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An Innovative Fabrication Process of Porous Metal Fiber Sintered Felts with Three-Dimensional Reticulated Structure

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A novel porous metal fiber sintered felt (PMFSF) with a three-dimensional reticulated structure has been produced by the solid-state sintering of copper fibers. The copper fibers, with several microstructures distributed onto the surface, were fabricated using the cutting method. The Scanning Electron Microscope (SEM) results revealed that there were two kinds of sintering joints present in the PMFSFs: fiber-to-fiber surface contact and crossing fiber meshing. In the sintering process, the surface microstructures of the fibers helped to improve the forming process of the PMFSFs, as a result of high surface energy. Furthermore, the effect of different sintering parameters on the forming process of the PMFSFs was studied in detail, including the sintering temperature and holding time. The sintering temperatures had a significant influence on the surface microstructures of single fiber and specific surface area of the PMFSFs, but the holding time did not. The optimal PMFSF with a three-dimensional reticulated structure and larger specific surface area was produced by sintering copper fibers at 800°C for 30 minutes in the reduction atmosphere.

Keywords Copper fiber; Metal fiber sintered felt; Porous metal; Sintering.

Introduction

Porous metal material, an interesting engineering material with excellent performances, is being explored due to its porous structure and unique properties. In recent years, porous metal materials have been widely used in aerospace, metallurgic, mechanic, petrochemical, energy, pharmaceutical, architectural, and transportation fields [1–5]. Porous metal fiber sintered felts (PMFSFs), made from metal fibers, attract increasing attention, because of their three-dimensional reticulated structure, interconnected pores, high porosity, and large specific surface area [6–8]. These structural characteristics create potential to overcome the shortfalls of other porous materials. For example, porous organic polymer materials have low intensity and cannot withstand high temperature; porous ceramics have poor brittleness and bad thermal shock resistance; metal wire mesh are easily destroyed and plugged; sintered powder materials are fragile, and often fail to allow a sufficient amount of liquid to pass through. Therefore, the research related to the new type of PMFSF helps to enrich the entire system of porous metal materials. As part of a new generation of high-performance structural and functional materials, the PMFSFs have been widely used in many fields, including catalyst supports [9–11], heat exchangers [12, 13], porous metal electrodes [14], energy-saving heat pipes [15, 16], clean energy convertors [17], surface combustion technology [18], shock absorbers [19], high temperature gas dust removal [20], automotive exhaust purification [21], fuel cells [6, 22], biomedicine [23], high temperature seal and wear-resistant components [24], and so on.

At present, a variety of processing techniques have been developed to fabricate the PMFSFs. Clyne and Markaki developed the transient liquid-phase sintering technology to sinter stainless steel fibers. In this way, the stainless steel sintered felts with porosity up to 85% and fracture energy higher than 1 kJ/m^2 were obtained [25]. Japanese researchers fabricated a PMFSF with high porosity, good insulation, and machinability by sintering and suppressing short, curly metal fibers [26]. Researchers from Germany Fraunhofer Institute for Manufacturing and Advanced Materials fabricated the porous metal materials by using short metal fibers produced by melt extraction, then used the fiber materials as filtering components [27–29]. When the bundle-drawing method and sintering process were combined, stainless steel fiber sintered felts were produced, and then applied to remove the dust in gas at high temperatures [20]. Recently, porous metal sheets with a fiber-core sandwich structure were developed, and further research work is underway [30–32].

In this study, a novel PMFSF with three-dimensional reticulated structure was produced by the solid-state sintering of copper fibers. Based on the microstructures characteristic of copper fibers, the forming process of the PMFSFs was analyzed. Finally, the effect of various sintering parameters on the forming process of the PMFSF was studied in detail.

