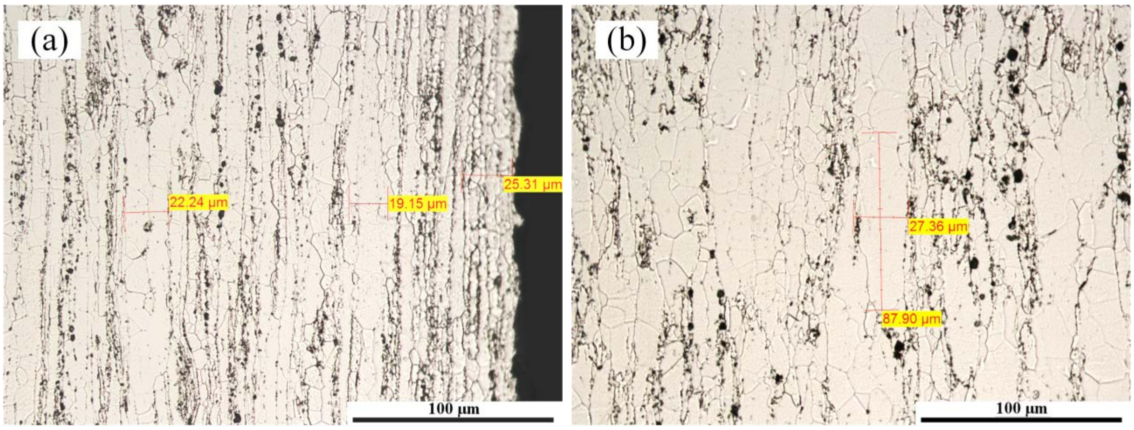

Fig. 5. Microstructure extruded and T6 heat treated material observed on longitudinal section at magnification 500X: (a) near the outer surface (b) in the center of the sample.

The macrostructure and microstructure of the extruded material on the longitudinal section are presented in Figs. 4-5, and on cross-section Fig. 6. Fig. 7-8 shows SEM microstructure and EDAX microanalyses for white and grey grains.

The longitudinal alignment of light and grey layers occurring on the longitudinal section of the extruded material (Fig. 4) is the result of the flow of material from the conical part through the sizing part of the die during extrusion. The width of the white bands was in the range of 16-50 µm near the outer surface and of the grey bands 50-52 µm, in the center the widths were 41-19 and 18-60 µm, respectively.

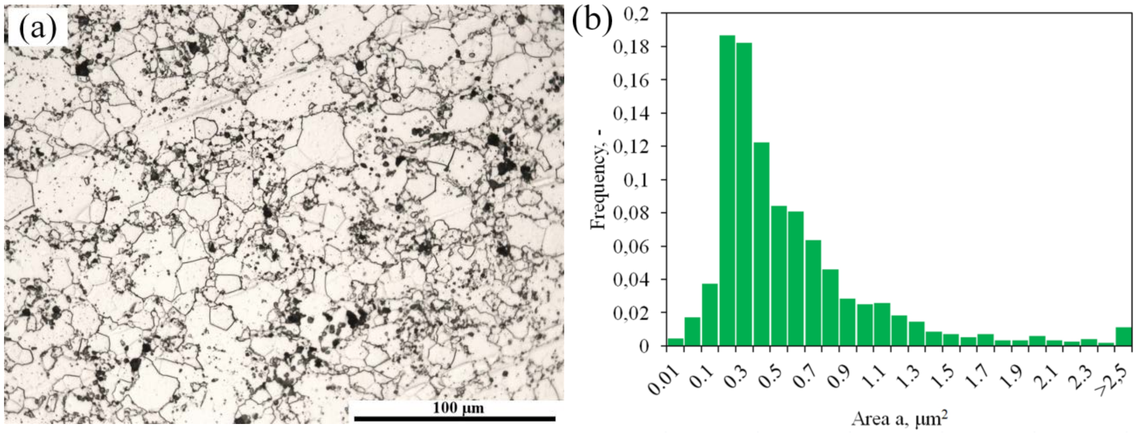

Fig. 6. Microstructure extruded and T6 heat treated material on a cross section at 500X magnification (a) and the frequency of occurrence vs. the surface of the precipitate particles (b).

