Microstructure and Mechanical Properties of Spark Plasma Sintered and Severely Deformed AA7075 Alloy

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Abstract: In this paper, the microstructure and mechanical properties of AA7075 with a coarse-fine-grained laminated microstructure produced by spark plasma sintering (SPS) and the cyclic extrusion severe deformation (KOBO) technique were investigated. It was found that an inhomogeneous grain microstructure was formed from coarse and fine grains by the SPS process and then was transformed into a coarse-fine-grained laminated microstructure by means of KOBO extrusion at room temperature. The grain refinement during KOBO extrusion resulted in a fine grained laminated microstructure created due to the formation of low-angle grain boundaries (LAGBs), followed by dynamic recrystallization, leading to high-angle grain boundaries (HAGBs). The EBSD analysis results reveal the formation of a deformed and partially recrystallized ultrafine grain microstructure owing to the generation and development of shear bands during KOBO extrusion. The ultimate tensile strength (UTS) of the AA7075 alloy rose after SPS-KOBO severe deformation up to 422 MPa, with high strains of about 33%. The obtained results clearly show that the SPS-KOBO extrusion technique allows a bimodal laminated finite gradient grain microstructure to be obtained due to deformation and dynamic recrystallization, which result in both high strength and good ductility. The new heterogeneous AA7075 alloys obtained by the SPS-KOBO combined techniques demonstrate that microstructural heterogeneities can assist in overcoming the strength–ductility trade-off.

Keywords: spark plasma sintering; KOBO extrusion; AA7075; microstructure; mechanical properties

1. Introduction

The spark plasma sintering (SPS) of Al alloys followed by post-sintering by means of KOBO extrusion is a pathway for producing ultrafine-grained materials by imposing severe plastic deformation [1]. SPS is known to be one of the most promising powder metallurgy (PM) technologies to produce fully dense sintered metals, alloys, and composites. The SPS densification and microstructure formation process combines the thermomechanical impact generated by the simultaneous electric field with an applied mechanical field [2]. During SPS, powder particles are compacted in graphite die, heated by a high-intensity and low voltage pulsed DC electric current, and deformed due to shear stresses generated by the mechanical field. This results in some advantages such as high heating and cooling rates, short sintering times, and the formation of specific microstructures with enhanced mechanical properties. However, spark plasma sintered metals demonstrate low tensile elongation, similar to typical powdered materials. A main reason for the low ductility of
PM Al based materials is related to the presence of a thin surface film of hydrated alumina on the particles. SPS is known to be a highly effective process for physical disruption of the Al₂O₃·3H₂O films which occur on the surface of the aluminium particles [3,4]. The authors of [4] demonstrated that SPS at optimized regimes led to disruption of the film on the surface of the Al particles, which allowed the wrought-like ductility of the sintered compacts to be achieved (tensile elongation of 40–50%). Powdered Al alloys, such as AA7075, demonstrate high stability of oxide films on the particle surface owing to the alloying elements, and it is difficult to effectively disrupt these films by SPS. Moreover, SPS of the AA7075-type alloyed powders led to the formation of a cellular microstructure with concentrated intercellular areas of the alloying elements [3,5], and neither SPS processing nor heat treatment could effectively disrupt this chemical segregation [3]. Thus, the tensile ductility of SPSed Al alloy compacts does not usually exceed 3–5%. The SPS route allows a high strength of AA7075 to be obtained, similar to that of cast AA7075 [6]. However, the total elongation of the cast and heat treated AA7075 alloy does not exceed 10%. That is why additional deformation processing of SPSed compacts is required to increase the ductility of Al alloys. From this viewpoint, the research of severe deformation methods to increase the ductility without compromising the material strength is of great interest.