Experimental

Fabrication of the Copper Fibers

The red copper bar with 41.90 mm in diameter and 400 mm in length was used as workpiece. It was processed on a common horizontal lathe (No. C6132A) using a multi-tooth tool. The multi-tooth tool was made

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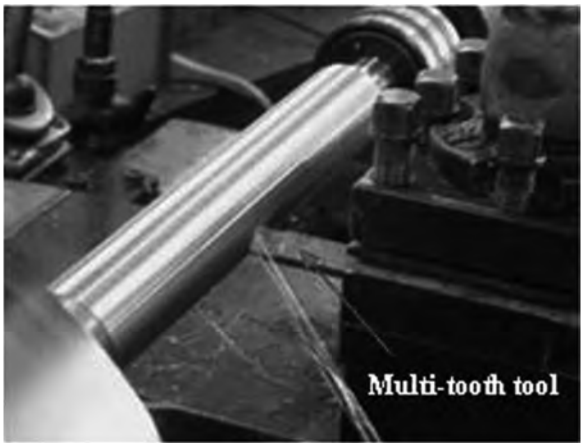

FIGURE 1.—Fabrication process of copper fibers using the multi-tooth tool.

from V3N superhard high-speed steel (Material model: W12Mo3Cr4V3N). Before it was processed, the multi-tooth tool was installed at angle θ ($\theta = 45°$) on the tool support, then was adjusted to be parallel with the axis of the workpiece. Figure 1 shows the fabrication process of copper fibers using the multi-tooth tool. The flank face of the multi-tooth tool was divided into many tiny triangle-shaped teeth by linear edge. In the cutting process, several tiny teeth were involved simultaneously. There was a chip-bifurcating action of multi-tooth tool during the cutting process. Thus, several continuous fine copper fibers were fabricated. In this way, the continuous fine copper fibers with high strength and ductility can be obtained by setting suitable cutting parameters. Figure 2 shows the appearance of continuous fine copper fibers fabricated by the multi-tooth tool.

Processing Procedure of the PMFSFs

The processing procedure of the PMFSFs is divided into the following five steps: chipping fibers, mold-pressing, sintering, cooling, and testing. Firstly, the continuous copper fibers were chipped into short fibers with a length range of 10 to 20 mm. Next, the as-prepared copper fibers were randomly packed into the predetermined packing chamber of the mold pressing equipment, and then a pressure from the bolts was applied to the metal fibers. In this way, the semifinished PMFSFs with the same shape as the predetermined packing chamber were obtained. The (a) appearance and (b) assembling principle of the metal fiber mold-pressing equipment is shown in Fig. 3. Moreover, the height and shape of the PMFSFs can be adjusted by changing the design of the packing chamber in the mold-pressing equipment.

FIGURE 2.—Appearance of continuous fine copper fibers fabricated by the multi-tooth tool.

To prevent the copper fibers from oxidizing, heat treatment was carried out in a box-type furnace (Model FXL-12-1, Foshan City Foxing Dianlu Co., Ltd., China) in a gas protection atmosphere. The electrical heating component of the furnace was a high temperature alloy resistance coil (Material model OGr27Al7Mo2). The sintering temperature was adjusted by a programmable temperature controller. The sintering time was set for either 30 or 60 minutes. To optimize the heating rate in the sintering process, we used the stage heating method. When the temperature was below 800°C, the heating rate was kept at 300°C/h, and when the temperature was above 800°C, the heating rate was decreased to 200°C/h. After the mold pressing equipment was put into the sintering furnace, nitrogen gas was used to flush the chamber. After the chamber had been purged of air, it was filled with hydrogen gas, which was later ignited. The pressure of the hydrogen gas in the furnace chamber was kept at 0.3 MPa. When the sintering was completed, the sample was moved

