

## THE INVESTIGATION OF THE Al7075 + 1 WT. % Zr ALLOY PREPARED USING SPARK PLASMA SINTERING TECHNOLOGY

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### Abstract

The microstructure and mechanical properties of powder metallurgical Al7075 alloy modified by the addition of 1 wt. % Zr were studied. The gas atomized powder was consolidated by spark plasma sintering (SPS) to a fully dense compact. The powder particles of the typical size below 50 µm had a cellular to columnar or equiaxed dendritic microstructure (the size in the order of µm) with a large fraction of intermetallic phases at the boundaries. During sintering at 500 °C for 3 min the grain structure remained nearly unchanged. Optical microscopy revealed a bimodal microstructure. The grain size remained unchanged during annealing at even 500 °C for 10 hours. The microhardness of the SPS compact material was 150 HV. The annealing at 500 °C for 10 hours resulted in a decrease of microhardness to 110 HV. Natural aging at room temperature gradually enhanced the microhardness to its initial value. Tensile tests performed on naturally aged samples at room temperature revealed a brittle behaviour. Ductility of 14 % was found at 500 °C. Both the measurements of the strain rate sensitivity parameter  $m$  and results of light and scanning electron microscopy revealed the operation of grain boundary sliding.

**Keywords:** Al7075 alloy, zirconium, spark plasma sintering, powder metallurgy

### 1. INTRODUCTION

The precipitation strengthened Al-Zn-Mg-Cu alloys (series 7XXX) are widely used in transportation and construction industry. Their high strength can be achieved by a suitable heat treatment consisting of solution annealing followed by artificial aging at elevated temperatures or natural aging at room temperature [1]. The strength can be also influenced by the grain size. A reduction in grain size enhances the strength at room temperature due to the strengthening effect of grain boundaries. On the other hand grain boundaries can act as sites of dislocation annihilation at elevated temperatures which can reduce the strength and result even in superplastic behaviour. Grain boundaries can also influence the precipitation sequence as they are preferential sites for precipitation of strengthening phases. The fine grained structure can be produced by a large variety of methods - e.g. by a special thermomechanical treatment resulting in a statically recrystallized microstructure [2] or by equal channel angular pressing resulting in the grain size in the sub-microcrystalline range [3]. Alternatively, the powder metallurgical route can be used for the production of materials with a very fine microstructure. The main problem of the powder metallurgical route is to retain this microstructure during the consolidation step performed usually at high temperatures [4].

Spark plasma sintering (SPS) represents a relatively novel technique for consolidation of powders [5]. This technique combines pressure with heating by low voltage pulsed DC current flowing through the sample. High current density and large Joule heat can be evolved at contact places between powder particles where the temperature highly exceeds the set one. This way, the sintering occurs at these affected surfaces whereas the particle cores retain their original microstructure. The rapid heating and reduced sintering time avoid undesirable processes like recrystallization or grain growth.

In this work, the Al7075 alloy modified by the addition of Zr as grain stabilizer was prepared by the combination of gas atomization and SPS. The microstructure and mechanical properties were investigated after various thermal treatments in order to verify especially the stability of the material at elevated temperatures.

## 2. EXPERIMENTALS

The Al7075 + 1 wt. % Zr alloy (the composition is given in **Table 1**) was remelted in silica crucibles of the high energy gas atomizer at 900 °C and the melt was atomized with nitrogen and then passivated in 2 steps in order to achieve a controlled oxidation. The resulting powder was sieved and the fraction with the powder particle size below 50 µm was used for further experiments.

The powder was consolidated by SPS using the following scheme: free heating from room temperature to 425 °C, heating to 500 °C with the heating rate of 100 °C/min simultaneously with the pressure increase up to 60 MPa, holding at 500 °C for 180 s, free cooling with parallel unloading. The cylinders with the radius of 25 mm and height of 10 mm were prepared with nearly 100 % density.

