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Sintering formation of oriented linear cutting copper fibers

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Abstract: The formation mechanism of oriented linear cutting copper sintered felt was analyzed. The influences of sintering temperature, sintering atmosphere and sintering time on the sintering formation were investigated. And the tensile mechanical properties of sintering fibers under different sintering conditions were also analyzed. The results indicate that the formation of sintered necks in the contacted area due to the materials migration of microstructures on the surfaces of fibers promotes the tight connection of oriented linear cutting copper fibers. It is also found that both sintering temperature and sintering atmosphere show important effects on the sintering formation while the influence of sintering time is not so obvious. Sintering at 800 °C for 60 min under low temperature reduction can produce sintered necks to make fibers tightly connect together and maintain the coarse microstructure on the surface of fibers, and the best tensile mechanical property can be obtained as well.
Key words: sintering; porosity; fibers; mechanical properties

1 Introduction

With the development of micro-fabrication techniques, microreactors are widely applied in many fields. Nowadays multiple laminated microchannel plates are usually used as catalyst support for microreactors [1−4]. Since the equivalent diameter of microchannel is generally less than 500 μm, it can effectively improve the specific surface area, and thus strengthen the heat and mass transfer process. Moreover, uniform distribution of parallel microchannel helps to achieve uniform flow distribution of the reactive fluid [5−8]. On the other hand, porous metal materials gradually become a new kind of catalyst support for microreactors [9−11] due to their characteristics of three-dimensional network pore structure, large porosity and high specific surface area. Compared with the laminated microchannel plate structure, the porous metal materials can effectively increase the amount of catalyst on the condition of same reaction volume.

Combined the feature of uniform distribution of parallel microchannels with high specific surface area of porous metal materials, a novel kind of oriented linear cutting copper fiber sintered felt was proposed as catalyst support in our pervious works [12,13], as shown in Fig. 1. Firstly, multiple long copper fibers were simultaneously fabricated by a cutting tool with multi-tooth. And then several cutting copper fibers were arranged in parallel direction and sintered together to form a felt with a uniform thickness under the condition of high temperature and sintering atmosphere. Compared with the traditional porous metal materials with a three-dimensional network pore structure, the oriented linear cutting copper fiber sintered felt presented a characteristic of microchannel-like structure. A relatively large specific surface area was also provided for the reason that the coarse pattern formed on the surface of cutting copper fiber.

In this work, the formation mechanism of oriented linear cutting copper sintered fiber was studied based on the analysis of surface structure and morphology of cutting copper fiber as well as sintered felt. And the influences of key sintering parameters on the formation

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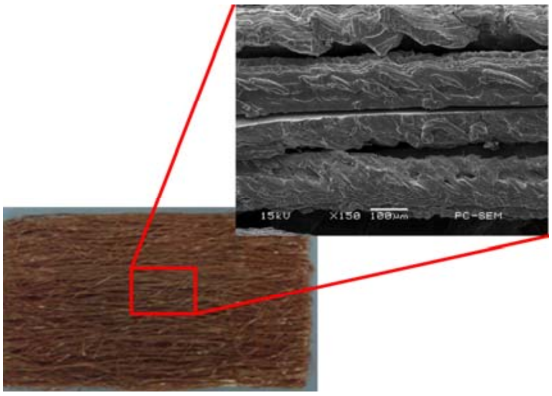

Fig. 1 Oriented linear cutting copper fiber sintered felt and its microchannel-like structure

of oriented linear cutting copper fiber sintered felt were experimentally studied. The tensile mechanical properties of sintered felt were further analyzed.

2 Experimental

The formation of oriented linear cutting copper fiber sintered felt could be divided into two stages: cutting copper fiber by a multi-tooth tool, mold pressing and sintering of oriented linear copper fibers.

2.1 Cutting copper fiber by multi-tooth tool

Firstly, the copper fibers were fabricated from a copper rod on a C6132A lathe by a tool with an installed angle of 45°, as shown in Fig. 2. Multiple teeth were fabricated on the main cutting edge by wire cutting technology, which could achieve the continuous cutting of multiple copper fibers simultaneously. In this experiment, the material of the multi-tooth tool was high-speed steel and the key tool parameters were set as follows: the nominal rake angle γ_0=25°−30°, the nominal clearance angle α_0=5°−8°, the tool cutting edge inclination s=45°−55°, the tooth space m=0.13 mm and the tooth depth h=0.12 mm. The copper rod was d41.90 mm × 400 mm in dimension and the cutting condition was dry. During the cutting process, feed speed, cutting depth, and cutting speed were set to be 0.08 mm/r, 0.25 mm and 180 r/min, respectively.

