Advanced Processes for Titanium Sintering

Evan Schumann

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School of Mechanical Engineering

Faculty of Engineering, Computer and Mathematical Sciences

The University of Adelaide

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Symbols and Abbreviations

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BE Blended Elemental

BCC Body Centred Cubic

CIP Cold Isostatic Press

CHIP Cold Isostatic Press followed by Hot Isostatic Press

CP Cold Press or Commercially Pure

DSC Differential Scanning Calorimetry

EDS Energy Dispersive Spectroscopy

FCC Face Centred Cubic

FCT Face Centred Tetragonal

FWHM Full Width at Half Maximum

GA Gas Atomisation

HDH Hydride De-Hydride

HIP Hot Isostatic Press

HP Hot (Uniaxial) Press

HV Vickers Hardness Number

ICP-OES Inductively Coupled Plasma - Optical Emission Spectroscopy

LPS Liquid Phase Sintering

MA Mechanically Alloyed

PA Pre-Alloyed

PM Powder Metallurgy

REP Rotating Electrode Process

SE Secondary Electrons

SEM Scanning Electron Microscopy

XRD X-Ray Diffraction

List of Symbols

$\AA$ Angstrom (10^{-10}m)

E Young's Modulus (MPa)

ρ Density (g/cm^3)

σ_f Flexural stress (MPa)

ϵ_f Flexural strain (mm/mm)

$at\%$ Atomic percent

$wt\%$ Weight percent

$m\%$ Mass percent

$V\%$ Volume percent

$\%el$ Percent elongation

$^{\circ}C$ Degrees Celsius

Abstract

A global objective of current research is to reduce the cost of manufacturing of titanium parts by improving the efficiency of near net-shape powder metallurgy (PM) technologies. These technologies are considered to be very promising as they eliminate waste and high machining costs. However, the cost of titanium components fabricated with PM remains relatively high due to the significant rate of energy consumption needed for various stages of PM, such as powder processing and sintering. Therefore, more research is needed to reduce the cost of production further, without compromising the mechanical properties and quality of the final product.

The current research is focused on the two latest developments addressing the efficiency problems of current PM: (I) the use of hydrogen as a temporary alloying element in the production of titanium powder, and (II) the application of the Liquid Phase Sintering (LPS) method to enhance the densification of materials. The following aspects of these developments are studied in this thesis: the effect of powder characteristics obtained with the ball milling method and the influence of sintering parameters on the microstructure and mechanical properties of fabricated samples.

The experimental approach includes the following stages: (a) synthesis of TiH_2 from a commercial titanium sponge; (b) particle size reduction through ball milling; and (c) hot press sintering with and without adding a liquid aluminium

phase. The TiH_2 powders were investigated for particle size and morphology by laser granulometry and Scanning Electron Microscopy (SEM), and the dehydrogenation kinetic was studied using Differential Scanning Calorimetry, The metallic impurities introduced during ball milling were measured through Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES). Sintered specimens were characterised by density using the Archimedes immersion method, and the microstructure and phase composition were examined using SEM Energy Dispersion Spectroscopy (EDS) and X-Ray diffraction (XRD). The hardness of pure titanium specimens was tested by microindentation and the flexural strength of selected LPS specimens was determined using the 3-point bending test. A relationship between the ball milling time and TiH_2 particle size alongside the level of contamination of the Ti powder were established. The influence of the particle size and sintering temperatures, specifically in the bottom range concerned with the energy efficiency, on the densification and dehydrogenation of TiH_2 was studied. The effect of an aluminium phase on the minimum sintering temperatures and quality of the fabricated samples was investigated by varying the concentration of liquid aluminium during hot press sintering.

The outcomes of the current research demonstrated that:

- the size of TiH_2 powder after ball milling greatly increases the density and dehydrogenation of the sintered specimens;
- the dehydrogenation is seen to be delayed by pressure assisted sintering inside a graphite mould;
- ball milling leads to the increased pickup of oxygen on the surface of fine TiH_2 due to the increased specific surface area;
- The aluminium liquid phase is shown to improve the density during pressure assisted sintering at concentrations of 5 to 10 at% aluminium;

- the use of fine particle sizes leads to a faster reaction between the liquid aluminium and titanium and promotes a solid intermetallic phase formation around the aluminium particle site;
- one interesting outcome of the completed research is that the use of a liquid aluminium phase to sinter titanium is shown to improve part density when using pressure-assisted sintering, when compared with previous studies using free sintering.

Overall, it is believed that the conducted study contributes to the understanding and further improvement of PM techniques and demonstrates a significant potential to reduce the fabrication costs of titanium components with ball milling and direct sintering TiH_2 methods. However, a further optimisation of the fabrication parameters and a more comprehensive assessment of mechanical properties are required in order to verify the quality of the fabricated components and for industry to adopt these methods.

Publications

Journal Papers

Schumann E., Silvain J.-F., Bobet J.-L., Bardet M., Lu Y., Kotousov A. and Lamirand-Majimel M. Advanced Processes for Titanium Sintering, *Materials Chemistry and Physics*, under review.

Conference Papers

Schumann E., Silvain J.-F., Bobet J.-L., Kotousov A. and Lamirand-Majimel M. Titanium Enhanced Sintering Through Liquid Phase Sintering, *International Conference on Composite Materials*, ICCM19, Montreal, Canada, July 28 - August 2, 2013. See Appendix A.

Declaration

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