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Warm die compaction and sintering of titanium and titanium alloy powders

S.D. Luo, Y.F. Yang, G.B. Schaffer, M. Qian*

The University of Queensland, School of Mechanical and Mining Engineering, ARC Centre of Excellence for Design in Light Metals, Brisbane, QLD 4072, Australia

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ABSTRACT

A study has been made of the effect of non-lubricated warm die (200 °C) compaction on the densification of hydride–dehydride (HDH) Ti powder, pre-alloyed (PA) Ti-6Al-4V and Ti-10V-2Fe-3Al powders, and HDH Ti and V-Fe-Al master alloy powder blends, compared to cold die compaction. Depending on the compaction pressure, which was varied from 200 to 1000 MPa, non-lubricated warm die (200 °C) compaction was very effective for −100 mesh HDH Ti powder, increasing the green density by 5.0–9.4% theoretical density (TD). Die wall lubrication with stearic acid showed no influence on the green density when compacted at 800 MPa. With warm die (200 °C) compaction, achieving a green density of greater than 90%TD was straightforward for HDH Ti powder when compacted at ≥750 MPa. Accordingly, near pore-free (≥99.5%TD) Ti microstructures were obtained after sintering at 1300 °C for 120 min in vacuum when compacted at 1000 MPa. The resulting increment in the sintered density was between 2.0%TD and 4.4%TD. Warm die (200 °C) compaction showed no effect on PA Ti-10V-2Fe-3Al powder and only a small effect on PA Ti-6Al-4V powder when compacted at 1000 MPa. However, it was still virtually effective for Ti-10V-2Fe-3Al powder blends made of HDH Ti powder and V-Fe-Al master alloy powder. The observations were compared with literature data and discussed in accordance with the yield strength of Ti, Ti-6Al-4V, Ti-10V-2Fe-3Al and Al_3V as a function of temperature.

1. Introduction

To reduce the cost, which arises from feedstock and manufacturing process, has been the main thrust for the research and development of powder metallurgy (PM) titanium (Ti) in past decades (Peter et al., 2012). Qian et al. (2012) assumed that cold compaction and pressureless sintering in combination with the use of inexpensive Ti powder such as sponge fines and hydride–dehydride (HDH) powders is the most cost-effective approach. However, this approach has difficulty in fully densifying the powder compacts by solid state sintering. Robertson and Schaffer (2010) pointed out that obtaining a sintered density in excess of 95% theoretical density (TD) is realistic only with the use of high-purity fine Ti powder (<45 μm). Qian (2010) came to a similar conclusion in a comprehensive review. The cost of Ti powder is known to increase dramatically with the decrease of its impurity and/or particle size. Alternatively, increasing green density is proven to be beneficial to sintering densification.

Warm compaction, including warm die and/or warm powder compaction, is an established technique to improve metal powder compressibility in the ferrous PM industry (Bocchini, 1999). This is a single-press and single-sinter technique with a cost only 25% higher than that of cold compaction (Xiao et al., 2003). In principle, the increased green density from warm compaction is attributed to the reduced compressive yield strength of metal powders together with the improved redistribution of internal lubricants/binders (Hanejko, 1998). High purity Ti is as soft as annealed iron (Qian, 2010) but interstitial impurities significantly strengthen unalloyed Ti. To improve the fabrication of PM Ti parts, several studies have been reported on the warm compaction of the HDH Ti powder, Ti-6Al-4V and Ti-6Al-4V-6TiB_2 powder blends made from HDH Ti powder and master alloy powder, and pre-alloyed (PA) Ti-6.8Mo-4.5Al-1.5Fe powder. Table 1 lists these efforts with detailed processing conditions and major observations available from the literature. In conjunction with the use of fine Ti powder or high compaction pressure, the use of warm compaction facilitated the fabrication of nearly fully dense PM Ti materials by pressureless sintering leading to largely improved mechanical properties including the fatigue performance (Kondoh et al., 2004).

* Corresponding author. Tel.: +61 73360771.
E-mail address: ma.qian@uq.edu.au (M. Qian).

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Table 1
Warm compaction of Ti and Ti alloy powders from the literature.