Severe plastic deformation (SPD) is well known as one of the most effective methods for the microstructure formation of metallic materials [7]. Considerable strain is made using workpiece SPD technologies such as high-pressure torsion (HPT), equal channel angular pressing (ECAP) [8], friction stir processing (FSP) [9], twist extrusion (TE) [10], and others. Classical extrusion and hydro extrusion methods are also applied, and they have demonstrated effectiveness in the consolidation and grain refinement of Al alloy powder composites [11]. The authors of [11] showed that AA5xxx alloys can be efficiently strengthened up to a flow stress exceeding 450 MPa by both the hydro extrusion of solid preforms and extrusion compaction of nanopowders, and both obtained composites exhibited tensile elongation values greater than 10%. It is important to note the excellent mechanical properties of severely deformed alloys, which are based on grain size reduction and strain hardening. The severe deformation regime generates a high density of dislocations, solid solution strengthening due to the presence of alloying elements (such as Zn, Mg, Cu) in the Al matrix, dynamic softening, and dynamic recrystallization processes. The complex twisting-extrusion method (KOBO) seems optimal for Al alloys [12,13] because of combining monotonic extrusion, responsible for the Al profile shape formation, with excessive cyclically changing deformation (reverse twisting) [12]. Generally, the main advantage of the KOBO method is due to the specific die oscillation that results in an effective reduction in the processing energy and increases the processing efficiency [14]. The lack of studies deeply analysing the microstructures and properties reached in SPSed 7xxx series alloys after severe deformation by the KOBO extrusion method hinders the efforts to find ways to effectively strengthen the alloy without compromising its tensile ductility.

The purpose of this paper is to examine the microstructure formation features underlying the SPS and KOBO extrusion processes of AA7075. The present investigation studied the effect of sintering and KOBO severe deformation on the microstructure, tensile properties, and nanohardness of AA7075 phase constituents. The specific objective is to examine the grain microstructure and precipitation formation controlled by KOBO severe deformation. The effects of annealing and age hardening of SPSed and KOBO-extruded AA7075 on the evolution of the microstructure and mechanical properties will be studied in future work.

2. Materials and Methods

Alumix 431 (ECKA Granules Germany GmbH, Velden, Germany) powder, without lubricant, exhibiting the chemical composition shown in Table 1, was spark plasma sintered using an HP D 25/3 furnace (FCT Systeme GmbH, Rauenstein, Germany), similar to the procedure described by the authors in [15]. The SPS parameters were chosen on the basis of the experimental results described in [5]. The powder was heated up to 500 °C at
100 °C/min in vacuum and held at the sintering temperature for 5 min. The time of pulse on was 125 ms and of pulse off was 5 ms. The powder was compressed with a constant compaction pressure of 50 MPa (compaction force 63 kN) throughout the whole sintering process. SPSed cylindrical samples 40 mm in diameter and 10 mm in height were extruded by the KOBOT technique at room temperature using a 25 MN press with an extrusion ratio of \( \lambda = 10 \), a die oscillation angle of ±8°, and an oscillation frequency of \( f = 6 \) Hz. The extrusion force was 400 kN and the extrusion rate selected was fixed at 0.5 mm/s. As a result, 5 rods with a diameter of 10 mm were manufactured. The specimens for the hardness measurements, microstructure analysis, and compression and tension tests were cut by wire electrical discharge machining (WEDM).

Table 1. Chemical composition of Alumix 431 powder delivered by ECKA Granules Germany GmbH (wt%).

|     | Al   | Cu   | Mg   | Zn   | Sn   |
|-----|------|------|------|------|------|
|     | Balance | 1.80 | 2.80 | 6.40 | 0.29 |

The microstructures of the specimens were observed on polished and unetched or etched (etching solution: 45 mL H₂O, 7.5 mL HCl and 5 mL HF) transverse and longitudinal cross-sectioned surfaces using an Eclipse L150 light microscope (LM) (Nikon, Kawasaki, Japan) and a MIRA 3 scanning electron microscope (SEM) (TESCAN, Brno, Czech Republic).

The high resolution imaging of the microstructure was performed using a TITAN 80-300 scanning transmission electron microscope (STEM) (FEI, Hillsboro, OR, USA) operating at 300 kV with a Z-sensitive high-angle annular dark-field (HAADF) detector. The chemical composition of the oxidation products was studied using energy-dispersive X-ray spectroscopy (EDX) under the STEM mode. Phase identification was performed under the TEM mode using convergent beam electron diffraction (CBED). The specimens for the STEM investigations were prepared using focused ion beam (FIB) on an Quanta 3D 200i dual-beam system (FEI, Hillsboro, OR, USA). During the FIB preparation of the specimens, Pt layers (15 µm × 2 µm × 2 µm) were applied on top of the specimens to protect them during the gallium ion thinning process.

