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Spark plasma sintering and spark plasma upsetting of an Al-Zn-Mg-Cu alloy

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ABSTRACT

Al-Zn-Mg-Cu alloy powder Alumix 431D was sintered at 400 °C by spark plasma sintering (SPS) and upset forging was applied to the sintered sample through SPS. Densities of 99.1 ± 0.3% and 99.8 ± 0.1% of theoretical were obtained for the sintered and forged samples, respectively. T6 temper was carried out on the samples and microstructure analysis and mechanical properties before and after heat treatment were evaluated. Microhardness of 173 ± 3 and 172 ± 3 HV were attained in the T6 temper of as-sintered and forged samples, respectively. The flexural strength and strain values were significantly improved after the forging process, which can be mainly attributed to the better particle bonding in addition to the occurrence of some recrystallization. Significant loss in the ductility was observed after the T6 temper.

1. Introduction

Powder metallurgy (P/M), which is the manufacturing of parts from powder material, offers high material utilization, production of near-net shaped parts, and reduction or elimination of the costs related with complex machining operations [1–3]. Al P/M alloys have a wide range of applications such as hand tools, office machinery and automotive. Nevertheless, the properties of the conventional press and sinter parts are frequently insufficient for many of the more demanding applications envisioned for the technology [3]. Alumix 431D alloy, is a commercial powder blend composed of a mixture of pure Al with a master alloy containing Al, Zn, Mg and Cu. This powder blend is chemically similar to AA7075, and it is used in the fabrication of products requiring low weight and high strength [4].

A technical challenge inherent to P/M processing of Al alloys is the native oxide film present on the powder particles. This tenacious oxide layer of Al is highly stable from a thermodynamic standpoint and will prevent sintering if left intact during the processing cycle [5]. Thus, it is necessary to break the oxide layer in order to form appropriate metallurgical bonding between powder particles. Successful sintering of high strength Al alloys such as Alumix 431D alloy, can be obtained by liquid phase sintering, which is capable of disrupting the stable oxide layer covering the Al particles [6]. However, this manifests heterogeneous shrinkage of the powder compacts which is disadvantageous in the context of near-*net*-shape processing [6].

Spark Plasma Sintering (SPS) is an axial pressure-assisted process that uses joule heating from the passage of a high DC pulsed current through a graphite die and powder [7]. Solid state transport mechanisms (bulk and grain boundary) are favored during the process [8]. SPS is considered as an important process due to the ability to clean particle surfaces [9]. This is accomplished by contributing to the rupture of the oxide layer at the surface of the pure aluminum powder, which has been demonstrated in previous studies [10–17]. However, it has been shown by Garbiec and Siwak [18] and Rudinsky et al. [19] that Alumix 431 powder sintered by SPS exhibits low ductility during tensile or bending tests due to the lack of bonding between particles. It has been suggested that the formation of the spinel layer arising from the reaction of Mg and the alumina layer would impose the requirement for a higher applied load to break the oxide layer [20]. Alternatively, forging of aluminum P/M products is known to close the residual porosity and disrupts the oxide film around the powder particles because of the high strains and strain rates involved [1]. This enhances the metallurgical bonding across the interfaces where the oxide layer is broken [1]. Upsetting is a type of forging where a workpiece is reduced in dimension between two parallel plates and reduction in initial height without extensive spreading and substantial flow along the tool surface occurs [21].

The purpose of this study is to investigate the effect of hydraulic press forging [22] through SPS upsetting on the mechanical properties of Alumix 431D compact which is sintered by SPS. Bulk mechanical properties were measured through three-point bending test and correlated with fracture surface analyses in order to assess the nature of particle bonding. In addition, electron backscattered diffraction (EBSD) was also utilized to analyze the nominal grain size and grain boundary

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Table 1
Chemical composition (wt%) of the Alumix 431D powder utilized.

Al	Zn	Mg	Cu	Sn
Bal.	6.1	2.5	1.6	0.3

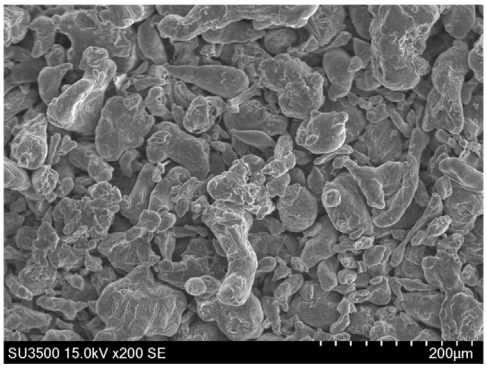

Fig. 1. SEM image of Alumix 431D powder.

mis-orientation angles after upset forging by SPS.

2. Experimental

Alumix 431D powder from ECKA granules with a chemical composition provided by the supplier's certificate of analysis, listed in Table 1, was used in the present study. A scanning electron microscopy (SEM) image of the starting powder is shown in Fig. 1. The morphology of the powder exhibits irregular shape as a result of production by air atomization.