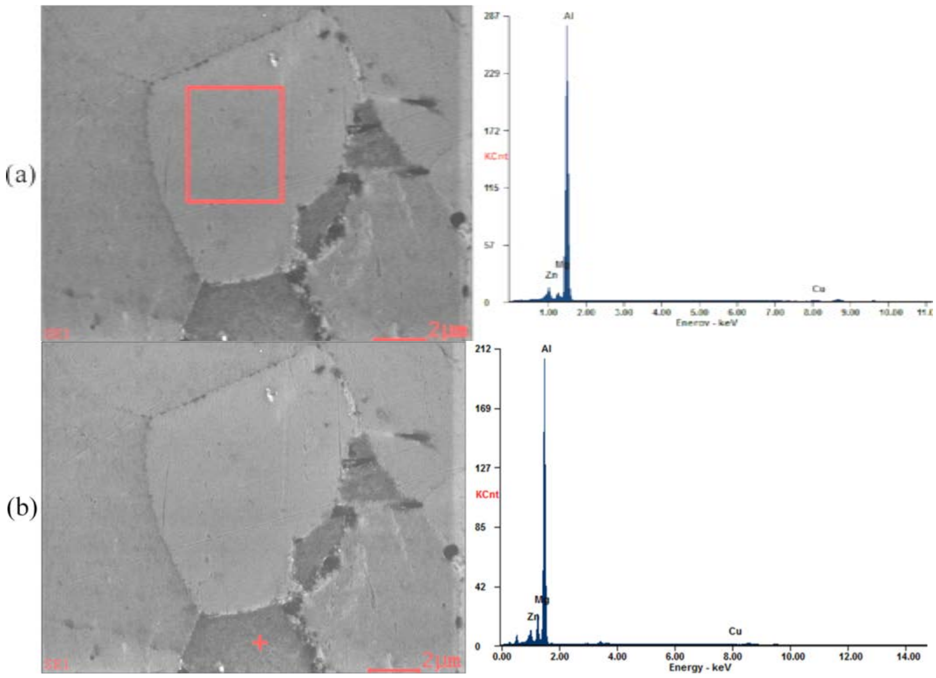

Fig. 7. EDAX microanalysis on white grain (a) and (b) grey grain, respectively 4.85% Mg, 71.98% Al, 2.04% Cu, 6.38% Zn (%At) and 10.22% Mg, 86.07% Al, 2.33% Cu, 2.69% Zn (%At).

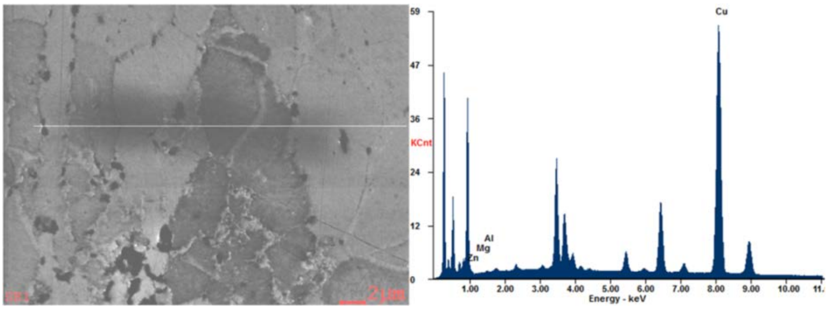

Fig. 8. EDAX Linear analysis composition on distance about 34 μm.

The average grain area on cross-section extruded and T6 treated material A=153.3 μm^2 and the average grain size was 13.6 μm. The average surface dark precipitates were 0.52 μm^2 in area, their diameters 0.81 μm and Feret diameter 0.94 μm. The distribution of black particles is presented in Fig. 6b. About 80% of particles have areas less than 1 μm^2. The dispersive particle separations are also visible on the grain surface.

In the microstructure observed in the SEM (Fig. 7), white and grey grains are clearly visible, as well as very small precipitates at grain boundaries, and individual light and dark particles within the grains. Dark particles and light layers on some of the grain boundaries are also visible. Chemical composition analysis showed that grey grains contain 14 At% more aluminum than white. The white grains are over 5 μm in size and the grey are up to about 5 μm. Dark particles visible on the white and grey grain boundaries approximate to 0.5 μm size.

