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Article

Degradation of Titanium Sintered with Magnesium: Effect of Hydrogen Uptake

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1. Materials and Methods

1.1. Preparation of Feedstock for Ti-6Al-4V and Mg-0.6Ca Powder

Ti-6Al-4V part The Ti-6Al-4V alloy powder Grade 5 (AP&C in Quebec, Canada) was produced by plasma atomisation. The used powder fraction had a particle diameter <45 µm. The powder particles are spherically shaped with a smooth surface, which is beneficial for the injection moulding process. The entire powder handling including feedstock preparation was performed in a glove box under argon atmosphere to prevent the fine powder from further oxidation. The used binder is a mixture of ethylene vinyl acetate (EVA), paraffin wax (PW) and stearic acid (SA). The feedstock, which contains 10 wt.% of this binder was prepared by mixing Ti-6Al-4V alloy powder and binder in a double Z-blade mixer (Femix KM 0.5 K, Linden, Marienheide, Germany) for two hours at a temperature of 120 °C. After solidification, the feedstock was granulated by means of a cutting mill.

1.2. Mg-0.6wt.%Ca Part

The procedure of Mg feedstock preparation has been described in detail in [1]. 85.4 wt.% of pure spherical and commercially available gas atomized Mg-powder (of particle size <45 µm, SFM, Martigny, Switzerland) and 14.6 wt.% of spherical gas atomized Mg-5Ca powder (of particle size between <45 µm, Zentrum für Funktionswerkstoffe GmbH , Clausthal, Germany) were mixed to prepare Mg-0.6Ca (all compositions in wt.%) powder blends. Below we will refer to this initial mixture as Mg-0.6Ca. Paraffin wax components, stearic acid and polypropylene components were used to prepare the feedstock for the injection moulding process. The powder loading was 77.3 wt.% (64 vol.%). To avoid any oxygen uptake of the magnesium powder components, the materials were handled under protective argon atmosphere in a glovebox system. Powder and binder were blended, preheated, filled into a sealable can under argon and finally mixed using a planetary mixer (ARE-250, Thinky Corporation, Tokyo, Japan). Analogous to the Ti-feedstock the Mg-feedstock was granulated after cooling.

1.3. Metal Injection Moulding (MIM)

An industrial injection moulding machine (320S Allrounder, Arburg, Germany) has been used to prepare dog-bone-shape tensile test specimens complying with ISO 2740-B. An injection pressure of 800 bar, an injection rate of 35 cm^3/s, an injection temperature of 112 °C and a mould temperature of 43 °C turned out to be adequate parameters for the injection moulding process.

The fabrication of the Mg-0.6Ca cover of the middle part of the titanium dog-bone has been performed by means of a Babyplast injection moulding device (Babyplast 6/12, RAMBALDI + CO.I.T. s.r.l. Molteno, Italy). A special mould, which was mounted on a titanium dog-bone, has been prepared and Mg-0.6Ca feedstock has been injection moulded.

1.4. Debinding and Sintering

The binder was removed in two debinding steps: solvent debinding in which wax and stearic acid are removed and thermal debinding in which the polymeric backbone of the binder system is removed by thermal decomposition. Solvent debinding was carried out at 40 °C for duration of 15 hours with hexane as solvent in a LÖMI EBA 50 (LÖMI, Grossostheim, Germany) debinding facility. In order to avoid oxygen contamination the parts were stored in a glove box under controlled Ar-atmosphere.

Thermal debinding and sintering of Ti parts were done using furnace (Nabertherm, Lilienthal, Germany). The thermal debinding took place under a slight argon flow of 70 l/h at 12 mbar in a temperature range between 250 and 400 °C. The specimens were sintered at 1300 °C; the dwell time was two hours under vacuum atmosphere (10^{-5} mbar) with heating and cooling both at 5 K/min [2].