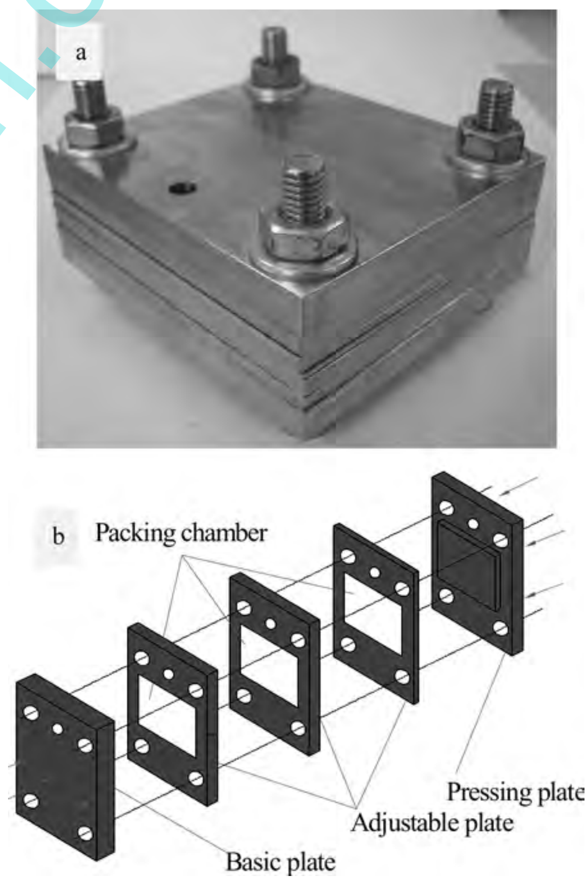

FIGURE 3.—(a) Appearance and (b) assembling principle of the plate-laminated mold-pressing equipment of metal fiber.

and cooled to room temperature, at which point the mold pressing equipment could be disassembled. After a final test and examination, the PMFSF with the specified shape was obtained.

Characterizations of the PMFSF

The porosity (E) of the PMFSF is calculated by the following formula:

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

where V is the volume of the PMFSF (cm^3), M is the mass of the PMFSF (g), and ρ is the density of red copper (g/cm^3).

The macroscopic appearances of the PMFSF were recorded by a digital camera. The microscopic morphology of the continuous fine copper fibers was observed by using a scanning electron microscope (Model JSM-6380LA, JOEL, Japan). The scale of the surface microstructures was determined in a confocal microscope (Model TALYSURF CLI 1000, Taylor Hobson precision Corp., USA) using the surface scanning method and atomic force microscope (Model CSPM-5000 being Nano-Instruments Ltd., China) with the contacting mode. The specific surface area of the copper fibers was characterized by the N_2 adsorption method at 77 K (Model ASAP 2010, Micromeritics Corp., USA). The phase components of the PMFSFs were analyzed by X-ray diffraction (XRD) (Model D/MAX-IIIA, Rigaku Corp., Japan).

Results and discussion

Surface Microstructures of the Copper Fibers

Figure 4 shows the surface microstructures of continuous copper fibers fabricated by the multi-tooth tool. To fabricate the copper fibers, the diameter of the workpiece (d_w), feed (f), back engagement (a_p), and cutting speed (n) were 41.90 mm, 0.08 mm/r, 0.25 mm, and 180 r/min, respectively. Compared to the metal fibers fabricated by melt extraction method [25, 28, 29], the copper fibers fabricated by the cutting method exhibited a rougher surface. Furthermore, we observed that a large number of microstructures were distributed onto the surface of the copper fibers, as shown in Fig. 4(a). The equivalent diameter of the copper fibers was less than 100 μm. The specific surface area was measured to be 0.832 m^2/g. Therefore, these surface microstructures significantly increase the specific surface area of the copper fibers. Figure 4(b) shows the surface profile image of the copper fibers. The height of surface microstructures of the copper fibers varied within a range of 30 μm in the scanning area about 60 μm in length and 20 μm in width. Figure 4(c) shows the AFM image of the copper fibers. It was noted that several mountain-like microstructures within nanometer scale had been distributed on the surface of the