The process of microstructure development begins with heating the powder for hot pressing and hot extrusion with static recrystallization during these processes with dynamic recrystallization. The final heat treatment yields the microstructure and mechanical properties of the extruded material. Heating of the powder to 460°C develops recrystallization of the components. This begins at 160°C for Al, for Cu and Mg at 200°C, and at 50°C for Zn melting is at 419.5°C. Zinc melting promotes densification and forms a eutectic fromaluminum already melted at 380°C, therefore it can increase sintering by generating liquid phases that dissolve in the matrix, where they contribute to precipitation hardening [1].
Brick [3] has determined that the white phases present in the microstructure of the GC compact are mainly $Mg_{32}(Al,Zn)_{49}$ (the T phase). $MgZn_2$ phase is the major intermetallic phase detectable in the microstructure after de-lubrication or pre-sintering, but the morphology and size in the DL and PS microstructures are different.

3.2 Mechanical Properties

Tensile stress - strain curves for the PM material are shown in Fig. 9 for extruded and heat treated specimens and compression - strain plots on Fig. 10.

Tensile properties for extruded material: E=71.23 GPa, Yield stress=254 MPa, UTS=402 MPa, YS0.2/UTS=63%, El=10,4% and RA=13.8%. For heat treated material the corresponding values are 71.5 GPa, 457 MPa, 492 MPa, YS0.2/UTS=92%, El 6.4% and RA 8.0%. For compression: yield strength 310-345 MPa, compression stress CS=730-746 MPa, strain 33.5-36.1%. Hardness was 112±4 HV for extruded and 170±4 HV for heat treated material.

Failure surface of a tensile sample is presented in Fig. 11. Precipitates and craters, where particles had fallen out, are visible.
The chemical composition in marked places was determined for the matrix (Fig. 11a): 4.60% Mg, 65.0% Al, 2.76% Cu, 9.30% Zn (%At) and for the precipitate 3.19% Mg , 24.74% Al, 14.52% Cu, 44.69% Zn (%At) (Fig. 11b).

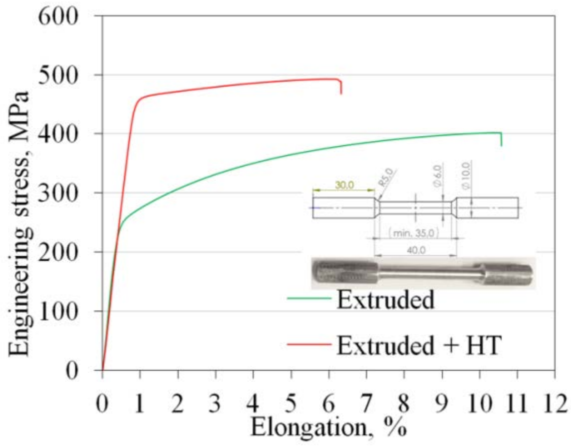

Fig. 9. Engineering stress-elongation plots for extruded ad and T6 heat treated materials.

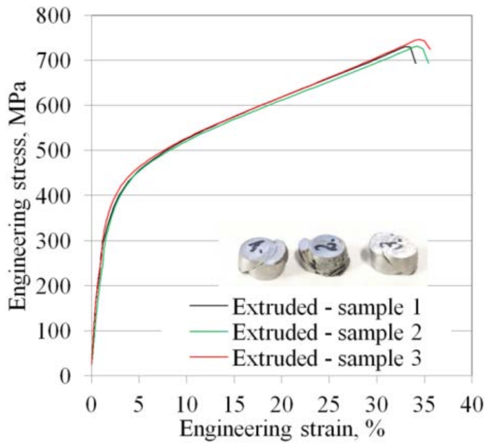

Fig. 10. Engineering stress - engineering strain compressive plot for extruded material.