Morphology and microstructural investigation was performed both on powder and sintered samples using light microscope Olympus IX70 and scanning electron microscope (SEM) FEI Quanta 200F which was equipped by EDAX Trident for X-ray analysis. The electron back scatter diffraction (EBSD) experiments were carried out using the high-resolution field-emission scanning microscope LEO 1530 (Carl Zeiss) equipped with a Nordlys II (Oxford Instruments) detector.

The microhardness was measured using an automatic microhardness tester Qness Q10A+ with Vickers indenter and applied load of 50 g. The microhardness of powder material was measured on particles embedded in an acrylic cold mounting resin. The measurement on compacted samples was performed on planes parallel to the direction of the applied stress during sintering. In order to obtain good statistics the areas of 6 x 6 mm were investigated with the distance between individual indents of 200 µm. The duration of microhardness measurement on each sample was about 10 h.

Tensile tests were performed both at room temperature and 500 °C using an Instron 5882 machine. Rectangular dog bone shaped tensile specimens with a gauge length of 17 mm and cross section 5.8 mm x 1 mm were machined from compact material parallel to the direction of applied stress during SPS. The strain rate sensitivity parameter

$$m = \frac{\log \frac{\sigma_1}{\sigma_2}}{\log \frac{\dot{\epsilon}_1}{\dot{\epsilon}_2}} \quad (1)$$

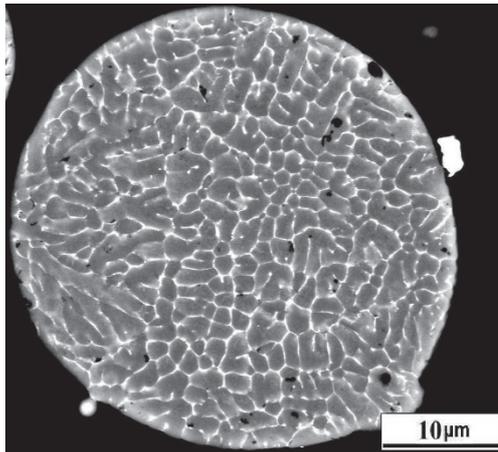
was evaluated by the strain rate change method. The tensile specimen was pre-strained at the strain rate of 10<sup>-3</sup> s<sup>-1</sup> up to 5 % of elongation, where the strain rate was abruptly reduced to 10<sup>-4</sup> s<sup>-1</sup>. Afterwards, the strain rate was increased in small steps up to fracture. Some tensile specimens were polished prior to straining and their surface after straining was studied using SEM.

**Table 1** The composition of examined alloy from energy dispersive spectroscopy

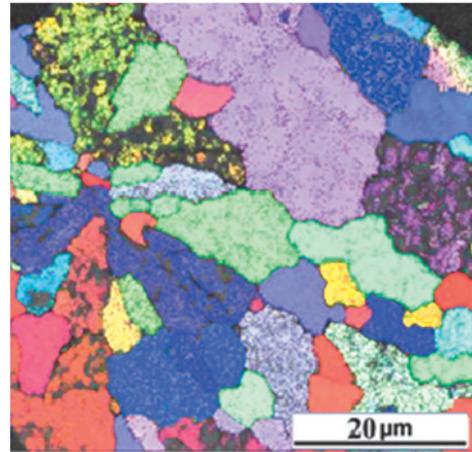
Element	Zn	Mg	Cu	Zr	Si	Al
wt. %	5.7	2.4	1.8	1.0	0.36	balance

### 3. RESULTS

The morphology and internal structure of nearly spherical powder particles is shown in **Fig. 1**. Cellular, columnar and dendritic microstructure with intermetallic phases located predominantly at boundaries was formed during rapid solidification. EBSD experiments performed on powder particles revealed the presence of grains divided by high angle boundaries (**Fig. 2**).