2.2 Mold pressing and sintering of oriented linear copper fibers

The collected cutting copper fibers were firstly annealed at 450 °C in a box-type furnace (No.RXL−12−11) for 30 min to remove the residual stress, which could effectively improve the toughness and avoid fracture in the orientation process of fibers. And then the copper fibers were arranged in parallel around a thin plate, of which the dimension matched

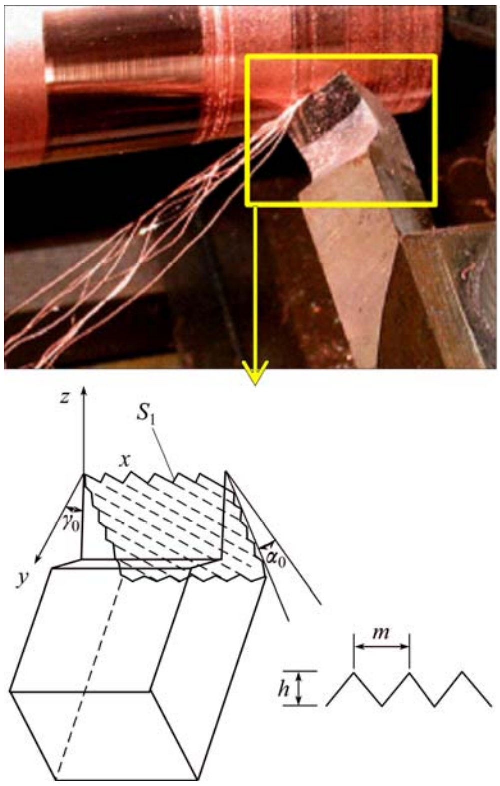

Fig. 2 Cutting copper fiber by multi-tooth tool

with the chamber for sintering. The shaped fibers were put into the chamber of a laminated-plate mold pressing equipment, which consisted of two cover plates and several thin hollow-chamber plates, as shown in Fig. 3. The thickness of sintered felt could be changed by adjusting the number of hollow-chamber plates. By this way, the same size of sintered felt as the chamber could be obtained.

After being tightened with four bolts, the mold pressing equipment was placed into the same furnace for sintering at 600−900 °C for 30−60 min under a pressure of 0.3−0.4 MPa and sintering atmosphere. The heating-up process was divided into two steps: a heating-up speed of 300 °C/h was used when the temperature was below 800 °C, whereas it was heated by 200 °C/h when the temperature was above 800 °C. After sintering, the mold pressing equipment was cooled down to 150 °C in the furnace and then taken out for natural cooling to room temperature. As a result, the sample of oriented linear cutting copper fiber sintered felt could be obtained.

The dimensions of the oriented linear cutting copper fiber sintered felt in this experiment were 60 mm × 20 mm × 2 mm. The porosity could be calculated by the Quality−Volume method as defined below:

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

where V and m are the volume and mass of the sintered

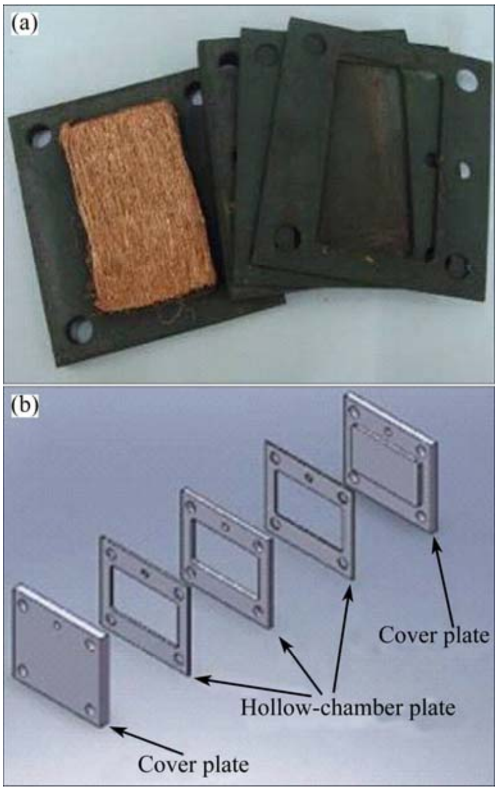

Fig. 3 Physical map (a) and principle (b) of laminated-plate mold pressing equipment

felt, respectively; ρ represents the copper density.

The sintering formation of oriented linear cutting copper fibers was influenced by many factors, such as sintering temperature, sintering time and sintering atmosphere. In our pervious works [14,15], the formation of sintered felts with three-dimensional network pore structure had been investigated. In this work, the influences of these key factors on the sintering formation of oriented linear cutting copper fibers were studied. Four sintering temperatures (600, 700, 800 and 900 °C) and two sintering time (30 and 60 min) were considered in the experiment. Three kinds of the sintering atmospheres were investigated here, which were complete reduction, high temperature for reduction and low temperature for reduction. The so-called high temperature reduction, nitrogen as the protective atmosphere was firstly fed into the furnace to empty the air until the furnace temperature reached 600 °C, and then the hydrogen was input for reduction. The difference between the high temperature reduction and low temperature reduction was the heating-up temperature for feeding into the hydrogen, which was 400 °C for the low temperature.

After the formation of the oriented linear cutting copper fiber sintered felt, an SANS microcomputer control universal material testing machine was used to detect the intensity of sintered felt. The loading error of this machine was no more than 1%. When mounting on the machine, two rubber gaskets were clamped at two sides of the sintered felts to avoid the structure damage and falling off during the stretched process. Therefore, the effective length of the testing sintered felt after clamping with gaskets was about 30 mm. The stretched speed of upper chuck was set to be 3 mm/min. In this work, sintering temperature, sintering time and sintering atmosphere as three key factors influencing the sintering formation were also used to study the effects on the tensile mechanical property of oriented linear cutting copper fiber sintered felt.