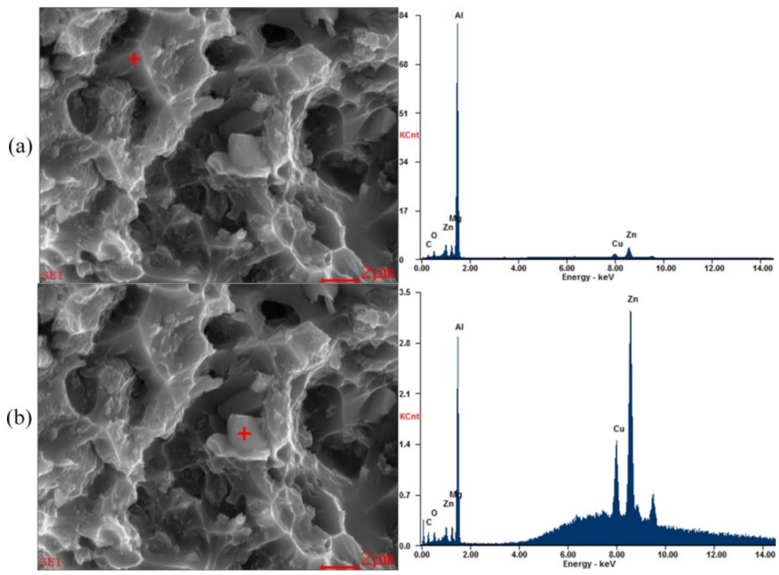

Fig. 11. Fracture surface with marked (a) the matrix and (b) individual precipitate for chemical analysis.

3.3 Microstructure of a forged squeeze cast 7075 alloy

For comparison, the microstructure of a forged squeeze cast and T6 heat treated 7075 alloy of similar chemical composition to the PM alloy, was also examined. Cuboidal sample from the alloy, after homogenization at 465°C for 20 h, was upset forged at this temperature with a deformation 50% and cooled. T6 heat treatment was 460°C for 1 h and ageing at 150^{o}C for 4 h. Fig. 12 presents the microstructure of transverse and longitudinal sections. The grain sizes were in range 37-226 µm long and 20-37 µm wide in the transverse direction, and in the longitudinal direction, 91-265 µm and 20-70 µm, respectively. The average grain size was 74.4 µm on transverse and 60.7 µm on longitudinal sections. Hardness was 150 HV, which approximates the yield and tensile strengths to 428 MPa and 500 MPa, respectively.

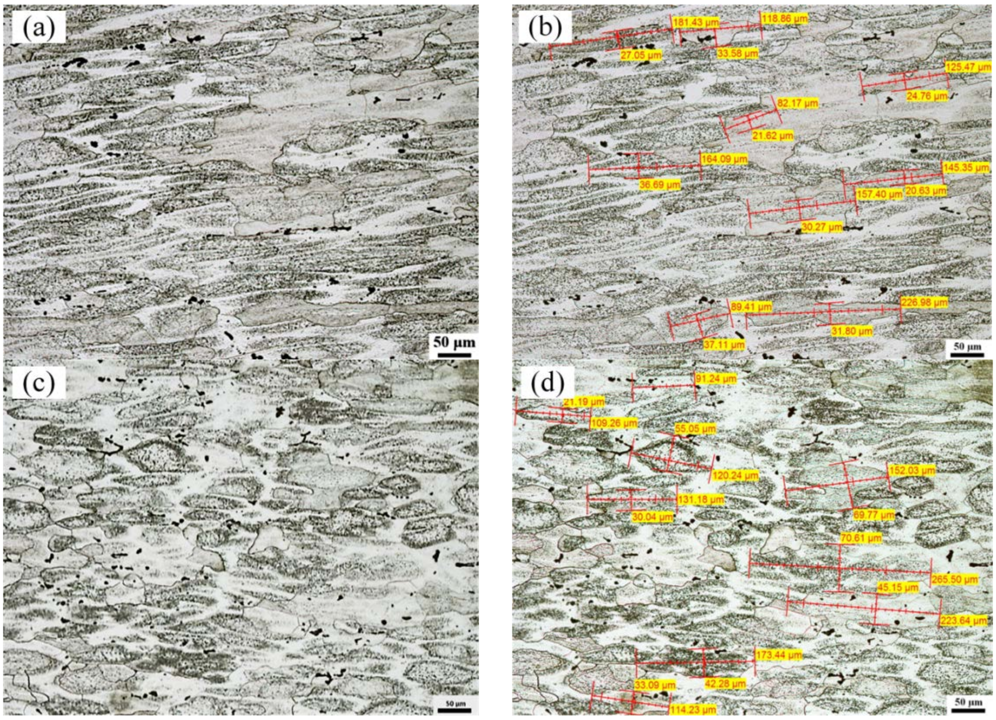

Fig. 12. Microstructure of T6 heat treated squezze cast 7075 alloy: (a), (b) transverse and (c), (d) longitudinal sections (magnification 200x).