The EBSD measurements were carried out using a Helios Nanolab 600i field emission gun microscope (FEGSEM) (FEI, Hillsboro, OR, USA) coupled with an HKL NordlysNano electron backscatter diffraction detector (Oxford Instruments, High Wycombe, UK) and equipped with the Channel 5.0 acquisition and analysis software package. The EBSD measurements were conducted at the accelerating voltage of 15 kV, with a beam current of 5.5 nA, and with a step size of 0.4 µm. The Vickers hardness measurements were carried out using an FM-800 hardness tester (Future-Tech, Kawasaki, Japan) and applying a load of 4.903 N for 15 s. The Vickers indentation nanohardness measurements were performed using a Picodentor HM500 nanoindenter (Fisher, Windsor, SC, USA). The applied load was 0.01 N and the holding time was 5 s. The compressive strength was measured using a 4483 Instron mechanical testing machine with a load cell of 150 kN (constant crosshead speed 2 mm/min) and an initial strain rate of 0.0033 s⁻¹. For the compressive test, cylindrical specimens with a 10 mm diameter and a 10 mm height were used. The tensile strength was measured using an MT5000HC tensile stage (Deben, Suffolk, UK) with a measuring range up to 5 kN (constant traverse speed 0.2 mm/min) and an initial strain rate of 0.0022 s⁻¹. The tensile specimens were made according to the ASTM E8 standard [16] and five specimens cut from each rod were tested for each investigated property. The yield strength, ultimate tensile strength and the tensile elongation of the materials were obtained from the engineering stress-strain curve. The yield strength was determined with the 0.2% offset strain method.
3. Results
3.1. Microstructure

To define some specific features of the microstructure formation during both the SPS and KOBO processes, extensive LM and SEM of unetched and etched specimens were carried out, and the grain core as well as the boundaries of the SPSed and KOBO processed specimens were observed. Representative LM and SEM micrographs of the SPSed specimens are presented in Figure 1. The SEM micrograph of the unetched microstructure (Figure 1b) clearly shows Al grains of different sizes (dark contrast phase) with grain boundaries containing intermetallic phases (light contrast phase) similar to [3,5]. The microstructure is heterogeneous because of the simultaneous presence of very large and fine grains. The LM micrograph of the etched SPSed microstructure (Figure 1a) reveals the presence of some pores and precipitates inside the grains. The observable porosity is about 2–3%. The SEM micrographs of the etched SPSed specimens at high magnification (Figure 1c) show an active etch dissolution of the base metal in the areas of the grain boundaries and demonstrate a slow dissolution process of the grain core in the acidic HCl and HF solution. The local etch dissolution conditions of the grain depend on the content of the alloying elements (Cu, Fe, and others), similar to the results shown in [17].

Figure 1. Microstructure of SPSed AA7075: transverse cross-sectional LM micrograph of etched microstructure (a); transverse cross-sectional SEM micrographs of unetched microstructure (b,c). Red circle shows nanoindentation imprint.

The presence of coarse grains in SPSed Al alloys was already reported in [18]. The authors of [18] attributed the abnormal grain growth to the high local current density due to small contact areas between the powder particles at the beginning of the SPS process. This results in an abnormal jump in the temperature up to the melting point [19].
The formation of second phases during SPS is shown in the HAADF and TEM micrographs (Figures 2 and 3). EDX analysis was performed during TEM examination to estimate the composition difference of the second phases. The results reveal coarse precipitates at the grain boundaries (Figure 2a,b) with various chemical compositions. Most of the particles formed at the grain boundaries during SPS are of flat and acicular shapes, whilst the grain cores are free of precipitates (Figure 2a). The EDX analysis of the chemical composition of the precipitates demonstrates the presence of Mg, Zn, and Cu, and the Zn/Mg ratio is in the range of 2–3 (Table 2). Analysis of the X-rays maps for Al, Mg, Zn, and Cu (Figure 3a–d), and the CBED patterns (inset in Figure 3e,f), leads to a similar conclusion and allows us to state that intermetallic particles such as MgZn$_2$, MgCuAl$_2$, and CuZn$_2$ are formed. This conclusion is in agreement with the data presented in [17,18].

![Figure 2](image1.png)

**Figure 2.** Transverse cross-sectional HAADF STEM micrographs of SPSed AA7075 (a,b).

![Figure 3](image2.png)

**Figure 3.** EDX elemental mapping of SPSed AA7075 in area indicated by red square in Figure 2a: aluminium (a); magnesium (b); zinc (c); copper (d); transverse cross-sectional BF-TEM micrographs (e,f). Insets of Figure 3e,f present CBED pattern of second phases.
Table 2. Chemical composition of precipitates in various areas presented in Figure 2b.

