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Original Research Paper

Sintering behavior and microstructure evolution in cp-titanium processed by spark plasma sintering

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ABSTRACT

This paper investigates the effect of various sintering temperatures from 800 to 1500 °C on the microstructure evolution of cp-titanium processed by Spark Plasma Sintering (SPS). The material processing conditions under consideration may change the obtained atomic order and microstructure. The different relation of the mechanical and corrosion resistance properties observed in the analyzed results could at the same time be shaped in the expected range. The SPS procedure allows the obtainment of nearly theoretical densities of the compacts and excellent mechanical properties (UTS = 892 MPa, CS = 1442 MPa). The results confirm that the contact angle measurement could support the process control, particularly if a microstructure feature is considered.

1. Introduction

Despite growing interests in the newly introduced technological approach of material processing methods, it still requires basic research to confirm the fundamental relations and fulfill the standards of reproducibility, particularly in terms of economic profitability. A different approach that combines pressure and heat control applied in material processing [1,2] increasingly focuses on the starting materials. A practical possibility for tremendous savings in the processing costs that may reach nearly half of the product value [2], could be obtained for the near net shape approach. The processing paths remain as the best guide for the efforts aiming at validating or optimizing the processing conditions that lead directly to the enhancement of the properties. Therefore, the microstructure, as the key material feature, could be modeled by a composition or processing conditions including atmosphere, pressure, temperature and acting force. A change in the properties can be obtained through an introduction of slight differences in the processing ranges whose choice becomes crucial when cost calculation and selected method advantages are considered [3]. Titanium (Ti) and its alloys are some of the most commonly used metals in aerospace, petrochemical or naval applications [4]. Their excellent physical and mechanical properties, superior biocompatibility, the lowest (of metallic biomaterials) elastic modulus, low density and high corrosion resistance compared to those of stainless and cobalt-based alloys favor their increasing applicability especially in medicine. High titanium reactivity with the interstitial elements from the crucible, atmosphere or tool material contact makes it processing challenging especially when the additional but uneconomical preparation steps are needed. From the various processing methods of titanium, SPS is believed to be one of the most effective and considering other advantages also low-cost (when machining or turning operation could be minimized) and easy to scale up approach [2]. To confirm the above, it needs to be mentioned that the spark plasma method utilizes pressurized impact of the plasma from the high energy pulse of current that locally melts and breaks down the oxidized layer [5,6] from the contacted particle surfaces, thus, enhancing the mass transport [7,8]. Fast processing [9] and pressurized impact of the plasma, which can effectively reduce the grain growth effect [10] during sintering, also possible to get by other methods [11,12], remain the key and best recommendation for fully controllable and well suitable technology for "hard-to-sinter" or "challenging" materials processing. Typical of powder metallurgy, the compaction and the sintering step could be realized in a single operation in the case of the field-assisted method. Different works focusing on the SPS processing, investigate the influence of the starting material purity [13], size [14] sintering temperature, holding time, pressure and heating rates [15,16] on the final bulk properties. Despite the efforts made to date, more work is still needed to fully optimize the material procedures, especially in terms of

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control of its processing. The SPS method is beneficial in the prevention of grain growth and synthesis of nanostructured alloys [17,18]. In this paper, the effect of various sintering temperatures on the microstructure evolution of cp-titanium processed by Spark Plasma Sintering and its influence on the physical and mechanical properties were investigated.

2. Experimental procedure

2.1. Sample preparation

Ti grade 2 powder (Alfa Aesar, Germany) with an average particle size of 325 mesh and 99.5% purity was used as the starting material. For the compact preparation, Ti powder was placed into a graphite tool and then densified using an HP D 25-3 spark plasma sintering system (FCT System GmbH, Germany). Cylindrical specimens with dimensions of 40 mm in diameter and 10 mm high were fabricated.

The specimens were heated up to various temperatures in the range 800–1500, according to processing graph depictured in Fig. 1. The temperature was monitored by an optical pyrometer through a non-through hole in the graphite upper punch. For analyzed in this work microstructure evolution, reference Ti-G2 rod, with a diameter of 8 mm obtained by VAR (Stupino Titanium Company) was used in the annealed state.