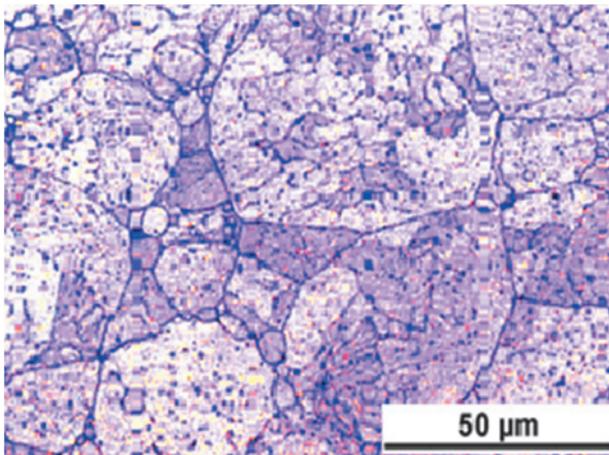


**Fig. 1** The microstructure of a powder particle, SEM

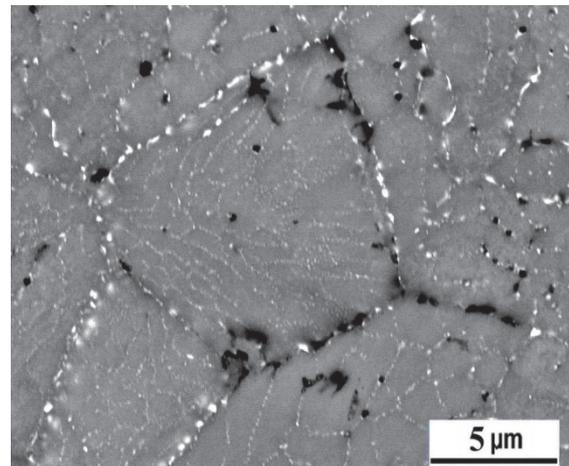


**Fig. 2** The EBSD micrograph of a powder particle

The microstructure of sintered samples exhibits a bimodal character (**Fig. 3**). The original powder particles of the size reaching up to 50  $\mu\text{m}$  are still clearly visible. Numerous smaller grains with the size in micrometer range can be found along boundaries of original powder particles. SEM investigation revealed that the internal microstructure of original powder particles was retained during SPS. The only difference can be found in the distribution of intermetallic phases. The continuous distribution of intermetallic phases along intercellular or interdendritic boundary regions observed in powder particles was replaced by discrete precipitates arranged into chain-like structures (**Fig. 4**).

