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Compressive properties of porous metal fiber sintered sheet produced by solid-state sintering process

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ABSTRACT

A novel porous metal fiber sintered sheet (PMFSS) with three-dimensional reticulated structure was fabricated by using solid-state sintering method of copper fibers. Uniaxial compressive test was carried out to investigate the effects of porosity and manufacturing parameters on the compressive properties of PMFSS. During the compressive process, it was found that the PMFSS initially exhibited short-term elastic deformation, and then quickly entered into the compact densification deformation stage. The stress-strain plots showed no obvious yield stage in the whole uniaxial compressive process. Under given stress, the PMFSS with higher porosity exhibited higher strain, hence implying lower effective stiffness. Additionally, our results showed that higher sintering temperature or longer sintering time would soften the PMFSS.

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1. Introduction

The porous metal fiber sintered sheet (PMFSS), a new type of porous metal materials, has a three-dimensional network structure featuring high porosity and large specific surface area. It has been widely used in the defense and military, petrochemical industry, metallurgical machinery, environmental protection for its excellent performance in filtration and separation [1], fluid distribution [2], energy absorption [3], biomedical device [4], catalytic reaction [5], heat transfer [6], and so on. In recent years, with the extension of its application field and increasing complexity of its working environment, it is important to understand the relationship between structure and properties of PMFSS to widen its application. However, the previous enormous research work focused on the mechanical properties of metal foam including aluminum foam [7], copper foam [8], and other metal foams [9]. There were not many reports available in the literatures that describe the relationship between microscopic structures and mechanical properties of porous fiber material sintered by using stainless steel fibers and wires [10,11].

As for the metal foam, Gibson and Ashby et al. [12,13] successfully developed a theory to predict the elastic modulus and strength as functions of density, as well as the stress–strain relationship under the compressive deformation process. Their theory was verified by a number of experimental results. The follow-up theoretical

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study on energy absorption shed much light on the properties of metal foam. Hyun and Nakajima [14] investigated the effect of pore structure on the compressive process of copper foam, and then conducted a preliminary analysis of the energy absorption. Koza et al. [15] started the preliminary research on the compressive process of aluminum foams with different porosities and dimensions. Liu [16,17] developed a theoretical model for the relationship between structural parameters and mechanical characteristics of metal foam with high porosity. A series of experiments were conducted using nickel foams prepared under different conditions, showing good consistence with the theoretical models.

As for the PMFSS, Ducheyne et al. [18] produced a less porous PMFSS using two types of AISI 316L stainless steel fibers with diameters of 50 μ m and 100 μ m, of which the tensile and compressive properties were studied. Clyne and Markaki [10,19] fabricated highly porous sheets by using liquid phase sintering of short stainless steel fibers with diameter of 100 µm, followed by an electroplating step to coat the surface with a 5 µm thick copper layer. The PMFSS was obtained after sintering the coated copper fibers at temperature of 1100-1200 °C for 5 min, giving rise to high porosity varying from 75% to 95%. And the tensile strength of all sheets was less than 1 MPa. Recently, Qiao et al. [20] fabricated a PMFSS through vacuum sintering of metal fiber with the diameter of 12 μm. The analysis of microscopy morphology was conducted using scanning electron microscopy. Furthermore, the quasi-static compressive properties of PMFSS were studied by using MTS858 system at room temperature [20]. In the present study, the PMFSS with a three-dimensional reticulated structure is produced using the solid-state sintering of copper fibers. The compressive process of PMFSS is investigated by using the uniaxial compressive test. Finally, the effect of the porosity and manufacturing parameters on the compressive properties of PMFSS was studied in detail.

