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Study on the properties of PPS composite modified with Tungsten powder

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Abstract: The tungsten powder (W) and polyphenylene sulfide (PPS) as the main raw material, through mixing, extrusion molding and extrusion molding method contrast, determines the extrusion method for the preparation of tungsten powder/PPS (W/PPS) composites, was studied by DSC, XRD and SEM after modification, the W powder for W/PPS material microstructure and the influence of thermal performance. W was mixed evenly in PPS, and the thermal decomposition temperature of PPS was increased.

1. Introduction

PPS is a new kind of functional engineering plastics, it has a very good corrosion resistance, high temperature resistant, radiation resistant and flame retardant. Because of its high mechanical properties and excellent dimensional stability and excellent characteristics, it is widely used as structural high polymer material and special engineering plastics [1]. At the same time, PPS can also be made into a variety of functional film, coating and composite materials, in aerospace, electronics, electrical, automotive transportation and other fields have been successfully applied. Although the melting temperature of PPS is high, the melt viscosity is low and the fluidity is good. PPS can be used by thermoplastic processing methods such as extrusion, injection, pressing, etc. At high temperature, the mechanical properties of PPS decrease little and have good creep resistance and fatigue resistance. However, the brittleness, toughness and impact strength of pure PPS products are poor. PPS molding and processing on mold temperature (130-150 °C), high demand in the molten viscosity is not stable, easy oxidation at a high temperature as crosslinking in the air, and prices high, limiting the use of pure PPS. In order to improve the toughness of PPs, blending modification is usually used. The main blending systems are: (1) PPs blends with amorphous engineering plastics [2,3]; (2) PPs was blended with general crystalline polymer [4]; (3) PPs is blended with crystalline engineering plastics [5,6]. However, these traditional blending modification methods often sacrifice the comprehensive properties of materials to improve the target properties.

Tungsten has a high subatomic number and high density, and the shielding effect of gamma rays is very good, especially the shielding ability of gamma rays is much higher than that of lead [7]. In addition, tungsten material does not produce secondary electron radiation, so it is an ideal radiation-proof material that can replace lead [8]. However, tungsten is hard, brittle and difficult to process, which limits its application. Currently, Yunbo Li [9,10] et al. have studied nylon PA6 / tungsten powder and PA12 / W shielding composites, but the low hydrogen content of nylon is unfavorable to the neutron shielding



performance of the composites. In addition, the density of nylon is higher than that of polypropylene, which is not conducive to reducing the quality and volume of the shielding system [11].

So far, less study on W/PPS composites at home and abroad. In this paper, we carried out a modification of the tungsten powder added in the PPS, investigated the properties of W/PPS. In this study, W as rigid particles and functional elements distribution in the PPS, W/PPS blend the characteristics of tungsten is a good way to solve the difficult processing, at the same time, qualitative light, excellent comprehensive shielding effect and heat resistance, W/PPS has a good mechanical property, is a kind of friendly new radiation shielding material. It provides a new idea for the application of PPS.

2. Experimental section

2.1. Raw material

Tungsten powder: 7 μm , Xiamen Jinlu special alloy co., LTD., black;

Powder polyphenylene sulfide resin: injection grade, white;

Pentaerythritol ester: PM641951;

Ethylenebistearamide: Q/CNGERUVB;

Silicone lubricant: 791;

Silane coupling agent: A151 north China experimental products mall.

Pentaerythritol ester is an antioxidant, which can delay or inhibit the process of polymer oxidation. Ethylenebistearamide is a dispersant, which can adjust the viscosity of hot melt adhesive, improve fluidity, increase hardness and brightness. Silicone lubricants are lubricants. Silicone lubricants can improve the fluidity of PPS and play three roles: internal lubrication can improve the crosslinking state of PPS molecular chain and facilitate the activity of PPS molecular chain. The chemical reaction between the lubricant and the PPS can improve the fluidity of the PPS, reduce the friction between the tungsten powder particles of the filler and the PPS and the die opening. Silane coupling agent was used as a coupling agent to improve the dispersion and adhesion of the resin.

