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Influence of porosity on mechanical behaviour and gas permeability of Ti compacts prepared by slip casting

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ABSTRACT

Porous titanium compacts with porosity in the range of 12.3–35.3 vol% were fabricated by slip casting. Slip casting is emerging to be an attractive process for fabrication of porous titanium products. The mechanical properties, fracture morphology, gas permeability, pore size analysis and pore shape factors for the porous Ti compacts were determined for different porosity levels. A decreasing porosity level resulted in less open porosity and gas permeability, reduced pore size, and an increased tensile stress and elongation. The mechanical properties of porous titanium compacts produced by slip casting were comparable with more conventional press and sintered materials at the same porosity levels. Theoretical models for tensile stress and ductility as a function of porosity were examined and incorporated into the results and differences between the theoretical models and experimental results are discussed. The pore shape factor analysis showed that tensile loading would stretch the pores in the compacts to produce more irregular pores, which were acting as linkage sites to allow the propagation of cracks. Additionally, a novel interconnected pore characterisation method using ammonium meta-tungstate solution is presented. By using backscatter scanning electron microscopy, the interconnected pores can be directly observed.

1. Introduction

Porous titanium (Ti) is an excellent filter material used in the chemical and processing industries, owing to its outstanding corrosion resistance, high strength-to-weight ratio, large specific surface area and high gas and liquid permeability [1]. A stable and permanent thin layer of TiO_2 forms on the surface of titanium at room temperature, and it is able to regenerate immediately after damage. This thin film of TiO_2 allows Ti to be used in highly corrosive environments and aggressive media in which stainless steel cannot provide sufficient corrosion resistance, such as seawater, oxidising chlorine, salt acidic solutions containing oxidants, etc. [2,3]. For ceramic filters, fragility is a main issue in practice and thermal shock is another potential problem which limits their use when there are frequent and rapid changes in process gas temperature [4]. Typical uses of porous Ti filters can be found in air and water purification processes [5,6], filtering of titanium tetrachloride liquid [7] and in pulp and paper plants [2].

Ti has a strong chemical affinity to oxygen, especially at temperatures above 400 °C, followed by fast diffusion of oxygen into the matrix, which results in embrittlement [8]. Because of the large surface area of porous Ti and therefore a high propensity to oxygen contamination, a low temperature powder metallurgy route is a promising way to fabricate porous Ti products.

In powder metallurgy, the sintered density, porosity and mechanical properties of Ti compacts can be affected by sintering conditions, powder particle size and shape, alloying elements, interstitial elements, etc. Oh et al. [9] fabricated porous Ti with a porosity ranging from 5.0 to 37.1 vol% by sintering spherical titanium powder particles. Their work showed that the initial powder size and sintering pressure are two dominant factors in controlling the densification process of porous Ti compacts, whereas sintering temperature has only a small effect. A sintering study examined the effect of sintering atmospheres on the density and mechanical properties of sintered Ti compacts, revealing that at similar levels of the sintered density, the Ti compacts sintered in argon had much lower tensile properties than those sintered under vacuum [10]. Recent research has shown that additions of alloying elements (Fe and Zr) to Ti powder could enhance the sintering densification of Ti compacts prepared by powder injection moulding, but the mechanical properties were not improved [11]. Moreover, Qian [12] has summarised the tensile properties of cold compacted and sintered Ti as a function of oxygen content. At a similar level of sintered density, an increase in oxygen content increases the strength of sintered Ti compacts, whereas the ductility reduces.

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The porous Ti compacts in this study were produced by a slip casting route, which, in a previous study was demonstrated to be a feasible process for manufacturing porous Ti products [13]. Ohkawa et al. studied Ti slip casting for dental applications using sodium alginate as a binder [14]. Due to the contamination from the decomposition of sodium alginate, a TiC phase was found in the sintered Ti compact. Thus, a brittle sintered Ti part was produced. In another Ti slip casting study, a slurry formed from a mixture of Ti and TiH_2 powder was used to make a Ti foam with a porosity ranging from 56.1 vol% to 65.2 vol% and compressive strengths ranging from 120 MPa to 150 MPa [15]. To the best of our knowledge, a comprehensive and quantitative understanding of the effect of porosity on the mechanical behaviour of Ti produced by slip casting has not yet been reported. In this study, the effect of porosity on gas permeability, pore size distribution, pore shape, degree of interconnected pores and mechanical behaviour of slip cast Ti has been examined. Moreover, theoretical models and experimental work from previous researchers have been compared with the current experimental results to enhance our understanding of the slip casting process.