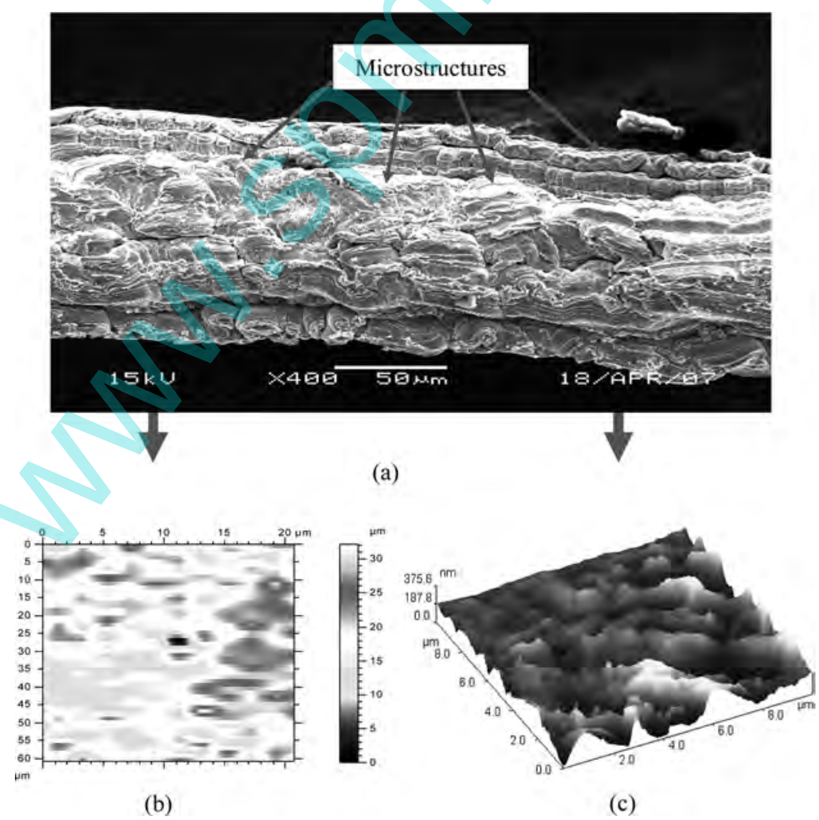

FIGURE 4.—(a) SEM image, (b) surface profile image, and (c) AFM image of the surface microstructures of continuous copper fibers fabricated by the multi-tooth tool.

FIGURE 5.—Appearance of the PMFSF with different specified shapes.

microstructures. These multiscale surface microstructures of the copper fibers can be explained as the micro/nanoscale particles which are formed on the surface of the copper fibers. They may be produced by the metallic plastic flow resulting from the combination of the cutting resistance and extrusion force in the forming process of the copper fibers.

Forming Process of the PMFSFs

Figure 5 shows the appearance of the PMFSFs with different specified shapes. As mentioned previously, the copper fibers were distributed randomly in the pressing mold. The pressurization resulted in a mesh with many contact regions between the fibers. In the sintering process, the sintering joints between fibers were easily formed under the setting temperature as a result of material migration, and then the metallurgy union between fibers started to happen. Finally, the PMFSFs with a three-dimensional reticulated structure were obtained. The porosity of the PMFSFs varied from 60% to 98%, depending on the weight of the copper fiber. In addition, the results show that there are two kinds of sintering joints between fibers present in the PMFSFs after the completion of sintering process: fiber-to-fiber surface contact [Fig. 6(a)] and crossing fiber meshing [Fig. 6(b)].

For fiber-to-fiber surface contacts, the material migration between fibers plays an important role in the sintering process, arising from the high surface energy of the microstructures. Before sintering, a large number of microstructures with high surface energy are distributed on the surface of the copper fibers, as shown in Fig. 4(a). When the copper fibers are filled into the packing chamber of pressing mold, some surface microstructures contact each other, and some surface microstructures are separated. According to the thermodynamics theory, the energy of whole system for the semi-finished PMFSFs is out of equilibrium situation, since the summation of the exterior energy is not the lowest situation. Therefore, matter migration spontaneously changes toward the lower surface energy in the sintering process. At the beginning of the sintering process, the sintering phenomenon happens on the surface of microstructures to form the contact faces between fibers. While the sintering process is going, the bonding faces become coarser and coarser to produce the strong sintering joints. Finally, the fiber-to-fiber surface contacts between fibers are obtained in the PMFSFs. These results are probably due to the material migration between microstructures distributed on the surface of the different copper fibers, which is propelled by the surface diffusion. Furthermore, the smaller surface microstructures is, the larger the specific surface area and surface energy are. The microstructures on the surface of the fibers are eager to emit energy to the lower state, making the surface diffusion more easily carried out, which helps to rapidly achieve the metallurgy union between fibers. Therefore, we concluded that the surface microstructures of the copper fibers improve the sintering process of the fibers.