3 Results and discussion

3.1Formation mechanism of oriented linear cutting copper fiber sintered felt

Figure 4 shows the comparison of the surface morphology between the cutting copper fiber and drawing copper fiber. It was indicated that the cross section of the cutting copper fiber was approximately rectangular or triangular. Compared with the smooth surface of the traditional drawing copper fiber, the surface microstructure of cutting copper fiber was exceptionally rough, which effectively increased the specific surface area. And another surface of the cutting fiber was somewhat smooth. This was because intensive plastic deformation occurred on the surface of fibers due to the interaction of extrusion and friction between the tool and copper rod during the cutting process, and resulted in the production of coarse microstructure morphology on the fiber surface. The rough surface of cutting fiber could promote the formation of the sintered neck, which made the cutting fibers connect with each other tightly. Moreover, when the sintered felt was used as the catalyst support, the rough microstructure on the surface of fiber could prevent the catalyst falling off during reaction, which was beneficial to increasing the stability of the catalytic reaction.

These coarse microstructures were essentially composed of lots of micro or nano particles. Some particles on the surface of fibers contacted with each other when the oriented fibers were pressed together in the mold equipment. However, there were still lots of pores among the particles on the surface of fibers. The surface tension of the particles resulted in a certain pressure difference (Δp) as defined as

$$\Delta p=2\gamma/r \tag{2}$$

where γ and r represent the surface tension and the spherical radius of particles, respectively. If the particles were not spherical, the pressure difference could be calculated by two radii of curvature r_1 and r_2:

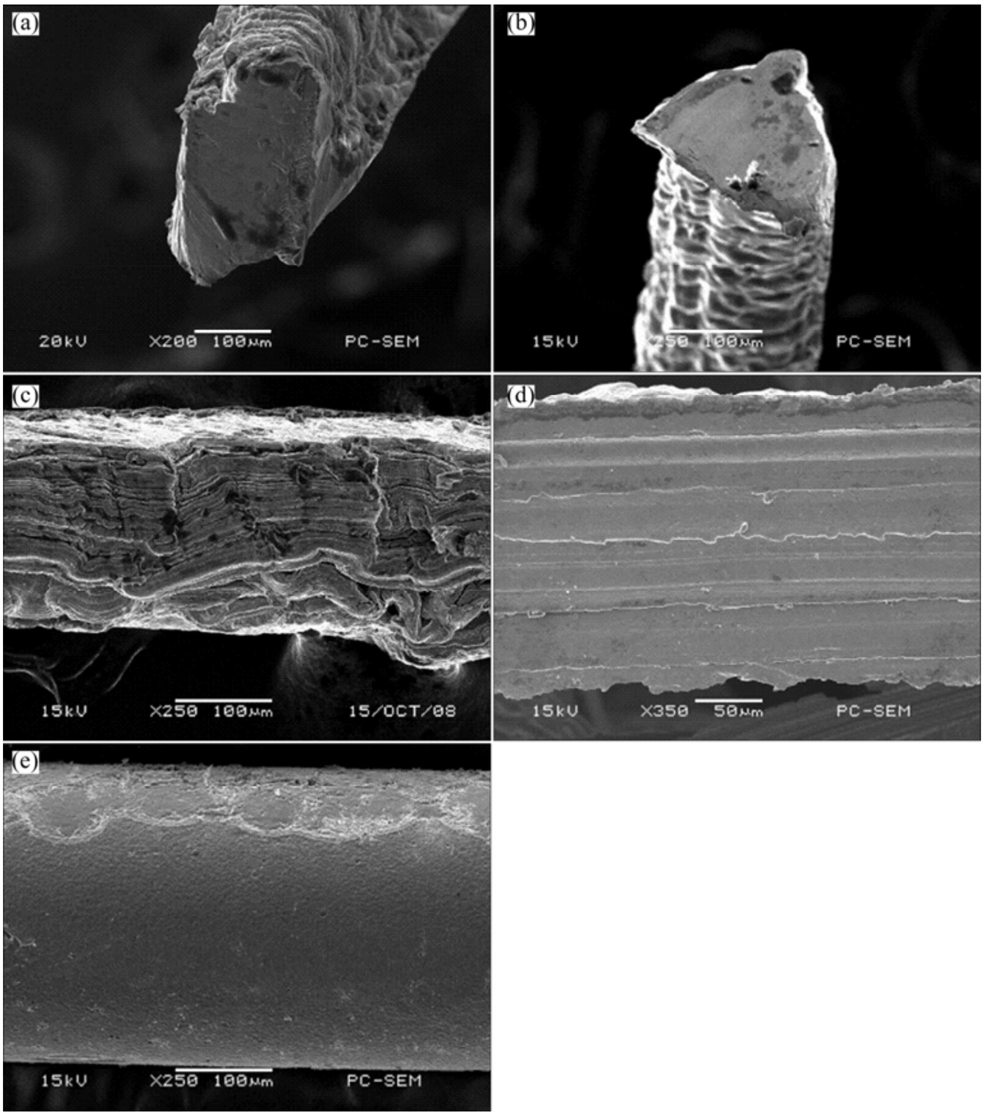

Fig. 4 Comparison of surface microstructure morphologies between cutting copper fiber and drawing copper fiber: (a) Rectangle-like cross section; (b) Triangle-like cross section; (c) Free face of cutting fiber; (d) Smooth face of cutting fiber; (e) Surface morphology of drawing fiber