2. Background

2.1. Tensile strength (TS) versus porosity relationship for sintered metals

Many theoretical models for the tensile behaviour of sintered metals have been derived by considering the micromechanisms of failure. Griffiths et al. [16] derived an analytical model of the TS of porous materials based on the ratio of load-bearing area and apparent cross-sectional area, as shown below:

$$\frac{\sigma_t}{\sigma_o} = \frac{A_b}{A_a} = 1 - \lambda P^{2/3} \quad (1)$$

where σ_t is the TS of the porous material, σ_o is the TS of the bulk material, A_a is the apparent cross-sectional area, A_b is the load-bearing area, λ is an empirical constant and P is the level of porosity in vol%. Griffith et al. suggested that the value of λ can be varied from 0.98 to 1.30, depending on the pore distribution and the interaction effect of the stress concentrations. With regard to the pore distribution, λ is equal to 0.98 for regular tetrahedrons and 1.077 for irregular tetrahedrons. However, when a porous material is subjected to a tensile stress, there is a local stress concentration induced around the pore cavity. To take into account the interaction effect of stress concentrations, the λ value would increase further.

In 1981, Fleck and Smith [17] developed a brick model, as shown below:

$$\frac{\sigma_t}{\sigma_o} = (1 - P^{2/3})^2 \quad (2)$$

This model simplified the Griffiths's model by treating the powder particles as solid cubes and pores as cubic spaces and assumed that there is no effect of the stress concentration on the pore geometry.

Haynes [18] also proposed a model by assuming no microstructural defects in the sintered metal, as shown below:

$$\frac{\sigma_t}{\sigma_o} = K(1 - P)^M \quad (3)$$

where K is a constant that depends on the test geometry and processing details and M is the exponential dependence on the level of porosity, which normally varies from 3 to 6. The value of M will be affected by the stress concentration and premature fracture that is produced by crack propagation from pore to pore. In this study, all the equations (1)–(3) have been compared with experimental data and the detailed results and discussion are presented in Section 4.

2.2. Ductility to porosity relationship for sintered metals

Haynes [19] proposed an empirical equation that related the porosity level with the ductility of sintered metals, as presented below:

$$\delta_{rel} = \frac{\delta}{\delta_o} = \frac{V_P}{V_s} = (1 - P)^{3/2}(1 + C\,P^2)^{-1/2} \quad (4)$$

where V_p is the notional branch volume of a porous material, V_s is the notional branch volume of a bulk material, δ_o is the ductility of the bulk material, δ_{rel} is the relative ductility of the porous material and C is a constant which measures the sensitivity of the ductility to porosity content. Haynes [19] defines the notional branch volume as the volume of pore-free material which will undergo the same total deformation as the porous specimen when strained to fracture. In his model, he assumed that powder particles are bonded together by necks to form a network in a sintered metal material. When the material is subjected to tensile stress, the individual necks in the material behave like miniature tensile specimens, whereas most of the deformation occurs at the narrower parts of the necks. Haynes suggested that the number of branches depends on the number of contacts between powder particles in the green state.

3. Experimental methods

3.1. Preparation methods

Porous Ti compacts with porosities in a range of 12.3–35.3 vol% were fabricated by slip casting (Table 1). Fine −325 mesh ($\leq 44\,\mu m$) titanium powder (purity: 99.95%; Xi'an Baode Powder Metallurgy Co. Ltd., China) produced by the hydride/dehydride (HDH) process were employed in this study, as shown in Fig. 1. Further details of the slip casting process for Ti have been reported elsewhere [13]. Each slip cast rectangular part (40 mm × 10 mm × 20 mm) was sintered under high vacuum (3×10^{-3} Pa). The porosity range was controlled using different sintering conditions such as sintering temperature (1000 °C,

Table 1
The total porosity, oxygen content and carbon content of porous Ti compacts prepared by slip casting and sintered under various conditions.

Sintering temperature (°C)	Sintering time (h)	Heating rate (°C/min)	Cooling rate (°C/min)	Total porosity (%)	Oxygen content (wt%)	Carbon content (wt%)
1000	1	10	Furnace cooling	35.3	–	–
	2	7	Furnace cooling	27.7	–	–
	2	10	Furnace cooling	30.6	0.45	0.051
1100	2	10	Furnace cooling	21.9	0.44	0.043
1200	0.5	10	Furnace cooling	16.4	–	–
	2	10	Furnace cooling	13.7	0.47	0.046
	2	7	5	12.3	–	–