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Effects of carbamide shape and content on processing and properties of steel foams

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ABSTRACT

Spherical and irregular carbamide particles covered with Fe–1.5% Mo steel powder have been employed to produce foams using space holder-water leaching technique in powder metallurgy. Foams having porosities between 49.2% and 71.0% were produced after sintering at 1200 °C for 60 min. The effect of carbamide shape and content on the foams' processing, microstructure and mechanical properties has been evaluated. Using irregular carbamide particles and increasing its content decreased leaching time. The extents of specimens' volumetric expansion after carbamide leaching and volumetric shrinkage after sintering also depended on volume fraction and carbamide shape. Final porosity was directly related to the added fraction of carbamide. Pore shape was similar to initial carbamide particle shape. Foams having irregular pore shape were observed to have compressive yield strengths between 20 MPa and 92 MPa and Young's moduli between 0.45 GPa and 2.69 GPa. The corresponding values for foams having spherical pore shape varied between 25 MPa and 112 MPa and between 0.71 GPa and 2.91 GPa, respectively. The foams' strength increased with increasing relative density. Microstructure having spherical pores resulted in better compressive behavior.

1. Introduction

Light-weight materials combining high performance and low cost are desired, especially for automotive, aerospace, and advanced power plant constructions where high temperatures involved. For structural applications, steel foams are much superior to aluminum foams due to their low cost, higher strength, higher heat resistance, and better weldability. Metal foams are generally fabricated by applying liquid- and solid-state processing methods (Ashby et al., 2000). Liquid-state-based techniques are more suited to metals with relatively low melting points, such as aluminum, lead, and zinc. On the other hand, liquid-state processing techniques for steel foams are more difficult to apply due to their high melting points. The solid-state-based powder metallurgy method can produce porous steel parts at much lower temperatures. Among powder-metallurgical metal foam production techniques, the space holder-sintering method can yield a highly porous part with desirable pore size, shape, and volume using an appropriate space holder material. The space holder can be removed from the green part either by using water leaching or thermally. Water leaching the space holder is more attractive since thermal removal takes longer and a low heating rate is required to obtain a crack-free part. In addition, decomposition of the space holder material may release harmful gas species that may interact with the metal. In addition to these reasons, carbamide is generally used in space holder-water leaching technique in powder metallurgy because of its low cost and very high solubility in water.

There are a few published reports on studies of steel foams by space holder-sintering method. Bram et al. (2000) manufactured 316 L stainless steel foams with porosities between 60% and 80% using angular and spherical carbamide and ammonium hydrogen carbonate particles as space-holder materials, and obtained spherical and angular pores between 0.1 mm and 2.5 mm depending on the space holder's shape and size distribution. Carbamide and ammonium hydrogen carbonate were removed thermally from the compacts. Zhang and Zhao (2008) applied the lost carbonate sintering method using potassium carbonate as leachable salt to produce micro-alloyed steel foams (<0.01% C, 0.2% Ni, and 0.15%Mo) with porosities between 50% and 85%, and discussed the effectiveness of carbonate loss and the decomposition route's characteristics in comparison with those of the dissolution route. Bakan (2006) used irregular carbamide as a space holder material to manufacture highly porous 316 L stainless steel specimens with 70% porosity by applying a water leaching technique to remove the spacer, and found that the pore shape, size, and distribution could be controlled with the water-leaching and sintering process. Gülsoy and German (2008) used an irregular carbamide particles as the spacer to produce 17-4 PH stainless steel foams with porosities between 40%

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and 60%, and found that final porosity was directly related to the added fraction of pore former. Mutlu and Oktay (2011a) successfully used irregular carbamide particles to fabricate highly porous 17-4 PH stainless steel specimens with porosities between 39% and 82%, and found that porosity was directly related to added fraction of carbamide. Mutlu and Oktay (2011b) also studied the effect of carbamide shape, size, and volume fraction on the pore properties of stainless steel foam, and found that proper space-holder size, shape, and content could be used to design pore properties. The published reports focused largely on successful production and on the steel foams' properties. There is no information on the effects of carbamide shape on steel-foam processing, such as its removal kinetics and dimensional changes resulting from water leaching and sintering. Dimensional control is also a significant concern during foam fabrication by powder metallurgy, since die size must be designed taking dimensional changes into account.

Molybdenum increases the steel's strength and heat resistance. Steel powder pre-alloyed with molybdenum could be suitable for producing steel foams for high-temperature applications using space holder-water leaching technique in powder metallurgy. Fe–1.5%Mo is a water-atomized, pre-alloyed steel powder known as Astaloy Mo. This grade exhibits high compressibility and a homogenous microstructure after sintering. Aly (2004) used the slip reaction foam sintering (SRFS) method to produce Fe–1.5Mo foams with densities between 1.3 g cm^{-3} and 2.0 g cm^{-3}. The foams' compressive strengths were less than 9 MPa at room temperature. Banhart (2001) pointed out that the SRFS method may yield lower strength values resulting from crack formation in the foamed material. Thus a space holder-sintering method could be used for processing Astaloy Mo foams having better mechanical properties.