| Element | Atomic % |
|---------|----------|
|         | # 1      | # 2      | # 3      | # 4      |
| Mg      | 02.47    | 02.68    | 02.68    | 03.38    |
| Al      | 88.22    | 78.86    | 78.86    | 86.85    |
| Fe      | 00.61    | 00.45    | 00.45    | 00.00    |
| Co      | 00.00    | 00.18    | 00.18    | 00.24    |
| Cu      | 01.09    | 07.70    | 07.70    | 00.96    |
| Zn      | 07.57    | 10.11    | 10.11    | 08.54    |

The microstructure of the SPS-KOBO extruded specimen is shown in Figure 4. The SEM micrograph of the microstructure without etching (Figure 4b) at low magnification shows the relatively uniform distribution of intermetallic phases achieved due to KOBO severe deformation. The dense grain (without pores) was selected to perform a nanoindentation mark and is shown in SEM micrographs at higher magnification (Figure 4c). The etched cavities are located in the longitudinal direction with respect to the KOBO extrusion, revealing the location of the heavily elongated grains due to KOBO severe deformation. The TEM micrograph (Figure 4d) shows the dislocations in some grains, precipitated particles due to the development of aging processes, and the particles at the grain boundaries owing to the coalescence process during KOBO severe deformation.

Figure 4. Microstructure of SPS-KOBO extruded AA7075: transverse cross-sectional LM micrograph of etched microstructure (a); longitudinal cross-sectional SEM micrographs of unetched microstructure (b,c); transverse cross-sectional BF-TEM micrograph of unetched microstructure (d).

The EBSD maps of boundaries superimposed to image quality (IQ) images and pole figures of the SPSed compacts and SPS-KOBO extruded rods in the transverse cross-sections are presented in Figures 5 and 6, respectively. The black lines in the maps indicate the high-angle grain boundaries (HAGBs with an angle higher than 15°) and the blue lines indicate the low-angle grain boundaries (LAGBs with an angle lower than 15°). The SPSed specimens exhibit LAGBs preferably in the fine grains, whilst the coarse grains are mostly
free of LAGBs (Figure 5a). KOBO extrusion caused a decrease in the average grain size from 9.32 (Figure 5c) to 2.57 µm (Figure 5d) and an increase in the overall content of LAGBs. A higher density of HAGBs is seen at the areas of fine-grain deformation due to probable strain localization. The transformation of LAGBs to HAGBs probably occurs in these places. Thus, the bimodal grain microstructure is formed due to dynamic recrystallization induced by KOBO severe deformation.

Figure 5. Maps of boundaries superimposed to image quality (IQ) images (a,b); grain size maps (c,d) of SPSed (a,c) and SPS-KOBO extruded (b,d) AA7075.

Figure 6a,b show the (100), (110), and (111) pole figures for the AA7075 after SPS and KOBO extrusion. The statistical description of the intensity of the pole image is known as the multiple of uniform density (MUD) and is quantified using the maximum intensity of the contoured pole figures [20]. The data show that the variation in the pole density of the SPSed specimens is in the range from 0.07 to 5.80 MUD. The map in Figure 6a shows that the grains are not oriented in the direction of the applied compaction pressure for SPSed aluminium crystals, being the MUD numbers in the range of 1. In this case, the variation in the pole density is between 0.11 and 3.40 MUD (Figure 6b), which indicates a more deformed and partially recrystallized microstructure.

KOBO severe deformation induces precipitation and intermetallic precipitate coalescence processes, which result in a considerable change in the sintered AA7075 microstructure. The formation of second phases during KOBO extrusion is shown in the HAADF and TEM micrographs (Figures 7 and 8). The coarse precipitates formed at the grain boundaries during SPS become globular (Figure 7a) owing to dynamic spheroidization, whilst the disperse particles seen in the core of the grains are the product of the aging process. The EDX analysis of the chemical composition (Figure 7b, Table 3) and EDX elemental mapping of the KOBO extruded AA7075 coarse precipitates (Figure 8a–d) demonstrate the presence of Mg, Zn, and Cu, similar to the SPSed material, which indicates their thermodynamic stability during KOBO deformation. Whilst the dynamic spheroidization mechanism is well described for the hot deformation of Ti alloys [21], examination of the dynamic mechanisms of the microstructure formation of Al alloys began only recently [22], and further
investigation of the dynamic spheroidization of intermetallic particles will be conducted in future work.