$$\Delta p=\gamma(1/r_1+1/r_2) \quad (3)$$

It was indicated that the pressure difference was inversely proportional to the radius of curvature, while directly proportional to the surface tension of particles. That was to say, the smaller the particles were, the greater the driving force for sintering was. It was exactly the driving energy that promoted the material migration of the particles. The severe relative motion of particles provided a great amount of surface energy. During the sintering process, materials spontaneously migrated to the lowest point of energy. By this way, a large amount of surface energy converted into the driving energy, and materials migrations of the microstructures in the contacted area among the oriented linear fibers occurred, which led to the formation of sintered necks as shown in Fig. 5. Gradually, more sintered necks were produced in the contacted area so that the oriented linear fibers could be tightly connected and the sintered felt with a certain

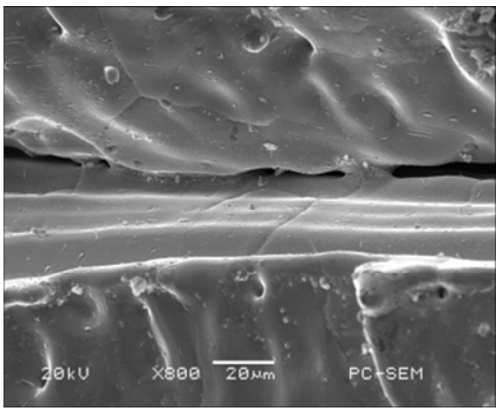

Fig. 5 Formation of sintered necks between oriented linear cutting copper fibers

tensile strength was finally obtained. Smaller coarse microstructure and higher specific surface area were beneficial to larger surface energy and driving energy, and the larger metallurgical bonding of oriented linear cutting copper fibers could be obtained.

On the other hand, since the copper fibers were regularly arranged in parallel, microchannels as flow passages were formed among the parallel cutting copper fibers. Unlike the traditional smooth microchannels patterned in the metal plate, a great amount of coarse antler microstructure morphology was generated on the surface of microchannels, as shown in Fig. 6. These coarse antler microstructures could increase the residence time when the reactant flowed through the microchannels, and hence the mutual effect between the reactant and the catalyst was effectively strengthened. Therefore, the catalytic reaction could be promoted. In our previous work [9], the oriented linear cutting copper fiber sintered felt was used as the catalyst support for methanol steam reforming. This indicated that the oriented linear cutting copper fiber sintered felt presented better performances of methanol steam reforming than the oriented linear copper wire sintered felt under the condition of low GHSV or reaction temperature.

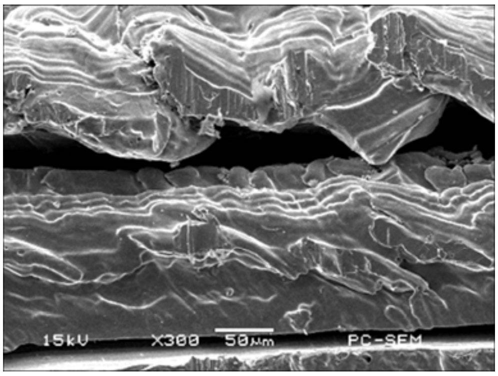

Fig. 6 Formation of microchannels inside oriented linear cutting copper fiber sintered felt

3.2 Key factors influencing sintering formation of oriented linear cutting copper fibers

3.2.1 Influences of sintering temperature

The sintering temperature was one of the most important sintering parameters for the sintering formation of oriented linear cutting fibers. The oriented linear cutting copper fibers were hard to tightly connect with each other at relatively low temperature, whereas the sintered neck among the oriented linear cutting copper fibers could be damaged at high temperature. What was more serious was that a part of copper fibers might even fuse and the particles had the tendency to grow up, which resulted in the decrease of the bending strength of sintered felt.

Figure 7 shows the SEM images of oriented cutting copper fiber sintered felts treated for 30 min at different sintering temperatures. The porosities of all the sintered felts were 80%. It was obvious that the cutting copper fibers were connected together by sintered necks and maintained a microchannel-like structure. As shown in Fig. 7(a), there were no obvious sintered necks formed among the oriented linear cutting copper fibers at the sintering temperature of 600 °C. And several tiny holes were produced on the surface of the cutting copper fibers, as shown in Fig. 7(b). The main reason was that some parts of fibers were oxidized during the cutting process due to the high cutting temperature, and the oxygen atoms inside the oxidized parts of fibers and hydrogen atoms inside the sintering atmosphere occurred and the water as the product was escaped from the surface of copper fibers at certain temperature and pressure.

When the sintering temperature was increased to 700 °C, the coarse microstructures on the surface of copper fibers were somewhat improved but still not ideal, as shown in Figs. 7(c) and (d). It was obvious that several sintered necks formed and relatively good sintering formation was obtained at a sintering temperature of 800 °C, as shown in Fig. 7(e). At the same time, the coarse microstructure on the surface of copper fibers was well preserved, as shown in Fig. 7(f). When the temperature reached 900 °C, as indicated in Figs. 7(g) and (h), the surface of the copper fibers became somewhat smooth and the coarse microstructures on the surface of fibers gradually disappeared, which led to the decrease of the specific surface area.

