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Microstructural and mechanical characterisation of 7075 aluminium alloy consolidated from a premixed powder by cold compaction and hot extrusion

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ABSTRACT

The present work concerns the processing of 7075 Al alloy by cold compaction and hot extrusion of a premixed powder. To this end, a premixed Al–Zn–Mg–Cu powder, Alumix 431D, was uniaxially cold pressed at 600 MPa into cylindrical compacts 25 mm in diameter and 15 mm thick. Subsequently, selected green compacts were subjected to either a delubrication or presintering heat treatment. Extrusion of the powder compacts was performed at 425 °C using an extrusion ratio of 25:1. No porosity was present in the microstructures of the extruded alloys. Heat treatment prior to extrusion had a great effect on the degree of alloy development in powder compacts and, as a direct consequence, remarkably affected the extrusion process and the as-extruded microstructures and mechanical properties of the processed materials. Hot extrusion caused banded structures for the alloys consolidated from the green and delubricated powder compacts. The alloy extruded from the presintered powder compact showed a fine, recrystallized microstructure which resulted in a superior combination of mechanical properties for the consolidated material.

1. Introduction

Having a superior combination of properties, such as high strength and fracture toughness [1], low density [2,3], good workability and weldability, and remarkable stress corrosion cracking resistance [4], Al–Zn–Mg–Cu alloys have long been regarded as some of the best candidates for demanding structural applications in the aerospace and automotive industries. In fact, 7xxx series Al alloys represent some of the highest strength Al alloys in commercial use [5].

Al powder metallurgy combines the superior properties of Al with the ability of powder metallurgy (PM) to produce high-performance, net- or near-net-shaped parts, thus reducing or eliminating the capital and operating costs associated with intricate machining operations. Powder extrusion (PE) is a PM processing method that has been developed for the production of fully dense, high-performance materials from powders. Compared with other PM routes such as sintering and hot pressing, the shear stresses involved in PE make it an ideal process for the production of bulk Al alloys and composites from powder mixtures. One of the main difficulties in sintering Al-based alloys is the presence of a surface oxide layer intrinsic to Al-based powders. In the case of PE, the shear stresses break the oxide layer covering the particle surfaces of these powders, leading to a well-bonded microstructure and superior after-extrusion mechanical properties [6]. According to Verlinden et al. [7], there are three methods for the extrusion of powder mixtures: loose powder extrusion, green billet extrusion, and powder extrusion using canning and degassing. The third method is mainly used for Al powders [8], but canning and degassing constitute a costly and inconvenient processing step. Consequently some researchers made efforts towards the extrusion of Al powder mixtures without canning and degassing [9,10]. These researchers directly extruded the green billets cold compacted from Al powder mixtures using a mixture of graphite and oil serving as both a lubricant [11] and an oxidation barrier [12]. They studied the microstructures and mechanical properties of the extruded products. Their promising results imply that the employed method can replace powder extrusion using canning and degassing for Al-based powders [13].

Compared with premixed Al-based powders, pre-alloyed powders are generally harder and exhibit higher flow stresses. As a result, their compressibility and hot deformability are lower than those of the premixes. In fact they are more difficult to process. Furthermore, the green density of a powder compact affects the as-extruded density and the mechanical properties of the extruded product. Song and He [14] studied the effects of die-pressing

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pressure and extrusion on the microstructures and mechanical properties of SiC reinforced pure Al composites. Their results indicate that a higher green density results in a higher as-extruded density and improved mechanical properties. Thus, the lower green densities obtainable for pre-alloyed powders can reduce after-extrusion density and, as a direct consequence, negatively affect the mechanical properties of the extrusion product. In this context, the processing of fully dense Al alloys by hot extrusion using premixed Al-based powders is of great importance.

In the past decade, premixed Al-based powders have attracted great attention. These powders are ready-to-press and can be sintered to high densities through liquid phase sintering. A number of leading powder producers have addressed this interest and developed commercial premixes mainly based on high strength Al alloys. Included are Alumix 13 and 123 (2xxx series), Alumix 321 (6xxx series), and Alumix 431D (7xxx series) produced by Ecka Granules, Germany. Martin and Castro [15] studied the sintering behaviour of the mentioned premixes and reached a maximum sintered density of 97% of the theoretical value. Min et al. [16] examined the sintering characteristics of AMB 2712 (2xxx series), a commercial premixed powder produced by Ampal, USA. Their sintering processes yielded PM parts with a maximum relative density of 93%. Porosity has a very detrimental effect on the mechanical properties of PM alloys. In addition, the liquid phase sintering of Al alloys causes distortion, which is problematical to accommodate during design and to modify after sintering [17]. Therefore, development of full density, net-shaped processes and optimisation of processing parameters for the consolidation of premixed Al-based powders is of great significance.