![Pole figures of SPSed (a) and SPS-KOBO extruded (b) AA7075.](image)

**Figure 6.** Pole figures of SPSed (a) and SPS-KOBO extruded (b) AA7075.

![HAADF STEM micrographs of SPS-KOBO extruded AA7075 (a,b).](image)

**Figure 7.** HAADF STEM micrographs of SPS-KOBO extruded AA7075 (a,b).

The classical AA7075 aging mechanism schematics can be presented as a supersaturated solid solution $\rightarrow$ Ginier-Preston zones $\rightarrow$ $\eta' \rightarrow \eta$, where $\eta$ is the equilibrium phase MgZn$_2$ and $\eta'$ is its metastable phase [18]. Indeed, the CBED pattern of the precipitates shown in the inset of Figure 8e demonstrates the presence of the MgZn$_2$ intermetallic compound. The dynamic strain aging process is enhanced by severe deformation and results in effective hardening of these alloys [18,23]. The kinetics of strain aging is believed to be accelerated due to the high density of the grain boundaries and dislocations resulting from KOBO severe deformation (see Figure 4d).
Figure 7. HAADF STEM micrographs of SPS-KOBO extruded AA7075. (a,b) 

Table 3. Chemical composition of precipitates in various areas presented in Figure 7b.

| Element | Atomic % |
|---------|----------|
|         | # 1      | # 2      | # 3      | # 4      |
| Mg      | 12.25    | 15.09    | 24.03    | 09.23    |
| Al      | 67.83    | 66.81    | 58.35    | 84.07    |
| Si      | 00.00    | 00.61    | 01.54    | 00.30    |
| Cu      | 04.84    | 03.45    | 03.27    | 00.00    |
| Zn      | 14.57    | 10.63    | 08.16    | 04.86    |
| Sn      | 00.49    | 03.38    | 04.62    | 01.50    |

3.2. Mechanical Properties

The density values and mechanical properties of the SPSed and KOBO extruded AA7075 specimens are shown in Table 4. Near-full density was obtained via SPS-KOBO extrusion technology. It should be noted that the microhardness of the SPSed compacts of 144 HV$_{0.5}$ was diminished to 98 HV$_{0.5}$ due to KOBO extrusion. A similar behaviour in the compressive strength (a decrease from 618 to 464 MPa) is observed. These results demonstrate that softening processes such as dynamic recrystallization and dynamic recovery are the main microstructure formation mechanisms during KOBO extrusion due to the high content of point defects generated by severe deformation and significant enhancement of diffusion [12–14]. Nonetheless, reduction in the grain size and dynamic strain aging are hardening processes which seem to increase the hardness. Similar data were obtained from the tension and compression tests. The mechanical behaviour of the SPSed and SPS-KOBO extruded materials at linear tension and compression are characterized by the stress–strain curves shown in Figure 9. The yield strength of the SPSed specimens...

Figure 8. EDX elemental mapping of SPS-KOBO extruded AA7075: aluminium (a); magnesium (b); zinc (c); copper (d); BF-TEM micrograph (e). Inset of Figure 8e presents CBED pattern of second phases.
is 345 MPa, whereas the SPSed-KOBO extruded specimens exhibit a yield strength of 277 MPa.

### Table 4. Bulk density, mechanical properties, nanohardness, and indentation size effect parameters of coarse and fine grains of SPSed and SPS-KOBO extruded AA7075.

| Parameter, Unit | SPS Longitudinal Cross-Section | KOBO Transverse Cross-Section |
|-----------------|---------------------------------|-------------------------------|
| Density, g/cm³  | Coarse Grain 2.78 ± 0.01  | Fine Grain 2.80 ± 0.01 |
| Microhardness, MPa | 1440 ± 40  | 980 ± 20 |
| Yield tensile strength, MPa | 345 ± 15  | 277 ± 18 |
| Ultimate tensile strength, MPa | 345 ± 15  | 422 ± 14 |
| Total elongation, % | 2.0 ± 0.3  | 33.2 ± 2.5 |
| Compressive strength, MPa | 618 ± 4  | 464 ± 32 |
| Nanohardness, MPa | Coarse Grain 1433  | Fine Grain 439 |
| Intrinsic hardness, $H_i$, GPa | 2.570  | 0.434 |
| Intrinsic length, $l_i$, µm | 0.0696  | 0.6792 |
| Linear simulation veracity, $R^2$ | 0.9905  | 0.9504 |
| Nanoindentation stochastically stored dislocation density | 7.34 × 10^{15}  | 2.46 × 10^{14} |

Figure 9. Tension and compression stress–strain curves of SPSed and SPS-KOBO extruded AA7075. Insets: tensile specimen drawing (dimensions in mm), photograph of specimen after compression test.

