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Original Research

Preparation and properties of low cost porous titanium by using rice husk as hold space☆

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A B S T R A C T

The present study demonstrates an effective low cost effective process for the production of porous titanium with 50–60 vol% porosity using 20 wt% rice husk (RH) with the hold spaces in the size ranges of 100–180, 180–250, 250–380, and 380–550 µm. The analysis of the samples revealed an interconnected pore microstructure consisting of a mixture of coarse channel pores, created during burnout of RH. The compressive strength of the developed samples was in the range of 17–70 MPa and depended strongly on their porosity and pore size. Large amounts of cleavage steps appeared on the brittle fracture surface after compression of the samples. The 3D morphology of porous titanium surface with rice husk sizes of 100–180 µm and 380–550 µm can be characterized by the micro-hole surface of pore size, and the size of the hole diameter and husk. The developed porous titanium is considered potentially useful in future medical or industrial application of biomass.

1. Introduction

China is an agricultural and rice producing country. The output of rice husk amounts to about 4×10^7 tons per year, but the comprehensive utilization rate is less than 10%. Most of rice husk is burned or abandoned, and only a small part is used as animal feed. This trend not only represents a serious waste of resources, but also causes environmental pollution [1]. Rice husk could instead be used as a cheap and abundant biomass source, having enormous potential in the field of renewable energy. The use of rice husk has been investigated as an additive for high performance concrete materials [2], leading to the production of high performance and low cost porous alumina and clay bricks [3]. The use of rice husk as hold space not only reduces waste in the grain industry but also increases the added value of sintered products. Rice husk can be classified in terms of the internal quality of the hold space formed as a consequence of its introduction in the material before the combustion of the organic component. The holes are formed contemporarily to the heat release by the reaction and have an important influence on the performance of the product. The larger the size of rice husk, the greater the hole formed during combustion and the greater the impact on the strength of the final material.

Porous materials have good thermal conductivity and excellent mechanical and biological properties and have therefore become the focus of research in recent years. They are employed in applications such as fluid filters, catalyst carriers, heat exchangers, thermal insulation, solid oxide fuel cells, combustion burners, and biomedical implant [4–10], etc., owing to their distinct properties such as low density, high porosity, high surface area, good thermal shock resistance and low thermal mass [11]. When preparing bioactive porous titanium materials, researchers usually rely on low melting point metals, such as Mg and Al [12,13], for the creation of hold space; additionally, high value materials, such as potassium chloride, sodium chloride, urea, and ammonium bicarbonate [14–17] are used as pore-forming agents. The use of these substances allows the control of the size of the hold space during the sintering process, which can be tailored to the desired porosity, pore structure, and elastic modulus of the final material [18,19], On the other hand, the use of low cost biomass derived materials, such as rice husk, for the preparation of porous titanium has not been reported in literatures. This paper studies the use of rice husk as hold space during the preparation of Ti porous material, describing both the preparation method and its influence on the overall performance. A theoretical basis for future medical or industrial application of biomass is also provided.

2. Experimental procedures

2.1. Processing and characterization of rice husk and titanium

The original rice husk(RH) used came from Hubei province. First,

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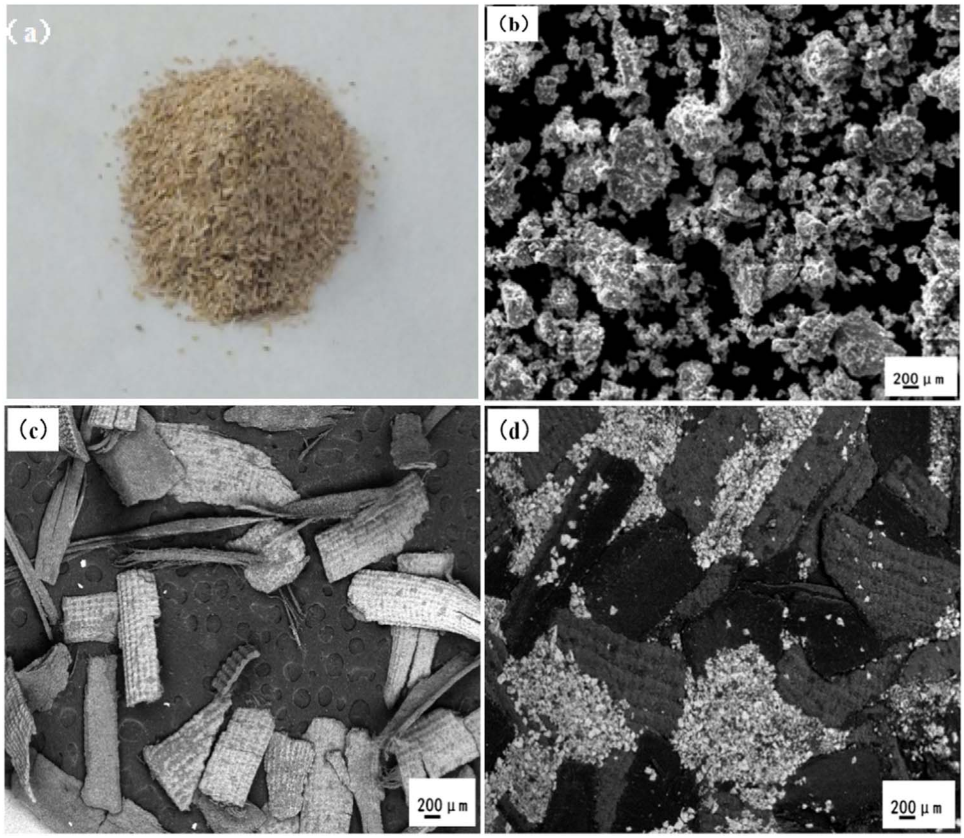