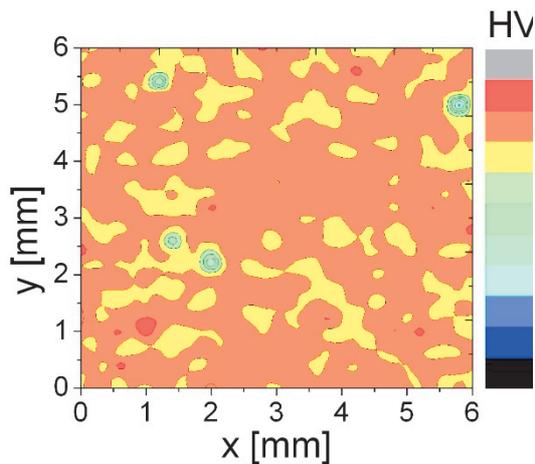


**Fig. 3** The microstructure of the sintered material, light microscopy

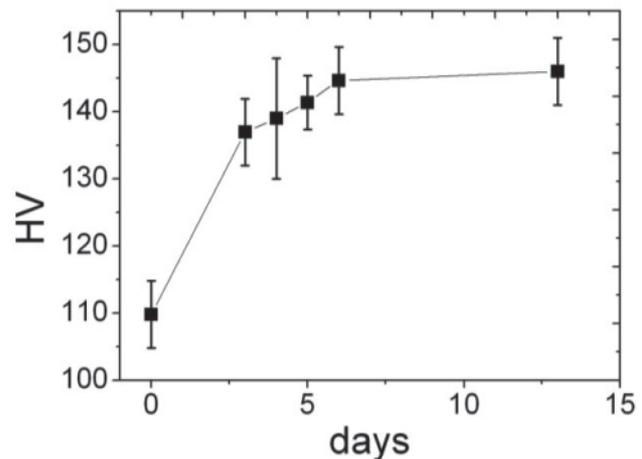


**Fig. 4** The microstructure of the sintered material, SEM

The results of microhardness measurements are summarized in **Table 2**. Microhardness was found to be close to 100 HV in the powder material and nearly 150 HV in the sintered material. The microhardness map obtained from a large area revealed a very good homogeneity of the sintered material (**Fig. 5**). The influence of heat treatment on microhardness was tested on the sintered material. Annealing at 500 °C for 5 and 10 h followed by water quenching resulted in a microhardness decrease to about 110 HV. Natural aging of the material annealed at 500 °C for 10 h was accompanied by a gradual increase of microhardness to values corresponding to the initial sintered material (**Fig. 6**).



**Fig. 5** The microhardness map of the sintered sample



**Fig. 6** The influence of natural aging on the microhardness for the sample annealed at 500 °C for 10 h

**Table 2** The dependence of microhardness on the heat treatment temperature and its duration

Material	Powder	Sintered		
Temperature of annealing (°C)	room	room	500	500
Duration of annealing (h)	undefined	undefined	5	10
HV	95 ± 18	148 ± 8	113 ± 5	110 ± 5

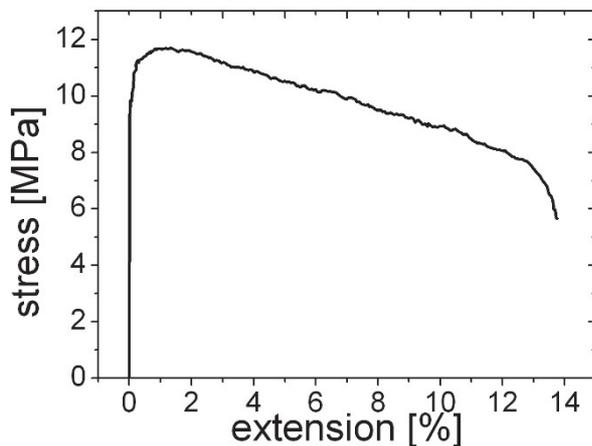
The tensile tests revealed a brittle behaviour of studied material at room temperature. High temperature mechanical properties were tested at 500 °C. The corresponding deformation curve in **Fig. 7** shows ductility of 14 %. The strain rate sensitivity parameter  $m$  was found to be 0.3. **Fig. 8** shows the surface of the preliminary polished sample after tensile test at 500 °C. The micrograph shows the operation of grain boundary sliding predominantly along boundaries of original powder particles.

#### 4. DISCUSSION

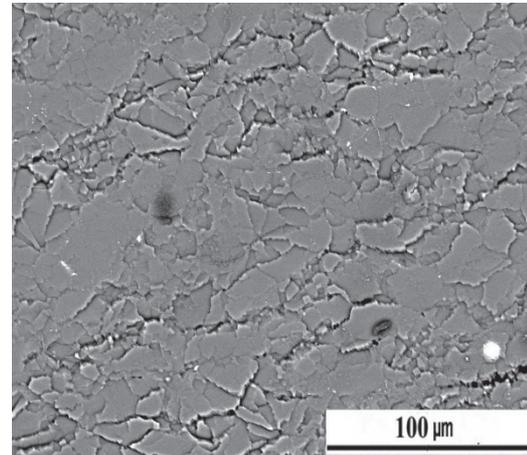
Gas atomization can be used for the preparation of fine grained materials. This positive influence of high solidification rate was confirmed also in our Al7075 + Zr alloy where a fine internal structure was observed in powder particles. Because of high content of alloying elements the boundaries between cells or dendrites are decorated by continuous layers of intermetallic phases. Because of mostly very different crystallographic orientation the cells can be considered as grains.