4. Discussion

Aluminium materials with additions of transition metals (TM) with fast solidification during atomization show a homogeneous microstructure with very fine and uniformly distributed TM-rich particles in a crystalline aluminium matrix [13]. This microstructure contrasts strongly with that of conventionally cast material, which has a relatively heterogeneous microstructure with the TM-rich phases being much larger. When a Al-TM 7000 series alloy powder manufactured by inert gas atomization by induction melting was placed in a can and directly hot extruded with extrusion ratios of 6.7 and 25, rods with a diameter of 82.6 and 42.7 mm, respectively were obtained. The achieved T6 properties were UTS 460-510 MPa, YS 375-405 MPa, EL 4.5-9%, HRB 75-80, depending on the extrusion ratio, comparable with those of wrought material.

Jabbgari et al [12] clearly demonstrated the importance of pre-extrusion material state in the production of resulting mechanical properties. Theirs were substantially improved by the 525°C presinter following cold compaction, material PS. The PS powder compact presents the highest degree of alloy development prior to extrusion, which results in mechanical properties of the PS extrudate. Higher degrees of alloy development mean higher incorporation of alloying elements into the Al solid solution matrix, which can enhance the mechanical properties of the extruded alloy through solid solution strengthening. The PS extrudate shows an ultrafine, recrystallized grain structure and a homogeneous distribution of fine second phase particles in the microstructure, which effects of the combined grain-size and dispersion strengthening. For these materials the mechanical properties attained were 94 hardness HV10, yield and tensile strengths of 307 and 450 MPa and elongation of 8.9%, higher than for extruded DL delubricated compact 82, 229 MPa, 338 MPa, 9.2 % respectively, and compressive strength 471 MPa. We used hot compaction at 460°C, obtaining, following extrusion at 450°C, yield and tensile strength values of 253 and 402 MPa and 13.8% elongation, the average compressive strength was 738 MPa and strain 35%. After the T6 heat treatment, however, attained were these mechanical properties: yield stress 457 MPa, tensile stress 492 MPa, 6.4 % elongation which matchthose specified for extruded cast alloy AA7075 by ASM [19]. It is therefore instructive to compare the microstructures after extrusion of PS and our material, then consider the T6 heat treatment. The PS extrudate had a homogeneous grain structure composed of fine equiaxed grains, from submicron to a few microns in size. The microstructure of our material, with a lower extrusion ratio and a slightly lower strength, contained light and grey bands, of greater width than DL (delubricated) material of Jabbgari et al [12]. Their grain sizes were slightly lower than ours with an average of 13.6 µm after the T6 heat treatment. The nano size precipitates around the grains (Fig. 6) affect dispersion strengthening. In our cast sample, the size of the majority of the TM-rich phases is in the range of 110-200 microns, whereas in the PM processed alloy the range is 30-250 nanometres. Finally, the squeeze cast alloy had considerably larger grains compared to the sintered, average grain size was 74.4 µm on transverse and 60.7 µm on longitudinal sections. Our results therefore are in agreement with the generally held view that grain size has little or no effect on the yield strength of aluminium alloys.

5. Conclusion

- It was confirmed that it is possible to get full density in PM Alumix 431 by hot extrusion.
- The macrostructure of the extruded material in the longitudinal section consists layers made of a tough and hardened white phase.
- The mechanical properties of hot pressed, hot extruded,T6 heat treated material are: yield and tensile stresses 457 and 492 MPa, respectively, and 6.4 % elongation, higher than of the also fully dense extruded de-lubricated preforms of Alumix 431, which had not undergone the ageing treatment. They are comparable to those of the direct extruded powder Al-TM 7000 series alloy, and of cast material, as specified in ASTM standard for 7075T6.

Acknowledgments

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Сажетак: *Екструзија на 450°C вруће пресованог праха алуминијумске легуре 7075 резултирала је пуном густином и тракастом микроструктуром са просечном попречном величином зрна од 13,6 μm након термичке обраде Т6. Поправила је механичка својства до напонатечења и затезања од 457 и 492 MPa, респективно, и издужења од 6,4 %. Ови материјали испуњавају захтеве АСТМ стандарда, АСМ за 7075Т6 конвенционално произведену легуру сличног хемијског састава. Одговарајуће микроструктуре су упоређене као и са претходно описаним екструдираним потпуно густим 7075 Al који није био термички обрађен, и тако поседовао ниже механичке особине, напона течења и затезања 254 и 402 MPa, респективно и издужење од 10,4 %.*

Кључне речи*: Алумик 431Д, врућа екструзија, микроструктура, механичка својства.*