2. Experimental procedure

2.1. Manufacturing process of PMFSS

As reported previously [21], the processing procedure of PMFSS was divided into following five steps: fiber chipping, mold-pressing, sintering, cooling, and testing. First of all, the continuous copper fibers were fabricated by cutting method with a multi-tooth lathe tool. These copper fibers were then cut into segments with the length ranging from 10 to 20 mm. Next, the copper fibers with random directions were uniformly put into the packing chamber of the mold pressing equipment, and then pressure was applied by screwing the bolts. In this way, the semi-finished PMFSS with the same shape as the predetermined packing chamber was obtained. Sintering was carried out in the box-type furnace which provided the hydrogen gas atmosphere with constant pressure of 0.3 MPa. The sintering temperature was in the range from 700 °C to 1000 °C and controlled by a programmable temperature controller. Stage heating method was used to optimize the heating rate. The heating rate was kept at 300 °C/h when the temperature was below 800 °C, while reduced to 200 °C/h as the temperature was above 800 °C. The sintering time was set as either 30 min or 60 min. When the sintering was completed, the sample was removed from the furnace and cooled in air to the room temperature. Finally, the mold pressing equipment was disassembled and the PMFSS was ready for characterization and compressive test.

2.2. Characterization and compressive test of PMFSS

Since the obtained PMFSS has a regular geometric shape, we can calculate the average porosity using the quality-volume method formulated by

$$E(\%) = (1 - \frac{M}{\rho V}) \times 100,\tag{1}$$

where *V* is the volume of PMFSS (cm³) *M* is the mass of PMFSS (g) and ρ is the density of red copper (g/cm³).

Scanning electron microscopy (JSM-6380LA, Japan) was adopted to observe the microscopic structure of PMFSS. The compressive test was carried out on an electronics universal testing machine (RGL-20A) with maximum load of 20 kN, performing both test control and data acquisition. To avoid the size effect, the



Fig. 1. Photographs of the PMFSS samples with different porosities prepared for compressive test.

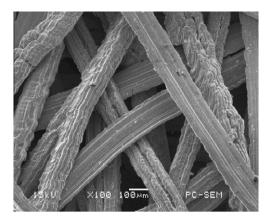


Fig. 2. SEM image of the PMFSS with 80% porosity sintered at 800 °C for 30 min.

minimal length, width, and height of samples are required to be at least seven times larger than the pore size [22]. The value of the average pore size ranges from 100 μm to 1 mm depended on the manufacturing parameters [21]. Therefore, all the PMFSS samples were designed to be 55 mm in length, 55 mm in width, and 10 mm in height. Fig. 1 shows the photographs of the PMFSS samples with different porosities prepared for compressive test. In this study, the compressive process of all test samples was conducted at a constant compressive rate of 1.5 mm/min using the crosshead, which was automatically controlled by the computer software. The load per unit apparent area was taken as the stress. The strains were calculated from the crosshead displacement and the initial measured length of the sample [23]. All the compressive tests were performed at room temperature (approximately 25 °C).

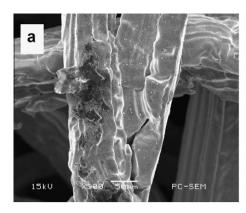
3. Results and discussions

3.1. Microstructures of PMFSS

Fig. 2 shows the SEM image of the PMFSS with 80% porosity sintered at 800 °C for 30 min. As mentioned previously, the copper fibers were distributed randomly in the pressing mold. The pressurization was conducted to produce the mesh structure with many contact regions between the fibers. In the sintering process, the sintering joints between fibers were formed under the set temperature as a result of material migration, and then fiber's metallurgy unification happened, resulting in the PMFSS with threedimensional reticulated structure. As shown in our previous work [21,24], we found that there were two kinds of sintering joints present in the PMFSS including fiber-to-fiber surface contact and crossing fiber meshing, as shown in Fig. 3a and b, respectively. It was also noted that the PMFSS had a wide porosity range from 60% to 98%, large pore size, and interconnected pores. Moreover, multiscale surface cracks were found on the fibers. These cracks were created during the fabrication process of the fibers. The number of these cracks in the fibers could be reduced by optimizing the fiber's processing parameters. In general, cracks affect the tensile instead of compressive properties of materials [25]. Therefore, here we will not advance further in this aspect but leave the discussion on the crack effect to the future work. In addition, the mechanical properties of PMFSS with three-dimensional reticulated structure can be tailored for different applications by varying the manufacturing parameters of PMFSS.