Consequently, the formation of oriented linear cutting copper fibers sintered felt treated at 800 °C, could not only produce sintered necks to make fibers tightly connect together, but also produce microchannel-like structures. Moreover, coarse microstructure was maintained on the surface of fibers. The increase of sintering temperature baffled the growth of sintered necks and even made them disappear at last.

3.2.2 Influences of sintering atmosphere

Figure 8 presents the influences of sintering atmosphere on the formation of oriented linear cutting copper fiber sintered felt treated at 800 °C for 30 min. From the view of Figs. 8(a) and (b), as for the high temperature reduction, the surface energy hardly played a role when the temperature was higher than 600 °C. Therefore, the result of high temperature reduction was not ideal as well. The cutting copper fiber still maintained the rough microstructure morphology under low temperature reduction atmosphere, as shown in Figs. 8(c) and (d). It can be seen that the substitution of

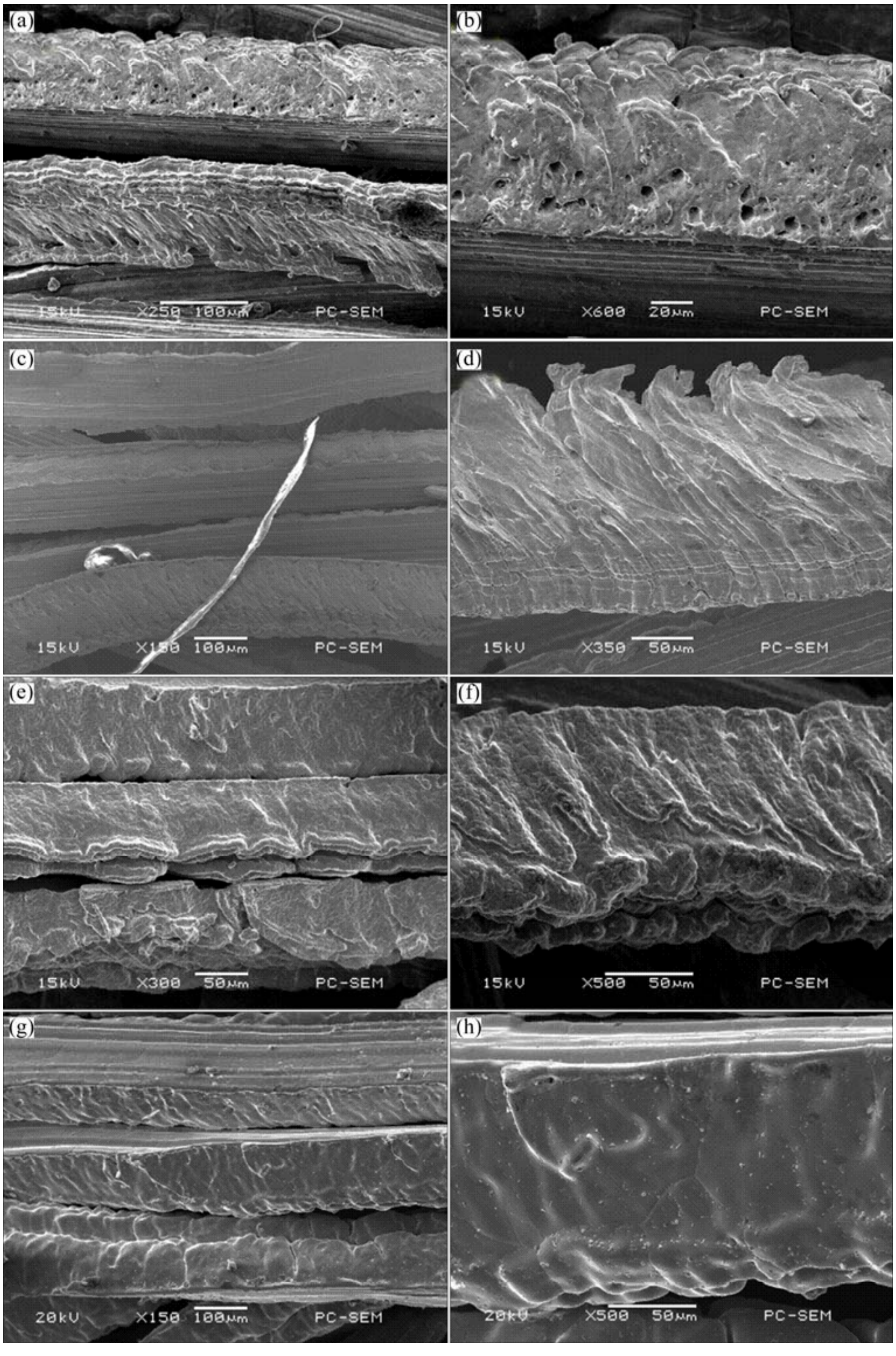

Fig. 7 Formation of oriented linear cutting copper fiber felts at different sintering temperatures: (a, b) 600 °C; (c, d) 700 °C; (e, f) 800 °C; (g, h) 900 °C

nitrogen with hydrogen at a temperature of 400 °C was beneficial to the formation of sintered neck and preservation of the rough microstructure morphology of cutting fibers. Figures 8(e) and (f) indicated that the sintered felt obtained under complete reduction atmosphere had almost the same rough microstructure morphology with that under low temperature reduction atmosphere. However, only hydrogen was used for complete reduction in the whole sintering process, it was somewhat dangerous compared with the low temperature reduction atmosphere. Consequently, it was more ideal to sinter the oriented linear cutting copper fibers under low temperature reduction atmosphere on the condition of taking the safety into consideration.