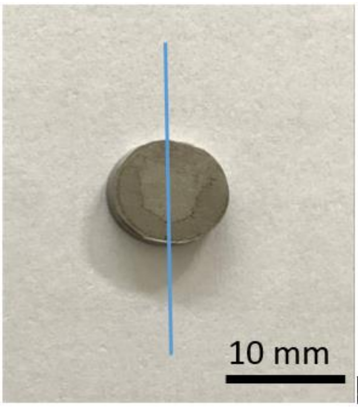
1.5. Mg-0.6Ca/Ti-6Al-4V Couple

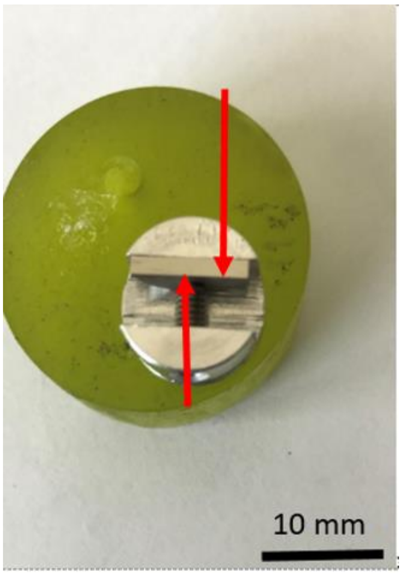
An as-sintered Ti part was placed inside of the mould and the MIM procedure was followed with the Mg-0.6Ca feedstock. The Mg-feedstock flows around the Ti-part. The composite obtained was then solvent debound and sintered at 640 °C for 8h at 1.05 bar of Ar 6.0 using a Mg grit gettered crucible setup in the furnace (MUT, RRO 350-900, MUT Advanced Heating, Jena, Germany). The sintering regime contains several flooding and evacuating cycles as described in [1].

2. Characterization

2.1. Imaging

A low-magnification overview of the Mg-0.9Ca/Ti interface (Figure 1) was obtained using optical microscopy (PMG3, Olympus, Tokyo, Japan). Smaller structures (Figure 1) were visualized via scanning electron microscopy (VEGA3 TESCAN-15kV, TESCAN, Kohoutovice, Czech Republic). Samples were cut, embedded, ground and polished before microstructural analysis.





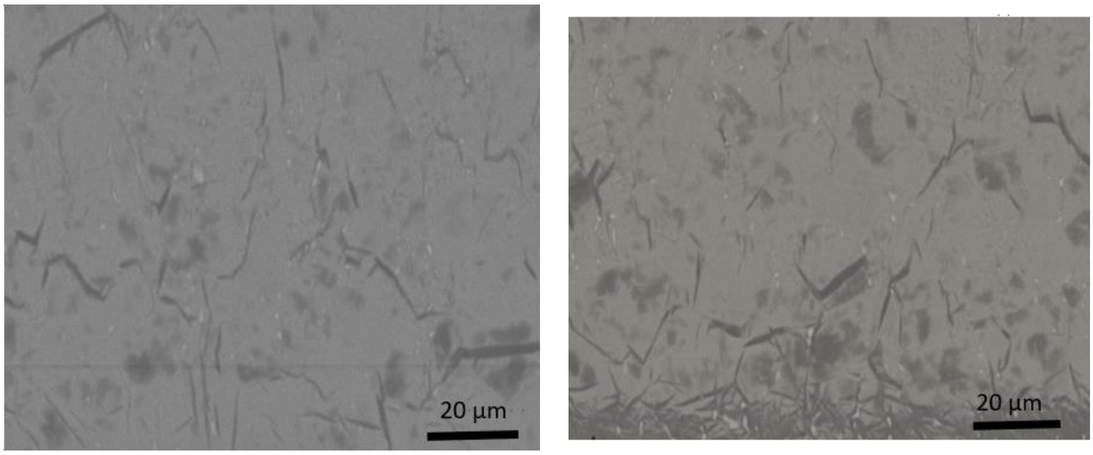


Figure 1. Photos of titanium plate (**upper row**) with cut line, fixation geometry and SEM micrographs (**down row**) in the centre (**left**) and at the border (**right**) of discs. Dark region in the border can be connected with location of H inside of the Ti plate.

2.2. GIXR

Structure analysis has been performed by grazing incidence X-ray diffraction grazing incidence X-ray diffraction (GIXRD) using Bruker D8 Advance X-ray diffractometer (Karlsruhe, Germany). The measurements were performed using Cu Kα radiation in the range of 2 theta from 20 to 80° (exposure time 10 s, step 0.02°) under 3° incident angle (thin film regime, grazing incidence X-ray diffraction, GIXRD). No rotation of specimen was used due to the possible substrate texturing. Samples have been measured in two direction 0° and 90° which show an effect of texture but the same set of diffraction peaks.

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