2.2. Material characterization

The structural analysis of the raw materials and the obtained body sinters was investigated by X-ray diffraction (XRD) with Cu Kα radiation – 1.54 Å (Panalytical Empyrean – Netherlands). The measurement conditions for all collected data were set up at 20–80° with a step size of 0.017°/15 s and 45 kV/40 mA parameters. The plot background for the peak intensity measurements was determined by automatic correction using HighScore software with a Sonneveld-Visser algorithm and granularity factor of 26. The collected data were processed to obtain the relative intensity ratio of the planes that occurred in the examined 2 theta range. The lattice parameter estimation was based on the Rietveld profile fitting method realized on the HighScore Plus software. Apply approach involve the simulation of the diffraction pattern based on the analyzed structural model (Ref. 01-89-2959). The calculated pattern of the model structure was fitted to the observed one by minimization of the sum of the squares and after refinement achieve high goodness of fit ($\chi^2 < 3$). The density of the samples was determined by three measurements based on the Archimedes Law of the sample immersion in water. The measured density values were eventually compared with the theoretical density; the percentage value of the relative density (RD) was determined with a standard deviation. For the surface wettability measurements, contact angles (CA) were determined by the Musial software (Elektronika Jadrowa, Poland) from the geometrical shape of the droplets recorded by the optical system with a digital camera. The placement of the water droplets was realized by a special micropipette system with a constant volume of the test liquid (2 μL) on clean, polished and alcohol-rinsed surface. The data were obtained after 10 s of keeping the droplet on the surface under ambient conditions. The procedure was repeated three times. The possible measuring errors for θ might have been caused by possible surface interactions, different shapes of the liquid droplets placed on the investigated surfaces as well as by different liquid vaporization rates and inaccuracy in the graphical interpretation of the images by the computer software. For each recorded image, related to a single drop, the geometrical shape analysis was repeated 10 times. The extreme values were rejected and the arithmetic mean value was calculated for the accepted findings. The microstructural characteristics of the obtained Ti-compacts were studied using the Olympus GX51 light microscope (Olympus, Japan). Standard metallographic preparation was used for sample observation; hot-mounting in conductive bakelite followed by grinding and polishing using silicon carbide papers and silica suspension 0.05 μm and finally etching with the Kroll's reagent for 10 s were performed to show microstructural details. For the corrosion measurements, the potentiodynamic method was applied with the Solartron 1285 potentiostat (Solatron analytical, UK). The corrosion resistance of different samples was measured in the Ringer's solution applying the potentiodynamic mode in range of −3 to 3 V, with the scan rate of 0.5 mV/s at a temperature of (37 ± 1) °C controlled by a thermostat. The corrosion potentials (*Ecorr*) and the corrosion current densities (*Icorr*) were estimated from the Tafel extrapolations of the corrosion curves, using the CorrView software. The hardness measurements were carried out at the polished surface of the samples by the Vickers diamond pyramid FM-800 (Future-Tech, Japan) with the applied load of 1.961 N for 15 s with 10 indents per sample to get the average value of hardness. The tensile strength was measured using the MT5000HC micro tester (Gatan, UK) with the measuring range of up to 5 kN (constant traverse speed) and initial strain rate of 0.0028 s^{-1} on a sample illustrated in Fig. 2.

The compressive strength was measured without lubricant using the 4483 Instron mechanical testing machine of the measuring range of up to 150 kN (constant crosshead speed) with the initial strain rate of 0.0033 s^{-1} for the obtained Ti-compacts and 0.0042 s^{-1} for the Ti-rod samples. For the tests, the samples of the dimensions of Ø10 × 10 mm for the Ti-compacts and Ø8 × 8

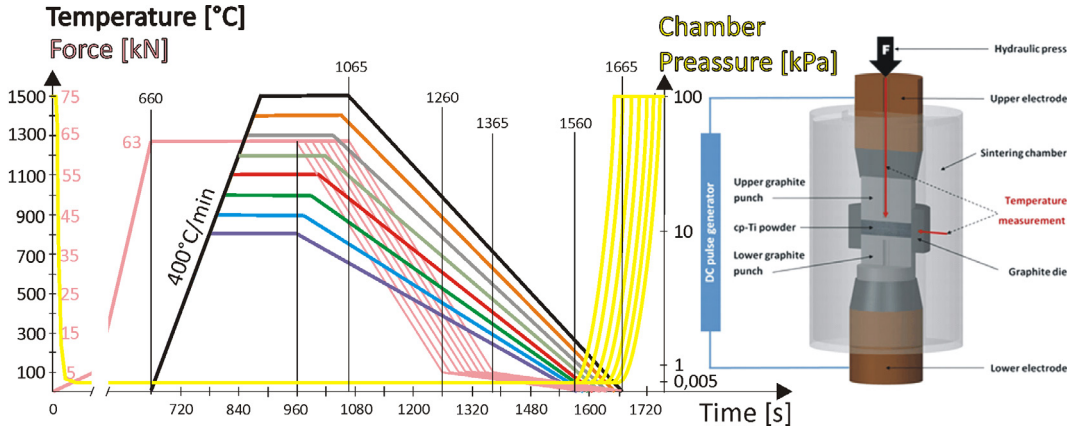

Fig. 1. Process parameter graph with SPS furnace layout for HP D25-3 system infrastructure.

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