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# On the compressive behavior of sintered porous coppers with low-to-medium porosities—Part II: Preparation and microstructure

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#### **Abstract**

A polymer space-holder method was used in this study to prepare porous coppers with low-to-medium porosity within the range 5–50%. This provides the possibility to control the pore size, distribution and structure. Optical microscopy and scanning electron microscopy (SEM) with energy dispersion spectrum (EDS) were utilized to characterize the porous samples. Two different sizes of copper powders, 5 and 45 μm, were used to investigate the effect of raw materials powder size. Microstructure results have shown that there exist two different types of pore in the sintered samples: round-shaped macro-pores left over by the burnout of the space holder and irregular micro-pores or the intervals among metal powders. No matter which size powder was used, the size of the macro-pore falls into a range 200–500 μm, but the pore structures are different in the two cases, interconnected or open pores for the 45 μm raw powders and closed pore for the 5 μm powders. The sizes of the micro-pores among the copper powders in the two cases are also different, several microns for the 5 μm powders and 10–20 μm for the 45 μm powders, though all micro-pores are interconnected for both powder sizes. From the viewpoint of pore structure, it is concluded that the 45 μm powder is more appropriate for use to prepare the porous metal. In addition, the effect of the binder was also investigated. It is suggested that a binder that can be easily and completely removed should be used in order to induce the residue. This paper, as Part II of the publication, focuses on the fabrication of the porous samples where Part I [Lemons JE, editor. Quantitative characterization and performance of porous implants for hard tissue application. ASTM STP 953; 1985] has been published earlier for the mechanical properties of the material.

Keywords: Porous metal; Powder metallurgy; Microstructure; Mechanical strength

#### 1. Introduction

Due to their unique mechanical and physical properties, such as high stiffness in conjunction with low specific weight, high gas permeability and thermal conductivity, porous solids have been widely used as both functional and structural materials for a variety of purposes, such as filters, insulators and cushions. More recently, the porous metals have been proposed for the application of biomedical implant materials, such as porous titanium and tantalum [1–7]. The advantages of cellular metals have been explicitly described in some recent investigates and

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reviews [8,9]. Of particular interest are the mechanical properties and the porous structure that open-cellular forms offer for the adjustable elastic Young's moduli, the appropriate mechanical strengths and the regeneration capability of the new-bone tissues and the transport of body fluids within the pore structure.

Most of research and development on porous materials for structural and mechanical application have been restricted to highly porous materials with over 70% porosity, such as honeycombs and metallic forms for applications primarily as the core of sandwich panels. Many processings have been developed to prepare high porosity metallic foam [10]. In contrast, less is understood for porous metals within the low-to-medium porosity range (10–70%) [11,12], partially due to the lack of availability of materials under this category.

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Powder metallurgy has been shown to be a suitable method in porous metal preparation [13–15]. By varying the compacting pressure for a metal powder alone, a range of porosity could be achieved. The disadvantage of using pressure alone is the limited control over the size and distribution of the voids formed in the materials and the complete lack of control of the void shape with random and irregular shapes of voids, which are often with sharp corners and edges, leading to stress concentration and crack formations. A method in analogy to the powder metallurgy process has been applied to make porous metal using gas entrapment technique [16–18]. However, theoretical considerations show that no more than 50% porosity can be expected from this technique [19]. It was reported that porous body with 20-40% unconnected porosity was obtained and typical pore diameters range from 10 to 100 µm [16]. Another method has then been developed based on space-holding filler [20–22], in which the bulk of space-holding materials are filled with fine metal powders. The space holding will be removed by various ways and a pore will be left at the position of the space holder. Porosity up to 90-95% was observed [23].

In this paper, we reported our primary results on the preparation and microstructure of porous coppers with medium porosities. The main purpose was focused on preparation method to control the pore size, which also provides the possibility to control the pore distribution and structure (interconnected or closed) for further application of this kind of porous metal. An experimental study on the mechanical properties of the sintered samples was reported in Part I of the work [24].

#### 2. Experimental

### 2.1. Raw materials

In order to test the feasibility of the preparation method, and the microstructure and the constitution relationship, copper powders of average diameters 5 and  $45\,\mu m$  were obtained with the purity higher than 99%. The choice of the spacer-holder materials was based on its chemical and mechanical properties, such as the minimum strength to keep the shape during the preparing and pre-sintering

process. PMMA polymer is strong enough to maintain the shape during the compacting process. Spherical PMMA beads of average size 600 µm were chosen.

#### 2.2. Preparation of semi-finished samples

Fig. 1 shows schematically the processing steps for the preparation of the porous metals. Copper powders and space-holder PMMA beads were mixed first in a dry air condition. The method of mixing was critical to achieving a homogeneous distribution within the blend because of the different densities of copper and the space holder. A horizontal mixer was used. Mixed powders were then blended with 0.6% zinc stearate as a lubricant and 2.0% sodium silicate solution as a binder (except for the sample with 5% porosity for which no binder was added) and then compacted by uniaxial cold pressing with one of four different pressures: 125, 150, 200 and 300 MPa corresponding to the porosities required, leading to the products of green samples.

#### 2.3. Sintering processing

The green samples were subsequently heated under controlled temperatures to remove the polymer space holder and finally sintered in a box furnace in hydrogen environment to avoid oxidation. This is named as lowtemperature removing process/or removing process and high-temperature sintering process/or sintering process, respectively. The removing temperature was determined based on the thermogravity property of the space holder (PMMA), as shown in Fig. 2. The thermogravity measurement condition is as follows: heating rate of 5 °C/min in N<sub>2</sub> gas environment with gas flow of 50 ml/min. The decomposition of PMMA mainly occurred in the temperature range 260–400 °C. In the experiment, the moving temperature was set at  $360 \pm 10$  °C, corresponding to the highest decomposing rate of PMMA. According to a normal sintering procedure, the sintering temperature is normally 100 °C below the melting point of the metal powder. In this experiment, the sintering temperature was set at 960 °C. The final sintering processing was heated at 350–370 °C for 30 min and sintered at 960 °C for 60 min with both heating

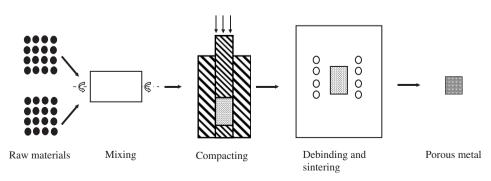


Fig. 1. Schematic illustration of the fabrication process for porous metal with low-to-medium porosity.

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