Pre-alloyed powders have been used in most of the previous works on the extrusion of Al-based powders, and studies on the extrusion of PM Al premixes are scarce. In a previous work by Zubizarreta et al. [18], Alumix 13 premix which is basically a mixture of elemental Al and Cu powders has been consolidated by hot extrusion. Although different heat treatments prior to extrusion have been performed, all of the bulk alloys processed by these researchers show banded microstructures and contain porosity and pure Cu particles, thus resulting in low hardness values for the extrusion products. Premixed powders are generally considered unsuitable for powder extrusion mainly because of the development of inhomogeneous and banded microstructures through the extrusion process, which may, however, be prevented by using a suitable premix and designing an appropriate processing route based on systematic thermal, microstructural, and mechanical characterisation of the alloys under study at different stages of the consolidation process. Considering all of the above, the present study examined the processing of 7075 Al alloy by direct hot extrusion of powder compacts cold pressed from a commercial Al–Zn–Mg–Cu premix. The main objective was to produce a bulk alloy with homogeneous microstructure and superior mechanical properties from the employed premixed powder.

2. Experimental procedure

The raw material used for this study is a commercially available premixed Al–Zn–Mg–Cu powder, Alumix 431D (Ecka Granules, Germany), with a chemical composition equivalent to 7075 Al alloy (5.6–6.4 wt% Zn, 2.4–3 wt% Mg, 1.5–2 wt% Cu, 0.1–0.3 wt% Sn, and the balance Al). The main component of this premix is atomised pure Al powder, which is mixed with a master alloy powder containing all of the alloying elements. As this mixture is a ready-to-press blend, it typically contains 1.5 wt% lubricant to facilitate the pressing step.

This premix was uniaxially cold pressed at 600 MPa into cylindrical billets 25 mm in diameter and 15 mm thick. Subsequently, selected green compacts were subjected to either a delubrication (heating to 400 °C and holding at this temperature for 20 min) or presintering (heating to 400 °C and holding at 400 °C for 20 min, followed by heating to 525 °C and soaking at this temperature for 45 min) heat treatment. The heating rate for all of the heating steps was 5 °C/min; and both heat treatments were performed in a high-purity nitrogen atmosphere. After heat treatment, the samples were furnace cooled to room temperature.

Extrusion of the powder compacts was performed at 425 °C, without caning and degassing, using an extrusion ratio of 25:1 to ensure full density after extrusion. Before heating to the extrusion temperature, a thin layer of a mixture of graphite and oil was applied to the surfaces of the powder compacts, serving as both a lubricant and an oxidation barrier. The compacts were extruded to form rods 5 mm in diameter and approximately 350 mm long. After extrusion, the extruded rods were air cooled to room temperature.

To investigate the microstructural evolution of the powder compacts during heating to extrusion temperature, selected compacts were impregnated with the graphite mixture, heated to 425 °C in air, and water quenched from this temperature.

Simultaneous thermal analysis (STA), scanning electron microscopy (SEM), X-ray diffractometry (XRD), and hardness, tension, and compression testing were employed for the microstructural and mechanical characterisation of the as-received premix, the powder compacts, and the extrudates. The reported hardness value for each material is the average value of twelve measurements. Tension and compression testing were conducted in accordance with the **ASTM: E8M** and **ASTM: E9** standards, and the number of specimens for tension and compression testing of each extruded alloy were five and three, respectively.

Both heat-treated and non-heat-treated powder compacts were sectioned parallel to the pressing direction for the microstructural and mechanical analyses. The sectioned samples were then mounted in a conductive resin, ground, and polished with diamond paste. The microstructures of the powder compacts were studied by SEM using a Philips XL30 microscope in backscattered electron (BSE) mode (accelerating voltage: 15 kV). Several samples were etched using Keller etchant prior to SEM imaging. XRD was also employed for the qualitative analysis of the phases present in the microstructure. A Philips diffractometer with Cu Kα radiation (λ = 0.15406 nm) generated at 40 kV and 40 mA was used for the XRD measurements. The XRD patterns were recorded in the 2θ range of 15–60° (step size: 0.05°, time per step: 20 s).

For the characterisation of the extruded materials, extrudates were sectioned parallel to the extrusion direction (i.e., longitudinal sections). To eliminate the possible effects of microstructural differences between different parts of the extrudates, all of the samples were cut from the middle of each extruded rod. The samples were then mounted and prepared using the same conventional metallographic techniques described above for the powder compacts. The extruded samples were characterised by SEM and XRD using the same parameters introduced before.

3. Results and discussion

3.1. Heat treatment prior to extrusion

The aim of the delubrication heat treatment was elimination of the pressing lubricant to study the effect of this lubricant on the as-extruded microstructure and mechanical properties of the consolidated alloy. Based on the STA curves presented in Fig. 1, temperatures as high as 350–400 °C are needed for complete elimination of the lubricant. Thus, heating to 400 °C and soaking at this