In this study, spherical and irregular carbamide particles covered with Fe–1.5% Mo steel powder have been employed to produce foams using space holder-water leaching technique in powder metallurgy and foams compressively tested. The effect of carbamide shape and volume fraction on processing, microstructure, and mechanical properties of the steel foams has been evaluated.

2. Experimental procedure

Powder metallurgy based space holder-water leaching technique involves four main stages: powder mixing, compaction, removing of space holder by water leaching, and sintering. A binder added steel powder and a space holder powder are first mixed at a given volume ratio. The powder mixture is compacted and then the space holder removed by water leaching to obtain a green steel foam. Finally, the green steel foam is sintered to increase its strength.

In this study, a pre-alloyed, water-atomized steel powder commercially known as Astaloy Mo obtained from Höganäs Company (Sweden) was used. The steel had a maximum carbon content of 0.01% and was alloyed with 1.5% Mo. The binder for green strength was paraffin wax, supplied by Merck, Germany. Carbamide was chosen as a space-holder material for its very high solubility in water. Spherical carbamide particles, supplied by Merck, Germany, had a density of 1.34 g cm^{-3}, melting temperature of 133 °C, and solubility in water of more than 1000 g L^{-1} at 20 °C. As-received spherically shaped carbamide particles were sieved to obtain the −1400 + 1000 μm fraction. In addition, as-received spherically shaped carbamide particles were crushed and sieved to obtain the −1400 + 1000 μm fraction with irregular shape.

Paraffin wax was used at 2 wt.% based on the steel mass. The steel powder was mixed manually with hot paraffin wax solution. The weight ratios of the steel powder to the amount of carbamide were calculated to obtain defined porosities of 50–80% in the specimens. The spacer was moistened with distilled water to form a sticky surface and waxed steel powder was added. Steel and spacer particle mixing was performed in a Turbula mixer for 60 min. Typical morphologies of the steel powder, carbamide particles, and coated carbamide particles with the steel powder are shown in Fig. 1. The steel powder had a size distribution between 45 μm and 150 μm with an average particle size of 109 μm and a rounded but irregular shape. A homogeneous coating of carbamide particles with the steel powder was obtained. The Arnold meter determined the coated carbamide particles' apparent densities.

A series of compaction experiments was carried out by varying compaction pressure from 100 MPa to 400 MPa to determine its optimum. Pressures greater than 200 MPa broke the carbamide particles thereby changing the designed pore size and morphology. In addition, pressures greater than 200 MPa did not allow the spacer to be leached out quickly. On the other hand, specimens compacted at pressure less than 200 MPa could not retain their shapes after removal of the spacer in water. In view of these facts, optimum compaction pressure leading to a sound product is determined to be 200 MPa for steel containing carbamide as the space holder. The coated carbamide particles then compacted uniaxially at 200 MPa into cylindrical specimens with a 12 mm diameter and a height of about 18 mm.

The green specimens were immersed in distilled water at room temperature to leach the carbamide. The conductivity method (Chin and Kroontje, 1961) was applied to determine dissolved carbamide. A Jenco 6350 PH/CON/TEM model conductometer, Taiwan, was used for this purpose. The specimens were dried in air at room temperature for one day after the spacer in the specimens was removed by water leaching. The green specimens' densities were determined from measurements of the specimens' weights and dimensions.

The specimens were sintered at 1200 °C for an hour under high-purity hydrogen in a tube furnace (Lenton, UK). The furnace was purged with N_2 gas before the sintering process. The sintered steel foam specimens' density and porosity content were determined employing Archimedes' principle in a Sartorius precision balance equipped with a density-determination kit. Fractions of open and closed porosities were determined by weight measurements prior to and after dipping the specimens in boiling paraffin at 150 °C. The pore morphology of the foam specimens was examined using the SEM. The pores' size distribution, size, and shape were determined using Clemex Vision PE commercial image-analyzer software. The area of each pore on the SEM image was calculated. Spherical diameter as pore size and sphericity as pore shape were then determined. The specimens' pores were filled with a cold-hardening epoxy resin before grinding and polishing then etched in 2% Nital solution for optical examination.

The foam specimens' mechanical properties were studied using the compression test performed on a Zwick-Roell Z050 material-testing machine. Compression tests were conducted at a crosshead speed of 0.5 mm min^{-1}. Stress was calculated using the respective specimen's apparent cross-sectional area. Young's modulus for each specimen was subsequently determined from the slope of the corresponding stress–strain graph.

3. Experimental results

Apparent densities of the coated carbamide particles and green densities of the compacts are given in Table 1. Spherical and irregular carbamide particles' apparent densities were determined to be 0.71 g cm^{-3} and 0.67 g cm^{-3}, respectively. As expected, the compacts' green densities increased with decreasing volume fraction of carbamide. The density of the compacts having irregular and spherical carbamide particles decreases by 15.1% and 16.4%, respectively as the volume fraction of carbamide increases from 50% to 80%.