A two-step process has been applied to the powder using SPS where the first step was the sintering of the powder and the second step was forging. A schematic of this process is presented in Fig. 2. Six cylindrical samples of 10 mm in height by 20 mm in diameter were spark plasma sintered using an SPS 10-3 apparatus from Thermal Technologies Inc., and using a 20 mm diameter Isocarb graphite I-85 die-punch set from Electrodes Inc. Sintering was performed at a temperature of 400 °C under 50 MPa of applied pressure and a 60 min holding time. The initial heating rate to reach 350 °C was 100 °C/min and a rate of 50 °C/min was applied subsequently up to 400 °C in order to avoid overshooting the target temperature. Temperature was measured using a C-type thermocouple placed in the bottom punch which had a hole drilled to 2 mm from the surface of the sample. The pressure was constant throughout the sintering cycle. Apart from sintering, a secondary forging process that imposed 32% compressive strain was applied to three sintered samples using the SPS apparatus. This represented the maximum amount of deformation that could be applied due to the limited length of the punch. This process was carried out by placing the 20 mm diameter sintered samples in a larger (25 mm diameter) Isocarb graphite I-85 die-punch set and heating them to 400 °C. All specimens were heated from ambient to 350 °C at rate of 100 °C/min and then at 50 °C/min from this point to the intended forging temperature of 400 °C. The samples were kept at 400 °C for a period of 4 min and then deformed at that temperature under a maximum pressure of 36 MPa, achieved by loading at 0.55 MPa/s which took 1 min to reach full pressure. The forged products were then cooled down to room temperature and removed from the SPS unit for analysis.

Bulk density was measured on sintered, forged and heat treated samples using the Archimedean method as described in ASTM standard B963-13 [23]. In order to heat treat the produced samples, differential scanning calorimetry (DSC) was first conducted with a NETZSCH STA 449F3 instrument to determine the initial melting temperature. For this purpose, 11 mg of the forged samples were heated up to 650 °C under flowing argon while using a heating rate of 10 °C/min. An initial melting event with a peak temperature of 475 °C, which corresponded to the melting of $MgZn_2$ phase [24], has been observed. Therefore, a solutionizing temperature of 450 °C was adopted. T6 temper was applied to both sintered and forged samples using 4 h solutionizing at 450 °C followed by artificial aging at 125 °C for 24 h after water quenching. The nomenclature and their descriptions for all of the samples are given in Table 2.

Sintered and forged samples (before and after heat treatment) were mounted, ground and polished using 320 and 400 grit papers, followed by 9 and 3 μm diamond suspensions with an end step of colloidal silica suspension on a Vibromet polisher. Microstructural analysis was performed with backscattered electron (BSE) imaging and energy dispersive spectroscopy (EDS) using a Hitachi SU3500 SEM. Electron backscattered diffraction (EBSD) was used to analyze the grain microstructure. X-ray diffraction (XRD) analysis was carried out using a Bruker D8 Discovery X-Ray Diffractometer (Cu-source radiation).

Mechanical properties were evaluated by microhardness and three-point bending tests. Microhardness was measured with a Clark Microhardness (CM-100AT) indenter using a load of 10 g. The reported hardness values represent an average of 10 indentations. Three-point bending tests (sample dimensions: 1.8 mm × 4.3 mm × 20 mm) were

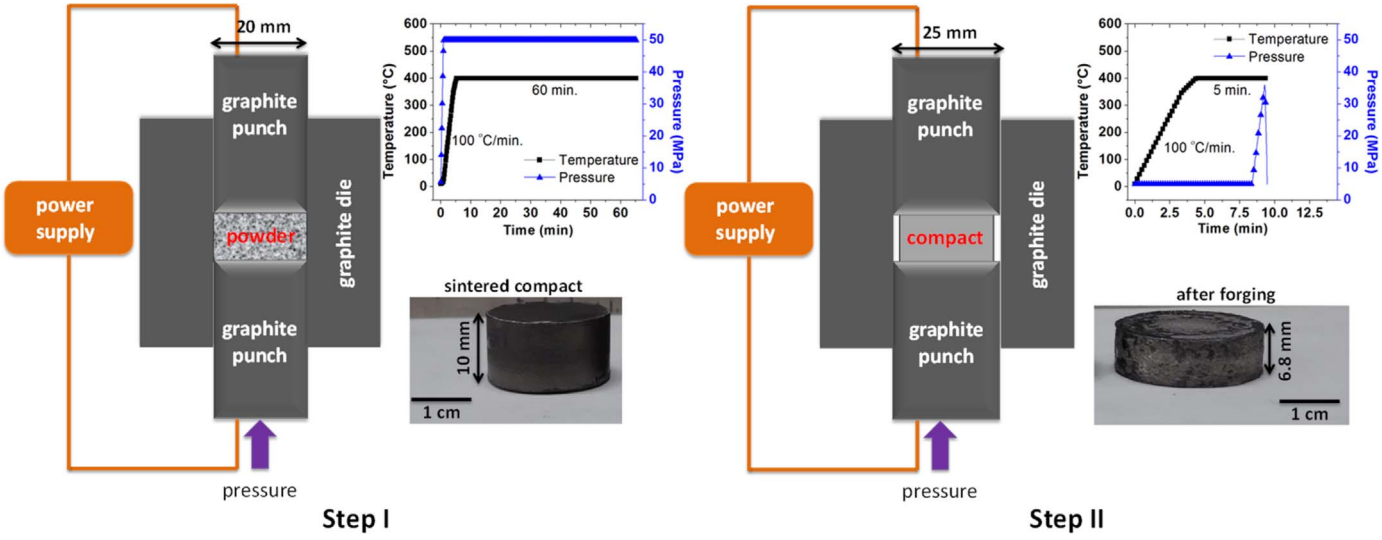

Fig. 2. Schematic of the two-step SPS process used in this study.