The anomalous high values of the yield stress of the SPSed specimens are believed to result from deformed grain boundaries and the presence of intermetallic compounds at the grain boundaries formed during SPS. The SPSed AA7075 composite exhibits a brittle mode of fracture resulting in low tensile ductility (Figure 9, Table 4). The SPSed tensile specimens exhibit fracture strain $\varepsilon_{fr} = 2$–$3\%$. However, the application of KOBO extrusion results in a considerable increase in the fracture strain up to 33% (Figure 9).

On the other hand, the UTS of the AA7075 increased after SPS-KOBO severe deformation up to 400 MPa, exhibiting high strain (33%). This effect was achieved due to the formation of a coarse–fine laminated grained microstructure [24,25]. For the KOBO extruded specimens, the average size of the coarse grains is about 3.5 µm whilst the fine grain size is about 1.7 µm (Figure 5).
The comparison of the compression and tension deformation behaviour in the SPSed specimens reveals that, during compression, the specimen achieves relatively higher total strains due to the compressive stresses, facilitating the accumulation of dislocations without nucleation or growth of microcracks. Nevertheless, it is difficult to connect the real macroscopic deformation mechanisms of the sintered AA7075 with a complex microstructure because of its inhomogeneity. From this viewpoint, a study of the micro-mechanical behaviour of the AA7075 microstructure constituents is important.

The micro-mechanical behaviour is characterized by nanohardness testing of the coarse and fine grain areas of AA7075 (Figure 10). The coarse and fine grain types are defined based on the EBSD analysis results presented in Figure 5. The coarse grain sizes (shown in Figure 5c,d) are in the range of 15–40 µm for the SPSed specimens and 8–10 µm for the SPS-KOBO extruded specimens. The fine grains were found to be 3–5 µm for the SPSed and 2–4 µm for the SPS-KOBO extruded specimens. To increase the test accuracy, 10 measurements per test were made. The indents in the grain core were chosen to avoid etching effects such as cavities and grain boundaries (for example see the indents in Figures 1c and 4c).

![Figure 10. Nanoindentation curves obtained in coarse and fine grains of SPSed and SPS-KOBO extruded AA7075.](image)

The results shown in Table 4 and Figure 10 indicate that the nanohardness of the coarse grains is higher than that of the fine grains for both the SPSed and SPS-KOBO extruded specimens. Considering the fact that the fine grains are formed due to the dynamic recrystallization processes during SPS [3,20], they exhibit a lower hardness as a result of a lower dislocation density as compared to that of their coarse counterparts. The results reveal that there are no significant differences between the nanohardness values obtained for the SPS-KOBO extruded specimens either in the longitudinal or transverse cross-sections. Thus, both the SPS-KOBO microstructures seem to be very isotropic. In some cases, the variation in the deformation mechanism and texture formation may result in obtaining different values of nanohardness. This effect will be studied in future work.

### 3.3. Fractographic Analysis

The fractography of the SPSed tensile specimens was examined to determine the fracture modes of this composite with low ductility. As it is shown in Figure 11, most of the fracture surface was dominated by three fracture types:

- dimples indicating the occurrence of mostly ductile fracture (Figure 11c,f);
- areas of a flat microstructure (cleavage), indicating intergranular fracture (Figure 11b,e);
• dimples indicating plastic deformation in addition to traditional void nucleation and coalescence processes, together with cleavage features corresponding to brittle fracture (Figure 11a,d).

The SPSed specimens of the first fracture type exhibit a rough cellular microstructure with well-defined thick boundaries, indicating that the brittle material fracture mainly occurred along the grain boundaries but without their deformation (Figure 11a,d). Additionally, the SEM micrograph of the fractured SPSed specimens shows cleavage areas attributed to the completely brittle fracture of the composite (Figure 11b). A magnified view, Figure 11e, from the location shown in Figure 11b, reveals some cavities, which may be the microcrack nucleation sites. The cleavage area diagonals are 5–15 µm, which is similar to the grain size (Figure 5a), indicative of the brittleness of the coarse grains. However, some of them exhibit the features of a typical dimple failure with a dimple size of 1–3 µm (Figure 11c,f), where some tear ridges are also observed. These micrographs demonstrate a limited occurrence of the ductile mode of failure of the SPSed specimen.