The forming process of the crossing fiber meshing can be explained as follows. On one hand, the copper fibers are distributed in a random, interlaced manner into the packing chamber of the pressing mold, and the meshing phenomena between fibers is generated, as a result of the displacement and distortion of the fibers under the pressure. So these characteristics give the meshing force to produce the meshing joints between fibers in the sintering process. On the other hand, the surface microstructures can increase the contact region between fibers to strengthen the crossing fiber meshing to some extent, as a result of larger specific surface area. Under the combined action of meshing force and surface microstructures, the sintering joints are easily formed in the meshing region during material migration in the sintering process. Finally, PMFSFs with a three-dimensional reticulated structure were obtained after the sintering procedure. Compared to the powder metallurgy product, the PMFSFs have higher porosity, larger pore size, and better penetrability. Thus, they have been successfully used as catalyst supports and wick materials [10].

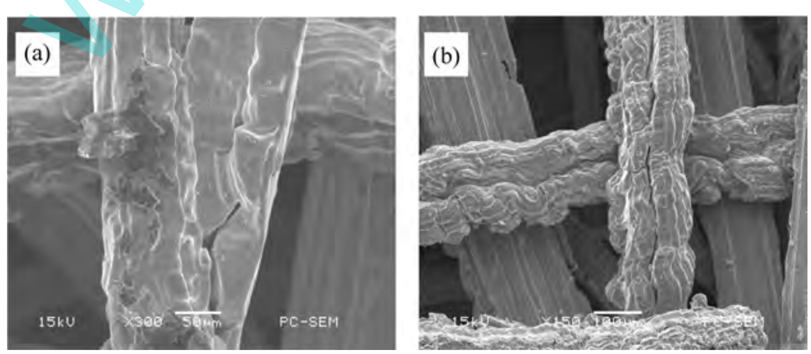

FIGURE 6.—Two kinds of sintering joints between fibers in the PMFSF: (a) fiber-to-fiber contact joints, and (b) fiber-to-fiber mechanical meshing.

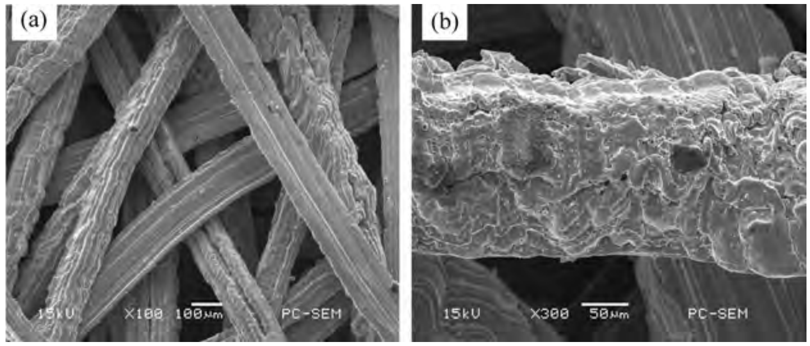

FIGURE 7.—SEM images of (a) the PMFSF (porosity is 80%) and (b) a single copper fiber after sintering at 800°C for 30 min.

Effect of Sintering Parameters on the Forming Process of the PMFSFs

In this study, the forming process of the PMFSFs can be considered as the solid-state sintering process of the copper fibers in a reduction atmosphere. The sintering temperature and holding time are two important sintering parameters in the forming process. The effect of these parameters on the PMFSFs is explained in the following section.

Sintering Temperature. The sintering temperature is an important sintering parameter in the forming process of the PMFSF. It is very difficult for the sintering joints to form under low sintering temperature. However, if the temperature is too high, the reticulated structure will be destroyed, and even some fibers could happen to fracture, so the required three-dimensional reticulated structure of the PMFSF will not be obtained. Within a reasonable range of sintering temperature, the increasing sintering temperature helps to accelerate the velocity of material migration in the sintering process. Figures 7(a) and (b) shows the SEM image of the PMFSF and a single copper fiber after sintering at 800°C for 30 minutes. Figures 8(a) and (b) shows the SEM image of the PMFSF and a single copper fiber after sintering at 1000°C for 30 minutes. Both of the PMFSFs have a porosity of 80%. It can be seen that there are several sintering joints between the fibers, and both of PMFSFs with the three-dimensional reticulated structure were obtained under the different sintering temperatures, as shown in Figs. 7(a) and 8(a).

