Materials Characterization 111 (2016) 21–30

Contents lists available at ScienceDirect

Materials Characterization

journal homepage: www.elsevier.com/locate/matchar

The distribution and mechanism of pore formation in copper foams fabricated by Lost Carbonate Sintering method

Mohammad Hosein Shahzeydi, Amir Masoud Parvanian, Masoud Panjepour *

Department of Materials Engineering, Isfahan University of Technology, 84156-83111 Isfahan, Iran

ARTICLE INFO

Article history:
Received 28 May 2015
Received in revised form 4 November 2015
Accepted 9 November 2015
Available online 11 November 2015

Keywords:
Copper foam
Powder metallurgy
LCS technique
X-ray computed tomography
Tension analysis
Thermal analysis

ABSTRACT

In this research, utilizing X-ray computed tomography (XCT), geometrical characterization, and pore formation mechanisms of highly porous copper foams manufactured by powder metallurgical (PM) process are investigated. Open-cell copper foams with porosity percentages of 60% and 80% and with a pore size within the range of 300–600 μm were manufactured by using potassium carbonate as a space holder agent via the Lost Carbonate Sintering (LCS) technique. XCT and SEM were also employed to investigate the three-dimensional structure of foams and to find the effect of the parameters of the space holders on the structural properties of copper foams. The result showed an excellent correlation between the structural properties of the foams including the size and shape of the pores, porosity percentage, volume percentage, particle size, and the shape of the sacrificial agent used. Also, the advanced image analysis of XCT images indicated fluctuations up to ±10% in porosity distribution across different cross-sections of the foams. Simultaneous thermal analysis (STA: DTA–TG) was also used to study the thermal history of the powders used during the manufacturing process of the foams. The results indicated that the melting and thermal decomposition of the potassium carbonate occurred simultaneously at 920 °C and created the porous structure of the foams. By combining the STA result with the result of the tension analysis of cell walls, the mechanisms of open-pore formation were suggested. In fact, most open pores in the samples were formed due to the direct contact of potassium carbonate particles with each other in green compact. Also, it was found that the thermal decomposition of potassium carbonate particles into gaseous CO_2 led to the production of gas pressure inside the closed pores, which eventually caused the creation of cracks on the cell walls and the opening of the pores in foam's structure.

1. Introduction

The unique features of the metallic foams such as high strength to weight ratio; high energy and sound absorption; good vibration absorption; and high heat transfer have received much attention from many researchers in recent years. Based on the connectivity of cells, porosity in metallic foams could be divided into two groups of closed and open pores. Metallic foams with closed pores possess higher moduli and strengths; consequently, they are suitable for structural applications such as energy absorption. The metallic foams with open interconnected pores are multi-functional, especially for mass and heat transfer applications. Therefore, the metallic foams with open pores have wider applications in functional structures [1–5]. In spite of the availability of numerous methods for the production of porous metals, a few are able to produce open-cell foams. One of the latest and most widely used processes is the powder metallurgical (PM) process based on using space holder. Up to now, metallic foams have been produced by a variety of space holder agents such as organic materials [4,6], inorganic materials [7], ceramic particles [8], and metallic hollow spheres [9]. The physical properties of foams produced by this technique are influenced by some intrinsic properties of the space holder particles. For instance, volume fraction, shape, and particle size of the space holders play an important role in altering structural features like porosity percentage and distribution; pore shapes and pore sizes of the foam products consequently determine the physical and mechanical properties of foams. For this reason, the study of this effect has been a base for many researches in recent years [6,10–13]. Some of these researches have also led to advances in techniques of metallic foam production [10,14]. For example, Zhao et al. [10] patented the Lost Carbonate Sintering (LCS) method in 2005. In this method, open-pore copper foams with porosity up to 80% have been produced by potassium carbonate space holder. Although this method enjoys a good ability to produce high-porosity copper foams, the researches performed on its ability to control the size, percentage, distribution of porosity, and prediction of pore opening mechanism are not many and unclear yet. On the other hand, study into each of these cases is only possible through the investigation of the three-dimensional structure of foams. High-resolution X-ray computed tomography (XCT) or MRI techniques have been used to probe complex porous structures in recent years [15–22].

* Corresponding author.
E-mail address: panjepour@cc.iut.ac.ir (M. Panjepour).

http://dx.doi.org/10.1016/j.matchar.2015.11.010
1044-5803/

Regarding LCS method, the 3-D structure of foams and connectivity of pores can also be examined by these techniques. Therefore, in this research the ability of LCS method to control the size and percentage of the porosity of the produced foams are first investigated. Also, by employing XCT and SEM images and a series of 3-D image analysis techniques, the manner of the generation of open pores and structural characterization of foams are studied.

2. Experimental procedure

2.1. Foam production method

In order to produce open-cell copper foams, the Lost Carbonate Sintering (LCS) method patented by Zhao et al. [10] has been used. The raw materials employed are commercial Cu powder with a purity of 99.9% and a particle size of <45 μm as the matrix phase. In the first step, in order to increase the strength of the final foams, the copper powder was mechanically activated in a Retsch planetary ball mill model PM100 at 350 rpm for about 5 h with balls per powder (BPP) ratio of 5 [3]. The typical morphologies of raw and ball-milled copper powders are shown in Fig. 1(a–c). Also, a commercial purity potassium carbonate powder with a spherical shape and particle size of 300–600 μm was used as a space holder agent (Fig. 1(d)). The potassium carbonate powder was divided into three different powder sizes according to ASTM standard of mesh numbers (Table 1).