The fracture topography micrographs of the SPS-KOBO extruded specimens after the tensile tests are presented in Figure 12. The SPS-KOBO extruded AA7075 exhibits the features of micron-sized dimple failure (Figure 12a,b), where some tear ridges can also be observed. This fractography clearly points out that the mode of failure in the SPS-KOBO extruded AA7075 is ductile. The micrographs in Figure 12c,d demonstrate that
the predominant fracture mode of the SPS-KOBO extruded AA7075 is ductile, in which homogeneous dispersion of the intermetallic precipitates can be seen along the fractured surface of the Al matrix. The intermetallic precipitates are similar to those found in the TEM micrographs (Figure 8e). The presence of numerous small dimples typical for ductile fracture confirms the plastic deformability of the SPS-KOBO extruded AA7075.

![Micrographs](a) (b) (c) (d)

Figure 12. Fracture topography micrographs of SPS-KOBO extruded AA7075 after tension test with various magnification: 5 kx (a); 25 kx (b); 50 kx (c); 100 kx (d).

The microstructural heterogeneities inherent to the SPS-KOBO extruded AA7075, including the distribution of second phase particles and the heterogeneous grain microstructure associated with polycrystalline materials, lead to localized dislocation accumulation. As shown in [26], the relationship between microstructural inhomogeneities and dislocation accumulation can be quantified and explored in detail. The authors of [26] showed that dislocations preferentially accumulate near the grain boundaries and intermetallic particles, with intermetallic particles leading to significantly higher levels of dislocation accumulation relative to the grain boundaries. These particles are assumed to be the sites of voids and crack nucleation (Figure 12d) due to high local strains achieved during the tension test, and the SPS-KOBO extruded composite micro-mechanical behaviour greatly depends on its dislocation microstructure, precipitates, and grain morphology.

4. Discussion

The effect of the dynamic recrystallization of the SPSed AA7075 due to the KOBO extrusion severe deformation process needs to be deeply discussed and analysed. The authors of [27] showed that the cyclic changes in the deformation path during KOBO extrusion led to the crossing of slip and forest dislocations, dislocation dipoles, and point
defects. This results in saturation of the crystalline with point defects, a decrease in the migration of self-interstitial atoms, and, consequently, dynamic recrystallization activation energies. The KOBO technology enables the extrusion of different alloys with an extremely high strain rate at even room temperature [12]. The authors of [13] note that, despite the fact that the KOBO process introduces severe deformation (extrusion ratio of $\lambda = 10$), there is no considerable strain induced phase transformation of the AA7075 microstructure. From this point of view, it seems reasonable to evaluate the dislocation microstructure parameters of both the SPSed and SPS-KOBO extruded specimens.

The experimental results demonstrate that the microstructure of the AA7075 specimens processed via the SPS and SPS-KOBO techniques (Figures 1, 4 and 5) is complex and contains regions of fully or partially refined grains and coarse grains with various dislocation densities. Thus, it is possible to state that the plastic deformation of the SPSed composites via KOBO extrusion does not occur homogenously. Therefore, the micromechanical characteristics of the plastic deformation of the SPSed composites is believed to be the superposition of deformation processes which occur in both the coarse and fine grains, because the dislocation activities depend on the grain characteristics [28,29]. That is the reason why the characterization of the microstructure and properties of the coarse and fine grains of the SPSed and SPS-KOBO extruded materials is important to shed light on the mechanisms of strengthening and dynamic recrystallization taking place.

Considering the strain gradient effects at the micro- and sub-microscale (0.1–50.0 $\mu$m) during the plastic deformation of polycrystalline materials [30], inhomogeneous distribution of the dislocations is one of the main microstructure parameters in the materials [30], and the dislocations can be described by two types: statistically stored dislocations (SSD) and geometrically necessary dislocations (GND). The SSD density is controlled by two competing dislocation generation and dissociation processes, which result in dislocations being arbitrarily distributed in the material. GND generation is a result of the strain gradient effects owing to the presence of a crystallographic texture and non-uniform loading conditions [30,31]. As shown in several papers [32–34], lattice continuity in the defect crystal lattice of the grains is accomplished by accommodation of the lattice curvature due to the generation of GNDs. Hence, the determination of the SSD and GND densities in grains of different sizes is important to fully characterize the AA7075 grain microstructure. In this paper, evaluation of the SSD density is made on the basis of the indentation size effect (ISE) characterization based on the strain gradient plasticity (SGP) theory developed in [35,36]. In the case of nanoindentation, ISE is described as:

$$\frac{H}{H_0} = \sqrt{1 + \left(\frac{h_1}{h}\right) \Theta} = \left(\frac{H}{H_0}\right)^2 = \left(\frac{1}{h}\right) + 1$$

