Experimental study of hot pressed sintered modified materials

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Abstract. The PTFE/Cu composite powder was selected as the experimental raw material, and the modified material PTFE/Cu with dense structure was prepared by hot pressing sintering process. The morphology of the product before and after the test was compared and observed by SEM. The effect of different material ratios on the densification results of the powder was studied, and the difference between molding and hot pressing sintering was analyzed. The results show that hot-pressed sintering can produce modified PTFE/Cu with a density of up to 99.9%, which is higher than that obtained by molding under the same pressure. The SEM image shows that, unlike the molding, hot-pressing sintering makes the crystal grains of the two materials exist independently in the modified polymer, and makes the powder crystal grains gradually perpendicular to the hot pressing direction.

1. Introduction

As the forefront of the tandem warhead, the dynamic impact mechanical property of pure PTFE is weak, and the jet penetration property is insufficient. In order to improve its mechanical properties and penetration properties, PTFE is filled with modified materials. Maintaining the advantages of the original material and integrating the composite effect of the filler material, part of the current research is to add Al to PTFE. Due to the characteristics of Al, the modified energetic materials are researched into its physical and chemical properties, thermal decomposition properties, and mechanical properties, to determine its constitutive relationship [1-6]. By changing the sintering and cooling process, the effect of the process system on the geometric deformation and mechanical properties of the PTFE/Al reaction material [7] was studied. Meanwhile, the mechanical properties of PTFE/Cu were studied, and the effect of Cu mass ratio on the stress-strain relationship of the modified materials was analyzed.

Based on the above research, this paper selects composite powder PTFE/Cu and conducts hot pressing sintering experiments according to three mass ratios. The effects of temperature field and pressure field on material forming were studied, the differences between the two forming methods on sample densification were analyzed, and the microstructure of the samples obtained by hot pressing sintering and compression forming were compared and analyzed.
2. Materials and methods

The experimental materials were composite powders composed of PTFE powder and Cu powder. They were filtered before weighing, and the agglomerated powder was removed first. In order to obtain samples of different densities, the composite powder was weighed according to three mass ratios weight, as shown in table 1. The weighed powder was poured into a V-type mixer for mixing. The mixing time was 45 minutes, and the rotation speed was 24 r/min.

| Material | Density/$\text{g cm}^{-3}$ | Size/$\mu\text{m}$ | Melting Point/$^\circ\text{C}$ | Thermal Conductivity/$\text{W} \cdot (\text{m} \cdot \text{K})^{-1}$ |
|----------|-----------------|-----------------|-----------------|------------------|
| PTFE     | 2.13-2.17       | 170             | 327             | 0.256            |
| Cu       | 8.96            | 75              | 1083            | 386.4            |

The mixed powder was filled into a mold which was made of high-strength graphite. In order to avoid adhesion during hot-press sintering and inconvenience during demolding, a layer of graphite paper was added between the powder and the mold for insulation, as shown in figure 1.

![Figure 1. Powder filling.](image1)

The equipment used in this experiment was a vacuum hot-pressing sintering furnace, as shown in figure 2. The main technical parameters were: rated power is 40KW, rated voltage is 380V, ultimate vacuum degree is $6.67 \times 10^{-3}$, working area size is $\phi 160 \times 160$mm, maximum temperature is 2000 $^\circ\text{C}$, the maximum sintering pressure is 20T. This equipment can monitor the temperature and pressure in real time, and can adjust the speed at which the temperature and pressure changed.

![Figure 2. Hot-pressing sintering equipment.](image2)

During hot pressing sintering, the changes of process parameters have a great impact on powder molding. With reference to previous studies, this experiment adopted a process system with variable...
temperature heating, uniform temperature cooling, and constant pressure applied at the beginning of the experiment. The setting of process parameters as shown in figure 3.

![Sintering Process Parameter Settings](image)

**Figure 3.** Sintering process parameter settings.

According to the characteristics of the powder material, during the entire hot-pressing sintering process, the constant pressure applied was 42 MPa, and the sintering temperature was 300 °C. At the first stage, the temperature was raised from room temperature to 210°C when heating up, while the time required was 210min. Then at the second stage, the temperature was raised from 210°C to 300°C and took 210min. At this stage, the powder was in the key process of densification. An excessively fast heating rate can easily cause the powder to unevenly densify and to generate more pores, so the heating rate of the second stage is lower than that of the first stage. After reaching the sintering temperature, it was held for 60 minutes. The proper holding time can ensure that the crystal grains have sufficient time to grow, make the pores smaller, and promote the densification of the powder. Finally, it was cooled down to 60°C at a slow rate and then cooled with the furnace.