To produce the foam, the ball-milled Cu powder was first mixed with potassium carbonate powder in three different sizes with a volume fraction of 60% and 80%. A small amount of ethanol, roughly 1% vol. fraction of the powder mixture, was added as a binder during mixing. Based on the particle size and volume fraction of potassium carbonate powder, a 7-character code in the form of PxxSyyy was assigned to each foam particle where xx demonstrated the volume fraction of the potassium carbonate powder and yyy represented the nominal mean size of Potassium carbonate powder. The coding system of foam is presented in Table 1. After blending the powders, the powder mixture was uniaxially pressed in a steel mold with a 30 × 30 mm cross-section, and green compacts with a height of 5 mm were prepared. Melting, decomposing and sintering processes of the green compacts were carried out in an electric furnace with an inert atmosphere. The thermal profile applied to the samples is presented in Fig. 2. As shown in this figure, the first green compacts were sintered at 850 °C for 4 h and then carbonate thermal decomposition was performed at 1000 °C for 2 h. Finally, the products of the porous samples were slowly cooled to room temperature.

Table 1
The different samples and details of the coding system.

Sample code	$f^*_{K_2CO_3}$	Potassium carbonate particle size range (mesh)	Potassium carbonate particle size range (μm)	Nominal mean size of potassium carbonate (μm)
P60S720	60	−20 + 30	585–841	720
P60S510	60	−30 + 40	420–595	510
P60S360	60	−40 + 50	297–420	360
P80S720	80	−20 + 30	585–841	720
P80S510	80	−30 + 40	420–595	510
P80S360	80	−40 + 50	297–420	360

$f_{K_2CO_3}$ = The volume percent of K_2CO_3 in powder samples.

2.2. Foams characterization

2.2.1. Thermal analysis

In order to determine the melting and decomposition temperature of potassium carbonate and to study the pore formation mechanism of the foams, simultaneous thermal analysis (STA: DTA–TG) of Cu, K_2CO_3, and Cu-K_2CO_3 powder mixture were performed at a rate of 10 °C/min by means of BAHR thermoanalyse instrument model DTA STA 503.

2.2.2. Microstructural characterization

Scanning Electron Microscopy (SEM) was used by means of Seron Technology model 550i setup to investigate the microstructure of the produced foams. X-ray tomography (XRT) was also used to study the microstructural features of the foams. The 3-D images presented in this study were produced by using the micro-computed tomography device with a spatial resolution (voxel size) of 8.1 μm. Each tomographic image is made of a cubic matrix (array) of the received X-ray values, each corresponding to a 10 × 10 × 10 $(\mu m)^3$ volume voxel (pixel) of the sample. In order to better study the internal structure of the foams, tomographic images were sectioned in x, y, and z direction and were analyzed by the Clemex image analysis and the GeoDict (trial version) software.

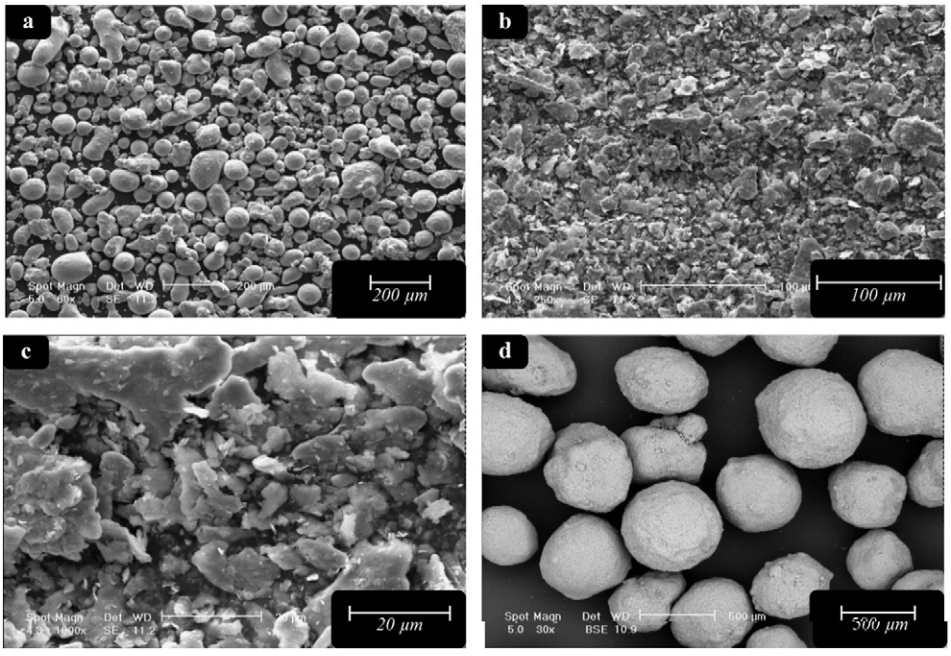

Fig. 1. SEM micrographs of (a) raw Cu powder, (b and c) ball-milled Cu powder at different magnification, and (d) potassium carbonate powder.