3.2. Uniaxial compressive process of PMFSS

Uniaxial compressive tests were conducted by using PMFSS fabricated under different manufacturing parameters. The compressed samples exhibited little change in both length and width



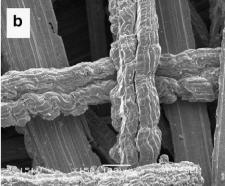
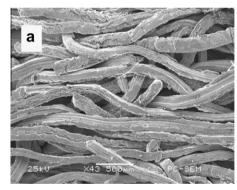


Fig. 3. SEM images of bonding types between fibers in the PMFSS: (a) fiber-to-fiber surface contact and (b) crossing fiber meshing.

Table 1Dimensions of PMFSS before and after compressive test.

No	Manufacturing parameters	Porosity (%)	Dimension before compression (length \times width \times height mm)	Dimension after compression (length × width × height mm)
1	Sintering at 800 °C for 30 min	70	$55\times55\times10$	$56.1\times55.5\times7.56$
2	Sintering at 800 °C for 30 min	80	$55 \times 55 \times 10$	$55.4 \times 55.4 \times 5.9$
3	Sintering at 800 °C for 30 min	90	$55 \times 55 \times 10$	$54.2\times55.1\times3.6$
4	Sintering at 800 °C for 60 min	80	$55 \times 55 \times 10$	$55.6 \times 55.1 \times 6.12$
5	Sintering at 1000 °C for 30 min	80	$55\times55\times10$	$55.3\times55.1\times6.13$



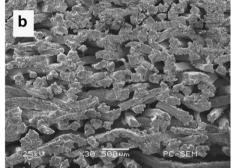


Fig. 4. Microstructures of the PMFSS after uniaxial compressive process (a) top view and (b) cross-section. Sample was sintered at 800 °C for 30 min.

even at large deformation in height compared to the sample without compression. Moreover, it was found that the compressed PMFSS with 70% porosity had the final height of 7.56 mm, and the sample with 90% porosity had the final height of 3.6 mm, as shown in Table 1. Therefore, these experimental results demonstrated that the porosity also has a great impact on the compressive properties. The PMFSS with the lower porosity retains higher height after compressive process. This characteristic of PMFSS provides the convenience for specific application areas with strict requirement of three-dimensional shape. In addition, the height of compressed PMFSS sintered at 800 °C and 1000 °C for 30 min was slightly higher than that sintered at 800 °C for 30 min. Therefore, we conclude that the compressed PMFSS showed small changes in height when the sintering temperature and sintering time are changed. After the uniaxial compressive process, the microstructures of the PMFSS sintered at 800 °C for 30 min are shown in Fig. 4. It can be seen that the three-dimensional reticulated structure of PMFSS disappeared, and a majority of fibers were compressed onto the plane perpendicular to the direction of compressive force, as shown in Fig. 4a. In addition, the SEM image of the cross-section showed that compact densification of PMFSS occurred after compression, as shown in Fig. 4b.