3. Result

3.1. Densification analysis

Three groups of powders with different ratios were hot-pressed and sintered under the same process system to obtain three different groups of samples. Figure 4 shows the molding samples at a certain ratio. The shape is consistent with the mold, and there aren’t any depressions on the surface.

![Hot-pressed Sintered Samples](image)

**Figure 4.** Hot-pressed sintered samples.

The Archimedes drainage method was used to measure the density of the sample, and the density of
the sample obtained by compression molding under the same pressure was compared. The results were shown in table 2.

**Table 2. Density table for compression molding and hot pressing sintering.**

| Mass Ratio  | Theoretical density/g cm\(^{-3}\) | Experimental density/g cm\(^{-3}\) | Densification |
|------------|----------------------------------|-----------------------------------|---------------|
|            | (Compression molding/Hot pressing sintering) | (Compression molding/Hot pressing sintering) |
| PTFE:Cu=82:18 | 2.5                              | 2.46/2.497                        | 98.4/99.9   |
| PTFE:Cu=63:37 | 3.0                              | 2.763/2.84                        | 92.1/94.7   |
| PTFE:Cu=49.5:50.5 | 3.5                             | 3.2/3.31                          | 91.4/94.5   |

As can be seen from the table, the density of the samples obtained by hot pressing sintering is greater than the density of the compression molded samples, and the density of both samples is getting lower and lower as the Cu content increases. The sintering temperature is much lower than the melting point of Cu. Therefore, in the entire hot-pressing sintering process, the two materials belong to the solid phase reaction. PTFE is a thermal expansion material, the crystal grains will be enlarged when it was heated, and the pores will be discharged to achieve densification. In other words, during the entire hot-pressing sintering process, there is no mutual reaction between PTFE and Cu. Only at high temperatures, PTFE and Cu adhere to each other to produce plastic flow. Under pressure, the particles break up and rearrange, and the grain boundary sliding gradually began to densify.

3.2. Morphology analysis
The hot-pressed sintered sample was sanded with water-abrasive paper to remove the surface graphite paper. A certain size of the sample was cut and polished, and then observed under a scanning electron microscope. Figure 5 shows the low magnification and high magnification morphology of the composite powder, the compression-molded sample, and the hot-pressed sintered sample, respectively.

(a) Low power morphology of composite powder.  (b) Low power morphology of compression moulding sample.  (c) Low power morphology of hot pressing sintering sample.
As can be seen from figures (a) and (d), the mixed composite powders are loosely arranged, of which the white is Cu and the black is PTFE. The two particles are adsorbed to each other. Cu is in a bundle shape, PTFE exists in a two-dimensional state and has a shape similar to a dimple. Compression molding simply reduces the gap between the particles by applying external force to achieve the molding requirements. This process requires higher molding pressure. As shown in figures (b) and (e), the molded PTFE / Cu has many cracks, and there is no plastic flow between PTFE and Cu. Only pressure causes particle rearrangement and shrinks the pores, but there are many cracks on the surface after molding, which results in poor mechanical properties of the sample obtained by molding. As can be seen from figures (c) and (f), after hot pressing sintering, the PTFE and Cu changed the original grain arrangement direction. The flaky grains gradually became perpendicular to the hot pressing direction, so that the samples obtained by hot pressing sintering have increasingly stronger structure and improved mechanical properties.

4. Conclusion
The heat radiation and heat conduction coexist in the hot pressing sintering process. The heat radiation exists between the heating element and the mold, and heat conduction exists between the mold and the powder. The hot pressing sintering process proposed in this paper can produce modifications without defects inside and outside. The material has a maximum density of 99.9%. Moulding makes PTFE and Cu adhere to each other, and hot-pressing sintering also causes the two particles to be independently distributed due to the presence of temperature, and the crystal grains are gradually perpendicular to the hot-pressing direction, resulting in a change of the properties of the modified material. Under the same pressure, the density of the sample obtained by molding is higher than that of hot-pressing sintering. Due to the characteristics of the matrix, the density of the sample gradually decreases with the increase of the content of the metal powder. Based on the existing results, it is necessary to try to increase the pressure appropriately to make the sample density close to the theoretical value in follow-up studies.

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