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Metal injection moulding of non-spherical titanium powders: Processing, microstructure and mechanical properties

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ABSTRACT

The critical processing parameters for metal injection moulding (MIM) of titanium using inexpensive non-spherical hydride-dehydride (HDH) powders with an average particle size of 45 μm were established through a series of laboratory scale injection moulding investigations. A slow heating rate of 1.0 K/min during thermal de-binding and sintering resulted in samples with negligible or limited distortion and nearly uniform shrinkage in all directions. Sintering at 1250 °C for 120 min was identified to be a suitable condition for MIM of HDH Ti, which produced samples with a relative density of 96.5%, uniform shrinkage of 13.9% along the length direction and 14.7% along the thickness direction. The as-sintered samples achieved a tensile strength of 395 MPa and elongation of 12.5%. The promising tensile elongation indicates that MIM of HDH titanium powder can cope with the relatively high oxygen level in the initial inexpensive HDH Ti powder as well as the unavoidable further increase in oxygen level during the MIM cycle.

1. Introduction

Titanium (Ti) and its alloys are advanced metallic materials with a range of unique properties including high strength to density ratio, superior corrosion resistance and excellent biocompatibility [1]. These properties make Ti products excellent candidates for many applications in chemical processing, aerospace, marine engineering, medical devices and implants, luxury goods and many other industry sectors [1]. However, the manufacture of Ti parts through traditional methods, such as machining from forged blank, is difficult and costly, often leading up to 90% being scrapped [2].

A number of new techniques have been developed over the recent decades and most of them are based on powder metallurgy (PM). Metal injection moulding (MIM) is one such development [3–5], which is the adaptation of PM into a plastic injection moulding technique. MIM combines the attributes of PM (e.g. low cost, simplicity, and flexibility of composition selection) with those of plastic injection moulding (e.g. the ability to manufacture complex parts and rapid production). This combination has enabled the MIM process to overcome the limitations of the slow production rates and shape restraint of traditional press-and-sinter PM process, the high cost and difficulties of machining, and the rough surface of conventional casting [6]. Also compare with the additive manufacturing (AM) techniques, MIM has the ability for economic manufacturing of small components in very high quantities, where is so challenging with AM or any other PM based techniques. Consequently, MIM has been widely used to produce small-to-medium sized intricate parts at high production volumes, and economic manufacturing of many of those parts is impossible by means of any other manufacturing techniques.

MIM is a simple process but need specific consideration for manufacturing of high quality components. This process is explained in detail elsewhere [7–9], but basically involves mixing of metal powder and polymer (binder) to form a feedstock with good flow-ability. The feedstock then injected into metallic dies using conventional plastic injection moulding machines. This fluid feedstock enables the MIM process to be used for manufacturing of geometrically complicated components [7]. The moulded parts (known as green parts), then subject to a usually two-step de-

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binding process to remove the binder components followed by sintering at high temperatures to produce a solid part. For Ti and its alloys, all the mixing, de-binding and sintering processes should be performed under protected environment to prevent any oxygen pickup.

The application of MIM to the manufacture of Ti components (Ti-MIM) has been under development for a number of years [3,9,10] but progress to date has been slow. The main barriers are (i) the high cost of low-oxygen fine ($\leq$45 μm) spherical powders, (ii) the easy pickup of oxygen and carbon from the binder during the MIM cycle, and (iii) the lack of successful business cases to encourage significant investment into Ti-MIM . Recent research has shown that the second and third issues are manageable through control of the binder system, mixing process, de-binding and sintering [4,9,11–14]. However, the high cost of fine spherical titanium powders remains significantly challenging. Although the issue is being actively addressed by the titanium additive manufacturing (AM) industry as the same fine titanium powder ($\leq$45 μm) is required for laser powder-bed-fusion based AM processes [2], no quick solutions seem to be available. On the other hand, non-spherical hydride-dehydride (HDH) titanium powder with higher oxygen contents are readily available at a fraction of the cost of low-oxygen spherical titanium powders. Additionally, the use of HDH powders can aid mechanical interlocking and therefore helps retain part shape after thermal de-binding. As such, much effort has been made to develop viable MIM HDH-Ti processes [4,9,15–20]. However, the low flow-ability, low packing density, uneven shrinkage during sintering, and high oxygen level of the non-spherical HDH Ti powders have limited their application in the MIM process. As a result, successful MIM of HDH Ti would require the use of a proper binder system, and optimum injection moulding, de-binding, and sintering parameters. While, as yet such a MIM process has not been established in industrial scale, many research and development activities have proven the success of different strategies for application of irregular shaped Ti powders for MIM process. For instance, Park et al. [21] used a milling process to modify the irregular shape of HDH powders and reduce their sharpness to make them more spherical. Their process improved the flow-ability of HDH powders, resulting an improvement in the solid loading and the final properties of MIM products [21]. In another attempt, German [4] mixed a fraction of small HDH powder with large gas atomised powders and successfully manufactured parts using a feedstock with a high solid loading of up to 72% (typical solid loading for Ti-MIM is in the range 62–68% for spherical powders). Replacing the titanium hydride (TiH_2) with pure HDH Ti has also shown a great potential for successful application of irregular shape powders in MIM process. The advantage of using TiH_2 is the release of hydrogen during de-binding/sintering process which can prevent extra oxygen pickup by the final sintered part [9]. For instance, Carrenõ-Morelli et al. [19] confirmed the potential of TiH_2 powders in Ti-MIM by manufacturing components with excellent elongation and tensile strength.