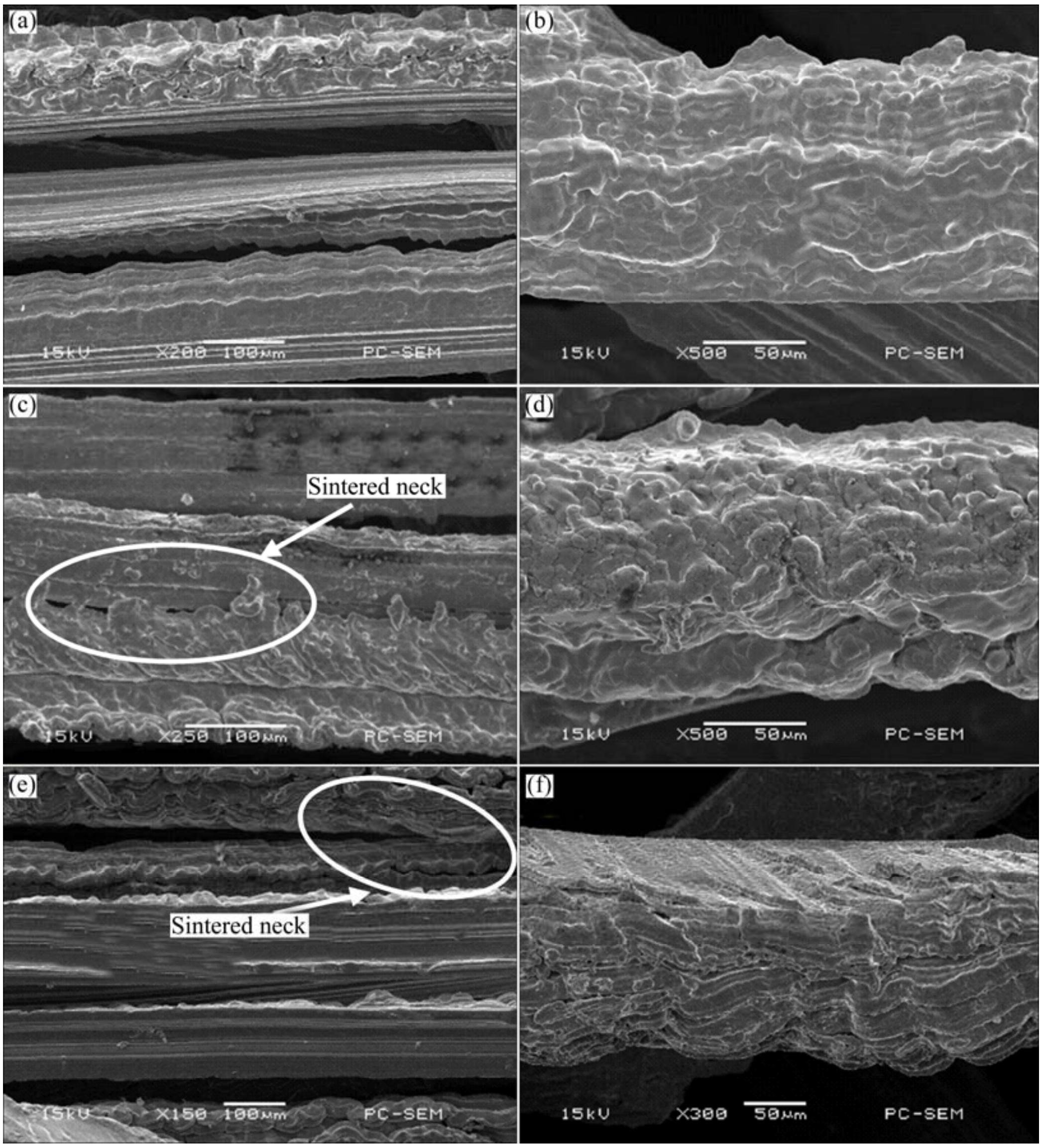

Fig. 8 Formation of oriented linear cutting copper fiber sintered felt under different sintering atmospheres: (a, b) High temperature reduction; (c, d) Low temperature reduction; (e, f) Complete reduction

3.2.3 Influences of sintering time

Sintering time was another factor influencing the sintering formation of oriented linear cutting copper fibers. In general, the proper extension of sintering time could effectively promote the diffusion and transfer of atoms on the surface of fibers, which was in favor of the formation and growth up of sintered necks and made the oriented linear cutting copper fibers tightly connect with each other. However, the recrystallization would be intensified under long sintering time, and then the grains would become bulky, which might weaken the mechanical properties of sintered felt. Figure 9 shows the SEM images of the sintered felts treated at 750 °C for 30 min and 60 min, respectively. It was found that there was almost no change of the microstructures on the surface of fibers.