Powder	Compaction temperature (°C)	Compaction pressure (MPa)	Lubricant and lubrication	Major outcomes with warm compaction, compared with cold compaction	Ref.
HDH Ti Ti + Al_3V Ti + Al_3V + TiB_2	150 for die set and powder but separately	392–1568	High fatty acid-based lubricant to inner die wall	● Green density of Ti (D_{50} = 42 μm)[a] increased by 6.3% at 588 MPa. ● Sintered Ti reached 98.7%TD at 980 MPa. ● Sintered Ti-6Al-4V reached 99.3%TD at 784 MPa. ● Sintered Ti-6Al-4V-6 TiB_2 reached 99.2%TD at 1568 MPa.	Kondoh et al. (2004)
HDH Ti Ti + Al_3V	150 for die set and powder but separately	196–1960	Lithium stearate to inner die wall	● Ejection force decreased substantially. ● Green density of Ti increased significantly. ● Sintered Ti (D_{50} = 22.1 μm) ≥99%TD at ≥392 MPa. ● Sintered Ti (D_{50} = 97.4 μm) ≥99%TD at ≥1176 MPa. ● Sintered Ti-6Al-4V reached 99.9%TD at 1568 MPa.	Takamiya et al. (2004)
PA & HDH: Ti-6.8Mo-4.5Al-1.5Fe	150 for die set, 80–180 for powder	500	Not provided	● Green density peaked at 140 °C. ● Sintered density reached 97.41%TD, 1.73% higher.	He et al. (2005)
HDH Ti	80–140 for die set and powder	350–600	Lithium stearate mixed to powder	● Green density increased with compaction temperature. ● Low and stable springback.	Simchi and Veltl (2006)
HDH Ti	140 for die set, 80–180 for powder	400–600	Macromolecule polymer to inner die wall	● Green density peaked at 140 °C. ● Green density increased by 4.7–4.9%.	He (2011)
HDH Ti PA & HDH: Ti-6Al-4V	25–300 for die set and powder	630 for Ti 840 for Ti-6Al-4V	Not provided	● Green density of Ti powder compact reached 90.3%TD at 250 °C, 15.3% higher. ● Green density of Ti-6Al-4V powder compact reached 77.3%TD at 300 °C, 2.5% higher.	Jia et al. (2011)

[a] D_{50}: average particle size.

Lubrication and powder preheating were both employed in these studies. The application of an organic lubricant to the die wall (external) or powder blend (internal) has some unique advantages. For instance, it can effectively reduce the ejection force and eliminate galling when the resulting high-density green compacts are ejected from the die. This not only ensures a long working life of the die but also the dimensional accuracy and consistency of the green compacts. However, lubrication can be problematic for PM Ti because it increases the contamination of carbon, oxygen and nitrogen impurities which are detrimental to the ductility and fatigue properties of as-sintered Ti components. Also, lubrication increases the cost of production as it requires an extra step of degassing or dewaxing prior to isothermal sintering. Preheating powder to the die wall temperature can ensure consistent compaction but similarly adds processing cost.

This study presents a systematic study of non-lubricated warm die (200 °C) compaction of HDH Ti powder, PA Ti-6Al-4V and Ti-10V-2Fe-3Al powders, and HDH Ti and V-Fe-Al master alloy powder blends and their subsequent responses to isothermal sintering, compared to cold die (room temperature) compaction. The selection of 200 °C of die temperature is based on the following considerations: titanium will soften substantially at 200 °C (50% reduction in the compressive yield strength) while being still resistant to oxidation. In addition, the die set can be manually handled with ease at 200 °C. First, warm die (200 °C) compaction of inexpensive −100 mesh HDH Ti powder over the pressure range from 200 to 1000 MPa was studied and the influence on subsequent sintering at 1300 °C was investigated. Then the effect of introducing V-Fe-Al master alloy powder on the warm die compaction and sintering of Ti-10V-2Fe-3Al was assessed. Last, warm die (200 °C) compaction of PA Ti-6Al-4V and Ti-10V-2Fe-3Al powders at 1000 MPa was studied.

2. Materials and methods

The starting powder materials used included HDH Ti powder (<150 μm, 99.4%Ti, 0.2%O, Kimet Special Metal Powder Co.