When analyzing the surface structures of single fiber, it is worth noting that the copper fibers have a significant number of surface microstructures after being sintered at 800°C for 30 minutes, as shown in Fig. 7(b). However, when the sintering temperature was increased to 1000°C, a majority of the microstructures disappeared, causing the surface of the fibers to become smooth, even the diameter of the fibers is also decreased slightly, as shown in Fig. 8(b). Furthermore, the BET test results show that the specific surface area of the PMFSF sintered at 800°C and 1000°C for 30 minutes, are 2.032 m^2/g and 0.789 m^2/g, respectively. When the sintering temperature is increased, the specific surface area is decreased greatly. Thus, a higher specific surface area can be obtained at a lower sintering temperature. In addition, the XRD spectral images indicate that the PMFSFs sintered at 800°C and 1000°C for 30 minutes in a reduction atmosphere pose the same phase, as shown in Figs. 9(a) and (c). The main phase of both felts was copper. There were no other impurities present in the PMFSFs. The components of the PMFSFs were stable and uniform. Therefore, the sintering temperature has little influence on the phase components.

Holding Time. The holding time is also a key sintering parameter in the sintering process. In general, extending the holding time is an effective way to improve the material migration between fibers. It promotes the growth and generation of sintering joints, helping to produce strong sintering joints. However, with unreasonable extension of the holding time, the speed of recrystallization is increased for the internal grains of the fibers, so it leads to the

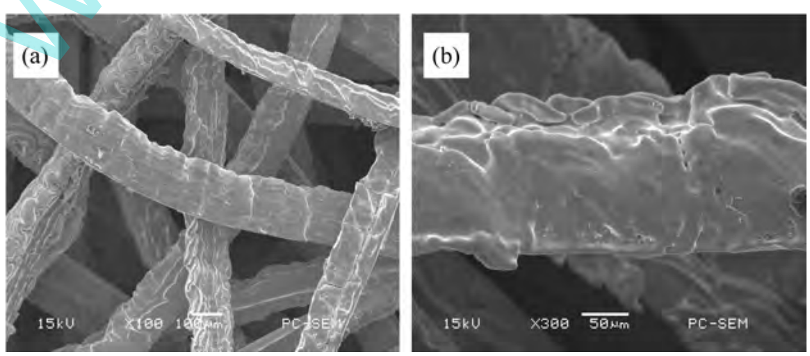

FIGURE 8.—SEM images of (a) the PMFSF (porosity is 80%) and (b) a single copper fiber after sintering at 1000°C for 30 min.