3.3 Tensile mechanical property of oriented linear cutting copper fiber sintered felt

Figure 10 shows the unilateral tensile process of the oriented linear cutting copper fiber sintered felt with a porosity of 80%, which was sintered at 750 °C for 60 min. At the beginning of the tensile process, plastic deformation of the oriented linear cutting copper fiber sintered felt as a whole metal frame firstly occurred. With the increase of the load, plastic deflection of the connected points and mechanical connections among the fibers appeared. And then a necking phenomenon appeared as the load increased to a certain value, as indicated in Fig. 10(a). When the stress was increased to the limit stress, the connected points among the copper fibers fractured so that the skeleton structure of the sintered felt was destroyed rapidly, as shown in

Fig. 10(b). The angle between the directions of fracture and the tensile load was about 45°. And the direction of fracture was consistent with that of the maximum shear stress.

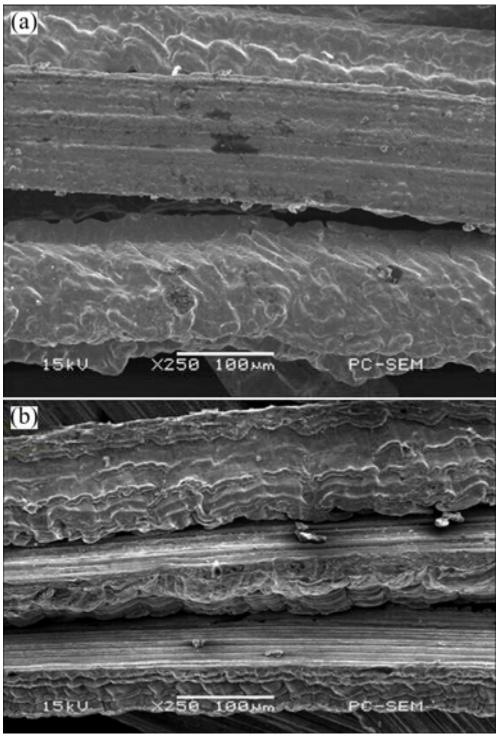

Fig. 9 SEM images of oriented linear cutting copper fiber sintered felts treated at 750 °C for different sintering time: (a) 30 min; (b) 60 min

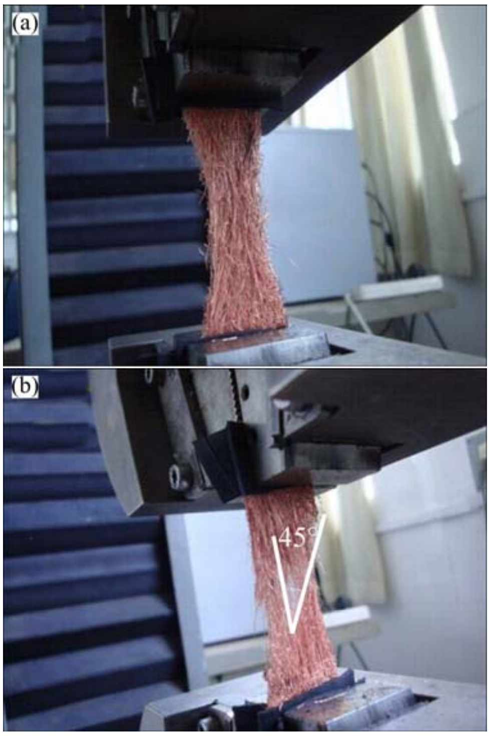

Fig. 10 Tensile process of oriented linear cutting copper fiber sintered felt: (a) Necking; (b) Tensile fracture

As mentioned above, the tensile fracture of the oriented linear cutting copper sintered felt was similar to that of general plastic materials, but there still existed some characteristics as well. From the macroscopic view, the sintered felt was a metal skeleton composed of lots of oriented linear cutting copper fibers. Therefore, similar fracture morphology with that of traditional plastic materials in tensile fracture process appeared. Due to the oriented arrangement of the copper fibers, the strain could transfer in the skeleton structure of sintered felt with the increased stress, and then the necking occurred. Once the internal stress reached the limit value, the sintered felt would fracture rapidly.

3.3.1 Influences of sintering temperature

Figure 11 shows the tension force−displacement curves of oriented linear cutting copper fiber sintered felts with a porosity of 80% sintered at different temperatures for 60 min. From the horizontal ordinate, it could be found that fracture of the sintered felt whose sintering temperature was 900 °C firstly occurred. The main reason was that the microstructure on the surface of cutting copper fibers almost disappeared at relatively high temperature, and hence the sintered necks diminished, which led to the decrease of the tensile strength. On the other hand, the sintered felt whose sintering temperature was 800 °C had the maximum tension. As analyzed above, the sintered necks were produced to make fibers tightly connect together at a sintering temperature of 800 °C, and hence improved the tensile mechanical property.