One of the serious challenges for Ti-MIM is the lack of dimensional accuracy and distortion of final products. These issues are more challenging and difficult to control for components manufactured using MIM of irregular shape Ti powders. In a recent review publication, the current authors [7] addressed the most important factors affecting dimensional reproducibility, uneven shrinkage and distortion of MIM components. They declared that size, geometry and wall thickness of parts (component factors), size, shape and distribution of powders, binder systems, mixing processes and powder loading (feedstock factors) and injection moulding, de-binding and sintering parameters (processing factors) are the most important parameters responsible for dimensional accuracy of MIM components. Despite such complex issues for dimensional control of MIM components different research have shown that this issue is controllable by accurate selection of powders, binders, injection and sintering parameters.

Table 1
Chemical composition of HDH Ti powders used in this study.

Element	C	O	N	Fe	Cl	Mn	H	Ti
Wt%	≪0.008	0.56	≪0.036	≪0.017	≪ 0.03	≪0.01	≪0.03	bal

Considering all improvement on MIM process for Ti and its alloys, the successful manufacturing of different Ti alloys with adequate properties have been reported in recent years. These include but no limited to: commercially pure Ti [16,17,19,22], Ti-6Al-4V [23–27], Ti-10V-2Fe-3Al [28,29], Titanium aluminide [30–34], Ti-15V-3Cr-3Sn-3Al [35], Ti-24Nb-4Sn-8Zr, TiNi [36], Ti-Nb-Zr [37], Ti-Mo [38,39], Ti-Mn [40] and Ti-Nb [41–44]. In addition, the first standard specifications for application of Ti-MIM in manufacturing of surgical implants from Ti-6Al-4V (ASTM F2885) and un-alloyed Ti (ASTM F2989), has recently been published by ASTM [45,46].

This study aims to identify the suitable process parameters for MIM of HDH Ti with minimal distortion and maximum size accuracy. In that regard, samples were fabricated by MIM of HDH Ti powder and the resulting microstructure, density, shrinkage behaviour, and mechanical properties were characterised over a range of sintering conditions.

2. Experimental procedure

Table 1 lists the chemical composition of the HDH Ti powder used, which has an average particle size of 45 μm and purity of 99.0%, supplied by Chaoyang Jinda Titanium Co., Ltd, China. A simple binder system consisting of 61 wt% paraffin wax (PW), 36 wt% high density polyethylene (HDPE) and 3 wt% stearic Acid (SA) was selected. A dry mixture of the HDH Ti powder and binder was loaded into a twin screw extruder (Thermo Scientific EuroLab 16) that was preheated to 175 °C and extruded ten times to produce a uniform mixture. Such preheat temperature of 175 °C selected based on the minimum temperature that extruder can work smoothly without building up the pressure inside the barrel and mix the powder and polymer well together. Also, our extrusion exercise showed that after 10 times extrusion of the mixture, the pressure inside the extruder barrel is minimal indicating a well-mixed feedstock. To find the optimum solid loading, the experiments started with a low solid loading of 54% and the fraction of binder gradually increased to the point where either of extrusion or injection moulding processes started to straggle by increasing more binder. The highest solid loading with smooth extrusion and injection moulding processes were obtained at around 61%. Then a mixture of 61 Vol% Ti powder and 39 Vol% binder has been selected for MIM in rest of the research.

The mixture was cooled to room temperature and manually crushed into particles of ≪3.0 mm to form the feedstock for the MIM process. Two different specimen geometries were moulded. Standard tensile samples manufactured using a Babyplast 610P injection moulder at maximum injection pressure of 85 MPa, nozzle temperature of 155 °C and cooling time of 15 s. Also, sub-sized tensile samples using a small hot press system to simulate the MIM process. In this setup, metallic die with tensile shape cavity was heated to 150 °C, feedstock placed in die cavity and pressed to 85 MPa and hold under pressure for 15 s. Then, the sample took out from die and cooled to the room temperature.

After moulding, samples were immersed in a hexane bath at 50 °C for 20 h to remove the paraffin wax component of the binder system. Thermal de-binding was performed by slow heating of samples to 550 °C under argon at a flow rate of 3l/min. Sintering was carried out in a vacuum of 10^{-3} Pa at a variety of temperature-time combinations. Fig. 1 schematically shows the detailed experimental