3.3. Effect of porosity on the uniaxial compressive properties

Porosity, as the most intrinsic feature of porous materials, plays an important role in determining the compressive properties of PMFSS [26]. To study the effect of porosity on the uniaxial compressive properties, the PMFSS samples with different porosities fabricated at the same sintering parameters were used to conduct the comparison test. The compressive test samples with three porosities were sintered at 800 °C for 30 min, including 70%, 80%, and 90%. Fig. 5 shows the uniaxial compressive stress-strain plots of the PMFSS sintered at 800 °C for 30 min with different porosities. It is the common trend of the curves that the stress increases gradually with the increasing strain. Under given stress, the PMFSS with higher porosity exhibited higher strain, giving rise to lower effective stiffness. This general trend is consistent with the results of previous study on aluminum foam [15], which implies that porosity is by far the most important variable influencing the mechanical properties of porous metal. Moreover, the PMFSS first experienced a temporary elastic deformation stage, and then directly entered into the compact deformation stage. No yield platform stage was observed throughout the uniaxial compressive process. This behavior is different from that of the metal fiber

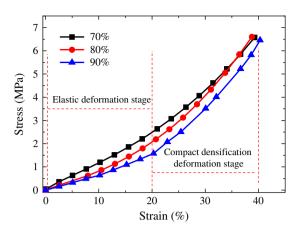


Fig. 5. Uniaxial compressive stress–strain plots of the PMFSS with different porosities. All samples were sintered at $800\,^{\circ}\text{C}$ for $30\,\text{min}$.

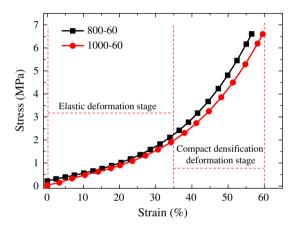


Fig. 6. Uniaxial compressive stress–strain plots of the PMFSS sintered at 800 $^{\circ}$ C and 1000 $^{\circ}$ C for 60 min. Both samples have porosity of 80%.

sintered sheet reported in the literature [20]. In general, the compressive process of the porous metal with low density is composed of three stages including linear elastic zone, yield plateau, and densification zone with rapidly increase in the stress [22,27]. The absence of the yield plateau region in our PMFSS may be attributed to the unique three-dimensional reticulated structure of PMFSS with high porosity.

3.4. Effect of manufacturing parameters on the uniaxial compressive properties

Several methods have been developed to produce the porous metal including the melt foaming, sintering, diffusion bonding and welding [10]. However, little has been reported about the relation between the manufacturing parameters and mechanical properties of PMFSS fabricated using the metal fibers [10,11]. Fig. 6 shows the uniaxial compressive stress-strain plots of the PMFSS with 80% porosity sintered at 800 °C and 1000 °C for 60 min. Comparison between Figs. 5 and 6 implies that the elongation of sintering time would soften the PMFSS. Furthermore, the sintering temperature also plays an important role in forming the porous structure during sintering [11]. It can be seen that samples sintered at 800 °C and 1000 °C exhibit similar stress-strain behavior. Under given strain, the stress value for the sample sintered at 800 °C was slightly higher than that for the sample sintered at 1000 °C. Therefore, increasing the sintering temperature could also soften the PMFSS. Such softening mechanism may be attributed to the combined action of the growth of the sintering joints and the coarsening of grains in the metal fibers [24]. The compressive properties of PMFSS can be tailored according to the applications by varying the sintering temperature and time.

4. Conclusions

The uniaxial compressive test was conducted to study the compressive properties of PMFSS produced by the solid-state sintering process under different manufacturing parameters. In the uniaxial compressive process, the PMFSS initially exhibited short-term elastic deformation, and then directly entered into the compact densification deformation stage. It was found that the PMFSS maintained the three dimensional rectangular shapes after the compressive process. Compared to the sample before compressive procedure, the compressed samples presented the little change both in length and width, but showed the obvious decrease in height. Moreover, the porosity and manufacturing parameters were changed to study the compressive properties of the PMFSS. Our experimental results indicated that the porosity has an important effect on the compressive properties. Under given compressive stress, the PMFSS with higher porosity exhibited larger strain, and therefore lower stiffness. In addition, it was found that increasing the sintering temperature or time would soften the PMFSS. The results obtained in this work will not only help the further development of porous materials, but also provide an important technical guideline for the application of PMFSS in catalysis and acoustic emission.

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