Fig. 1. Images of dried RHs powders (a) SEM images of Ti powder (b), RHs (c) and mixture powder(d).

the RH was washed for 30 min in a deionized water and citric acid solution(10 vol% concentration) to remove impurities and organic compounds. It was then dried in a stove at 60 °C for 4 h. The dried RHs can be seen in Fig. 1 (a) . Four standard test sieves with mesh sizes of 180, 250, 380 and 550 μm , respectively, were used to obtain the uniform sized samples. Accordingly, RH powders with sizes 100–180, 180–250, 250–380 and 380–550 μm were collected from each sieve. Each one of these powders was used as pore former.

The raw material was Ti powder having particle sized of 20 μm and purity of 99 wt%. The scanning electron microscope (SEM) picture of the titanium powder is shown in Fig. 1(b), and the chemical composition analysis is shown in Table 1. Fig. 1(c–d) shows the SEM images of the RHs and the mixture powder.

2.2. Preparation and characterization of porous titanium

To prepare the samples, RHs of one size group were initially mixed with the Ti powder, at a weight ratio of 1:3, in a specialized blender for three hours. The mixed powder(RH 20 wt%) was then placed in a homemade steel mold to prepare compacts having a size of 15×10 mm^2. The mold operated at a pressure of 400 MPa and the holding time was 120 s. After vacuum drying the sample at 2×10^{-2} Pa in the sintering furnace, the furnace was heated to 100 °C, at a rate of 5 °C/min, and insulated for half an hour to allow for the evaporation of the crystallization water. The vacuum sintering furnace was then further heated to 350 °C, at a rate of 5 °C/min, and insulated for half an hour to allow the pyrolysis of the rice husk. In the third heating step, the temperature was brought to 750 °C, at a rate of 10 °C/min, and maintained at this level for two hours to allow the complete carbonization of the RHs. Finally, the temperature was brought to 1300 °C, and the furnace was insulated for two hours, during which the thermal reaction between titanium and the carbonized RHs took place. The samples were then cooled to room temperature, at a rate of 10 °C/min. The resulting porous Ti composites served as test samples. The preparation process is shown schematically in Fig. 2.

Table 1
Content of pure Ti powder.

Elementary	Ti	O	Si	C	others
Content (wt%)	99.9	0.006	0.002	0.002	0.036

The porosity of sintered samples was determined by water immersion method based on Archimedes' principle. The characterization of the as-cast microstructures was performed by scanning electron microscopy (SEM) (MERLIN Compact, Japan) with energy dispersive spectrum (EDS). SEM images were taken of the cross section of the samples. The phase constituents of the porous samples were determined by X-ray diffraction (XRD) patterns exposed by an X-ray diffractometer (model 7000) with CuKa radiation and a Ni filter. The powder sample for the XRD was prepared by smearing a thin layer of powder on a glass plate. The glass plate had previously been coated with paraffin wax to allow for good adhesion. The XRD analysis was carried out at a voltage of 40 kV and 40 mA within diffraction angles ranging from 10°to 90° at a scanning speed of 8° min^{-1}. The 2q step was 0.04 and the step time was 40 s/step. The compressive test was carried out at a nominal strain rate of 10^{-3} s^{-1} using a universal testing machine (SHIMADZU AG-X, 100 kN, Japan) at room temperature while following the Chinese National Standard for compression testing (GB/T7314-2005) [20]. The porous titanium surface was determined from three-dimensional (3D)laser scanning microscope images. The tested cylindrical sample was 8 mm in diameter and 10 mm in height.