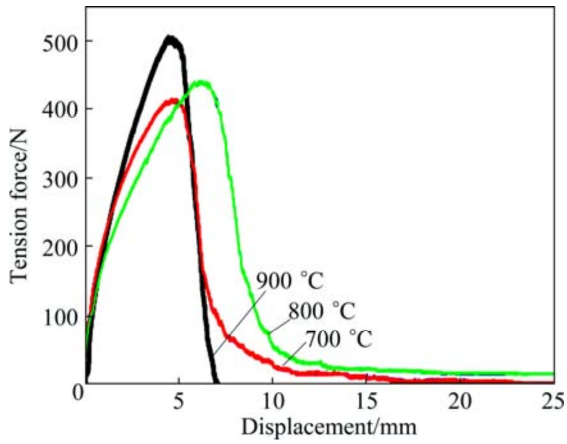

Fig. 11 Influences of sintering temperature on tensile mechanical property of oriented linear cutting copper fiber sintered felt

3.3.2 Influences of sintering atmosphere

Figure 12 shows the displacement of sintered felts with a porosity of 80% treated at 800 °C for 60 min under two kinds of sintering atmospheres as a function of tension load. It can be seen that the fracture displacement of sintered felt formed under low temperature reduction was larger that zero, which indicated that the sintered felt did not completely fracture on the given load. That was to say, the sintered felt had the best tensile mechanical property under low temperature reduction. The result was also consistent with the above one.

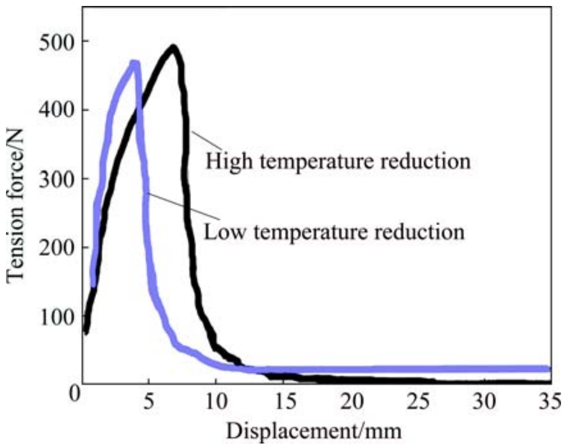

Fig. 12 Influences of sintering atmosphere on tensile mechanical property of oriented linear cutting copper fiber sintered felt

3.3.3 Influences of sintering time

Figure 13 presents the comparison of tensile mechanical property of sintered felts treated at 800 °C for 30 min and 60 min, respectively. It was obvious that the fracture displacements of two sintered felts were close to each other. But the maximum load of sintered felt treated for 60 min was a little larger than the one treated for 30 min. The main reason was that the longer sintering time could effectively promote the formation of sintered neck. As a result, the connections among the copper fibers became tighter, which improved the tensile strength of oriented linear cutting copper fiber sintered felt.

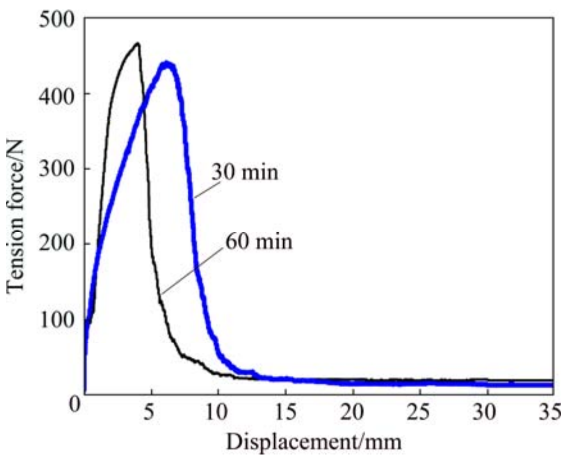

Fig. 13 Influence of sintering time on tensile mechanical property of oriented linear cutting copper fiber sintered felt

4 Conclusions

1) During the sintered process, materials migration of the microstructures in the contacted area among the oriented fibers occurred under the effects of driving energy, which led to the formation of sintered necks. Gradually, more sintered necks were produced in the contacted area so that the oriented linear fibers could be tightly connected and the sintered felt was finally obtained.

2) Both sintering temperature and sintering atmosphere showed important effects on the formation of oriented linear cutting copper fiber sintered felt while the influence of sintering time on the sintering formation was not so obvious. As for the effect of sintering atmosphere, the low temperature reduction and complete reduction were not only beneficial to the formation of sintered neck, but also maintain the rough microstructure morphology on the surface of cutting copper fibers.

3) The tensile mechanical properties of oriented linear cutting copper fiber sintered felts treated under different sintering conditions were consistent with the analysis of the influences of sintering temperature, sintering atmosphere and sintering time on the sintering formation of oriented linear cutting copper fibers. The oriented linear cutting copper fiber sintered felt treated at 800 °C for 60 min under low temperature reduction had the best tensile mechanical property.

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定向切削铜纤维烧结成形

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摘　要：分析定向切削铜纤维烧结载体的成形机理，研究烧结温度、烧结氛围和烧结时间对烧结成形的影响，并且分析不同烧结条件下烧结纤维的拉伸力学性能。结果表明，烧结温度和烧结氛围对烧结成形具有重要影响，而烧结时间的影响不是特别显著。在低温还原氛围和 800 °C 下烧结 60 min，可以形成烧结颈使得纤维之间紧紧连结在一起，并且在纤维表面形成粗糙的微结构，同时可以获得最佳的拉伸力学性能。

关键词：烧结；孔隙率；纤维；力学性能

(Edited by Xiang-qun LI)