where $H$ is the hardness, $h$ is the indentation depth, defined based on Figure 10 as $h = h_{total} - h_{elastic}$, $H_0$ is the intrinsic hardness defined by the SSD density in the absence of a strain gradient ($1/h \to 0$), and $h^*$ is the intrinsic length of the material. Parameter $h^*$ characterizes the depth dependence of the hardness [37]:

$$h^* = \frac{81}{2} b a^2 t g^2 \theta \left(\frac{\mu}{H_0}\right)^2$$

where $a$ is taken to be $\frac{1}{4}$, $b$ is the Burgers vector, $\mu$ is the shear modulus, and $\Theta$ is a half-angle of the indenter pyramid. Equation (1) predicts the linear dependence $(H/H_0)^2 = f(1/h)$, and $H_0$ may be determined from Equation (3):

$$H^2 = H_0^2 + \frac{81}{2} b a^2 \mu^2 t g^2 \frac{1}{h}$$
Based on the relation $H = 3\sqrt{3\alpha \mu b \sqrt{\rho}}$, the SSD density may be evaluated as:

$$\rho_s = \frac{H_0^2}{27(\alpha \mu b)^2}$$  \hspace{1cm} (4)

The experimental results of function $H^2 = f(1/h)$ and its linear approximation are shown in Figure 13 for both the coarse (Figure 13a) and fine (Figure 13b) grains.

Figure 13. $H^2 = f(1/h)$ functions and their linear approximation for coarse (a) and fine (b) grains. KOBO trans—transverse cross-section; KOBO long—longitudinal cross-section.

The linear approximation of $H^2 = f(1/h)$ for the SPSed coarse grains (grain size between 15–40 µm) fits Equation (1) with a simulation veracity of 0.95, which reveals the trustworthiness of the Nix and Gao strain gradient model [35]. It is worth mentioning that the simulation veracity of the linear approximations in the fine grain functions $H^2 = f(1/h)$
for the SPSed and SPS-KOBO extruded specimens is lower than that of the coarse counterparts due to the possible influence of some specific features of dynamic strengthening and softening processes, such as strain aging and recrystallization. The comparison of the $H_0$ and SSD density evaluated with Equation (4) (Table 4) highlights the effect of the decline in the dislocation density with the decrease in the grain size due to dynamic recrystallization processes. The SSD density defined by nanoindentation on the basis of the Nix and Gao model includes both SSDs and GNDs in the SPSed or SPS-KOBO extruded specimens. In fact, strain gradient analysis of the nanoindentation of separate grains allows their total dislocation density to be defined, which includes both SSD and GND dislocations generated during deformation processes and the formation of LAGBs and HAGBs. Nevertheless, it is impossible to define the real values of the SSD and GND densities. It will be analysed in detail using the high-resolution EBSD technique in future works.

The results obtained in this research clearly show that the combination of both the SPS and KOBO extrusion techniques allows a coarse/fine gradient grain microstructure to be obtained due to deformation and dynamic recrystallization, resulting in a good balance between high strength and ductility. The new AA7075 heterogeneous composite obtained by the SPS-KOBO technique demonstrates that microstructural heterogeneities can assist in overcoming the strength–ductility trade-off, similar to [24,25].

5. Conclusions

In this work, the microstructure and mechanical properties of spark plasma sintered and severely deformed AA7075 were studied. The main conclusions are as follows:

- It was found that the microstructure synthesized by the SPS process was inhomogeneous, formed by coarse and fine grains randomly distributed within the matrix, and developed into a coarse- and fine-grained laminated microstructure by a subsequent KOBO extrusion process at room temperature. The dominant microstructure formation mechanisms were dislocation accumulation and dynamic recrystallization during SPS plus KOBO extrusion severe deformation processes;
- The ultimate tensile strength (UTS) of the AA7075 rose after the SPS-KOBO severe deformation up to 400 MPa, exhibiting high strain of 33%. This effect was achieved due to the formation of a coarse–fine laminated grained microstructure;
- It was found that the intermetallic precipitates seemed to be preferred sites for the nucleation of voids and cracks due to the high local strains achieved during the tensile tests, and the SPS-KOBO extruded composite micro-mechanical behaviour greatly depended on its dislocation microstructure, precipitates, and grain morphology;
- Based on the strain gradient analysis of the nanoindentation of separate grains, the total dislocation density was evaluated. A drop in the dislocation density together with a decrease in the grain size was highlighted owing to dynamic recrystallization processes.

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