Spark plasma sintering was selected for the consolidation of powder particles. During this process, the powder material is exposed to elevated temperatures for a relatively short time and only regions close to the contact points between powder particles are strongly affected. The particle interiors are nearly unaffected and retain their fine-grained structure. The sintering temperature of 500 °C is sufficient for the processing of compact

material with a nearly 100% density. On the other hand the temperature is low enough to prevent the changes in internal grain structure, especially the grain growth. The microstructural stability is also supported by the presence of numerous intermetallic phases located along internal boundaries in original powder particles and also by the presence of Zr. This element forms in Al-based alloys small particles of the  $Al_3Zr$  phase which are considered as one of the most efficient inhibitors for grain boundary migration [6].



**Fig. 7** The tensile test curve of the sample strained at 500 °C with a strain rate of 1 mm/min



**Fig. 8** The surface of the sample with a mirror-like polished surface after the tensile test at 500 °C

Microhardness measurements were performed both on the powder and sintered materials. A large standard deviation observed in the powder material can be explained by the fact that only one indent was applied to each powder particle. Thus, the measured values of HV correspond to different powder particles which can have slightly different solidification microstructure. On the other hand, a low standard deviation of microhardness in sintered material proves its very good homogeneity. The microhardness of the Al7075-based alloys depends strongly on their phase composition, i.e. on the history of thermal treatment. The peak values of microhardness close to 160 HV were found in materials containing a dense distribution of metastable  $\eta'$ - $MgZn_2$  particles [7]. Our powder material contains predominantly continuous layers of the  $(Zn,Al,Cu)_{49}Mg_{32}$  phase with a low fraction of  $Mg_2(Zn,Al,Cu)_{11}$  phase [8]. Consequently, relatively low values of microhardness were found. During sintering the material was exposed to the temperature of 500 °C which is higher than the temperature of solution treatment. It can be therefore expected that the intermetallic phases will be at least partially dissolved. After sintering the compact material was free cooled and during this stage new particles were formed usually as chains of discrete precipitates along boundaries. The fraction of  $(Zn,Al,Cu)_{49}Mg_{32}$  phase decreased at the expense of  $Mg(Zn,Al,Cu)_2$  phase [8]. These changes in phase composition are responsible for high microhardness values of the sintered material.

The observed changes in microhardness during thermal treatment are similar to those observed in classical ingot metallurgical materials. Annealing at 500 °C followed by water quenching represents the solution treatment resulting in a significant softening. A gradual time dependent increase in microhardness reflects the natural aging and formation of strengthening precipitates from the oversaturated solid solution formed during solution treatment.

The brittleness of sintered materials reflects probably the presence of oxide particles at the surface of original powder particles. The formation of cracks can be partially suppressed at elevated temperatures. Nevertheless the ductility even at 500 °C remains low. The measurement of the parameter  $m$  and the observed grain boundary sliding suggests the possibility of superplastic behaviour in case that the negative influence of oxides is removed.

## 5. CONCLUSION

The microstructure and mechanical properties of the Al7075 + 1 wt. % Zr alloy prepared by gas atomization and spark plasma sintering were investigated. The microstructure of both powder and sintered material is fine grained. The composition and morphology of strengthening phases is changed during sintering. Consequently, the microhardness of sintered material is higher. The microhardness of the sintered material can be changed by thermal treatment similarly to classical ingot metallurgical Al7075 alloys. The fine microstructure is preserved even during long term annealing at 500 °C. The mechanism of high temperature plastic deformation is similar to that observed in superplastic materials however ductility is reduced by oxide particles on the surface of original powder particles.

## ACKNOWLEDGEMENTS

*This work was financially supported by the grant GACR 15-15609S. O.M. is grateful for financial support from the grant SVV-2014-260091 and acknowledges the support from the Grant Agency of Charles University under contract Nr. 1882314 and 946213.*

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