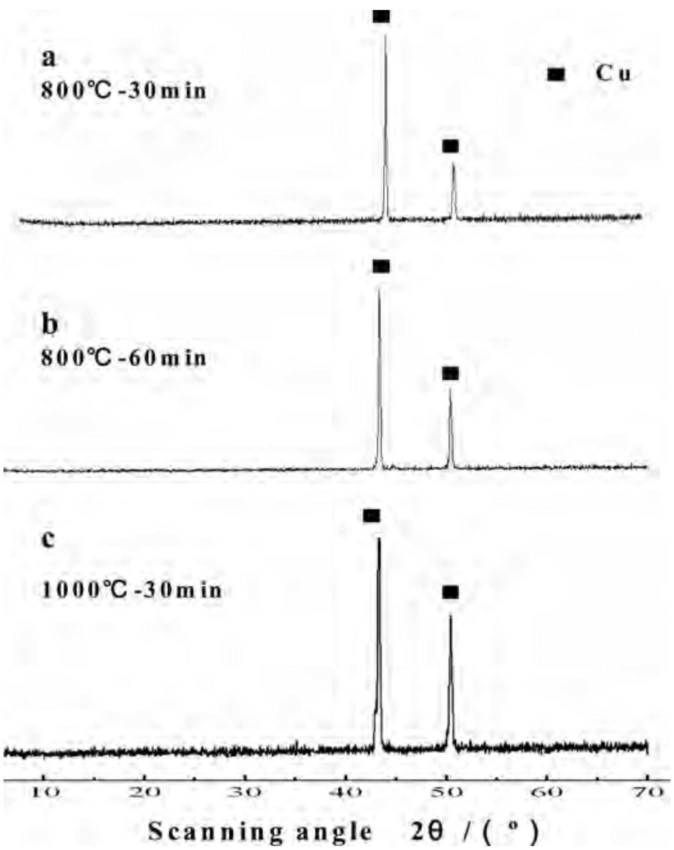

FIGURE 9.—XRD images of the PMFSF sintered under different sintering temperatures and holding times.

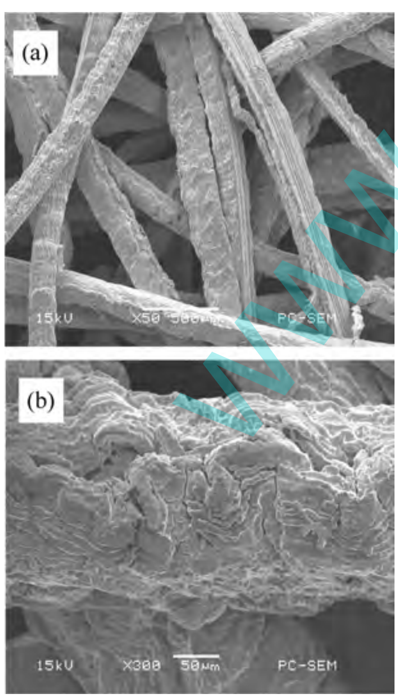

FIGURE 10.—SEM images of (a) the PMFSF (porosity is 80%) and (b) a single copper fiber after sintering at 800°C for 60 min.

poor mechanical properties of the PMFSFs. Figures 10(a) and (b) show the SEM images of the PMFSFs and single copper fiber after sintering at 800°C for 60 min, respectively. When compared to Fig. 7, it was found that both of felts had a good three-dimensional reticulated structure, and the surfaces of both metal fibers retained some microstructures. Accordingly, the specific surface area of the PMFSFs sintered at 800°C for 60 minutes is measured to be 1.792 m^2/g. So the specific surface area varies slightly under different holding times. Moreover, both of the felts had the same phase image shown in Figs. 9(a) and (b), and the main phase components was Cu. These results further validate that the holding time has little effect on the phase components. According to the above results, it is concluded that the sintering temperature has a significant influence on the forming process of the PMFSF, but the holding time do little. On the condition of meeting the application requirement, the most effective way to achieve the forming process of the PMFSF is by increasing the sintering temperature and keeping a short holding time.

CONCLUSIONS

A novel PMFSF with three-dimensional reticulated structure has been produced by the solid-state sintering of copper fibers. The copper fibers, with a lot of micro/nanoscale structures distributed onto the surface, were fabricated using the cutting method. The shape and porosity (60–98%) of the PMFSFs were adjusted using the plate-laminated mold pressing equipment. It was found that the PMFSFs exhibited two kinds of sintering joints between fibers after the completion of sintering process: fiber-to-fiber surface contact and crossing fiber meshing. The PMFSF with three-dimensional reticulated structure was obtained within a temperature range of 800°C–1000°C. In the sintering process, the surface microstructures of the fibers helped to improve the forming process of the PMFSF, as a result of high surface energy. In addition, the sintering temperatures had a significant influence on the surface microstructures of single fiber and the specific surface area of the PMFSFs, but the holding time had little effect. Both the sintering temperature and holding time had little effect on the phase components according to the XRD images. In this study, the PMFSF sintered at 800°C for 30 minutes showed an excellent three-dimensional reticulated structure and higher specific surface area. It is proposed that increasing the sintering temperature and shorting the holding time is the best way to performance the sintering process of the PMFSF. Moreover, we anticipate that the PMFSF will become an excellent candidate as an application in a variety fields (such as catalyst support, wick materials, and gas diffusion layer for fuel cell), due to its unique porous structure and larger specific surface area. Our group is in the process of researching these possibilities.

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