2.2. Equipment and instruments

The main experimental equipment used in this experiment is shown in table 1.

Table 1 experimental equipment and instruments

Name	Type	Quantity/Tai
FS-400D High-speed disperser	FS-400D	1
Electric sand bath pan	Constant DK - type 2	1
Blast drying oven	CS101-III	1
Twin screw extruder	CTE-35	1
High-speed mixer	GH19-DY	1
Magnetic stirrer with constant temperature and heat	DF-101S	1
Vacuum atmosphere pressure furnace	R-C-ZKQY-07	1
X-ray diffractometer	German Bruker D8advance type	1
Scanning electron microscope	Hitachi S4800	1
Differential scanning calorimeter	METTLER TOLEDO	1
9050 cantilever beam impact tester	INSTRON 5567 INSTRON	
Universal material testing machine	INSTRON CEAST	

2.3. Sample preparation

Due to the difficulty in tungsten powder processing, three schemes were used to prepare W/PPS composites. 1) simple melting blending; 2) hot-pressing molding; 3) twin screw extrusion, injection molding. When simply fused blending, tungsten powder and polyphenylene sulfide are not easy to form and take a long time, which is not conducive to processing and application. The process of hot press

forming experiment is complex, and the temperature is not easy to control, easy to produce high-temperature viscous flow phenomenon or low temperature is not easy to form phenomenon. Both processes are not suitable for processing applications, twin screw extrusion injection molding operation is simple, stable temperature setting. Therefore, samples were prepared by this process.

The W/PPS was prepared by twin - screw extruder. The machine is started up and heated to the set temperature. After the temperature in each zone is stabilized, PPS pure material is used as the washing material to wash the machine. After washing, the mixed materials used in the experiment were added into the feeding bucket and extruded into shape. Choose the best molding conditions under extrusion, and coolinBg at room temperature. The specific process is shown in figure 1.

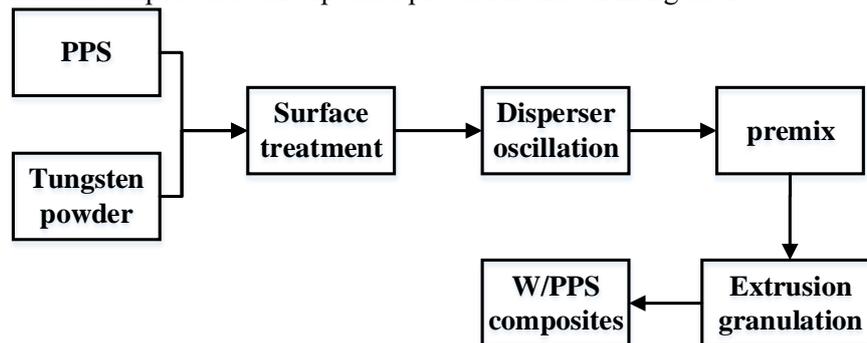


Figure. 1 Preparation process of tungsten powder modified PPS

3. Results and discussion

3.1. SEM analysis

The surface morphology of W/PPS is shown in figure 2. The effect of microstructure on the properties of composites is very important. For PPS/ tungsten composites, the microstructure of the material may be very different for the same material ratio, leading to a great degree of difference in performance. The distribution of tungsten powder in PPS matrix directly affects the thermal properties, mechanical properties and X-ray shielding properties of the composites. Figure 2a is a scanning electron microscope image with a magnification of 4000. It can be seen from the figure that tungsten powder is uniformly dispersed in polyphenylene sulfide resin. During the plasticization of PPS and metal tungsten powder, solid tungsten powder is continuously wrapped and carried by the PPS in viscous flow state, and finally the filler tungsten powder is uniformly embedded in the solidified matrix PPS. FIG. 2b shows the scanning electron microscope image with a high magnification of 15000. It can be seen from the figure that when PPS/ tungsten powder composite sample is hot-pressed, the PPS with viscous flow state fill the gap between the tungsten powder particles while covering the metal tungsten powder particles, and the filling effect is good.

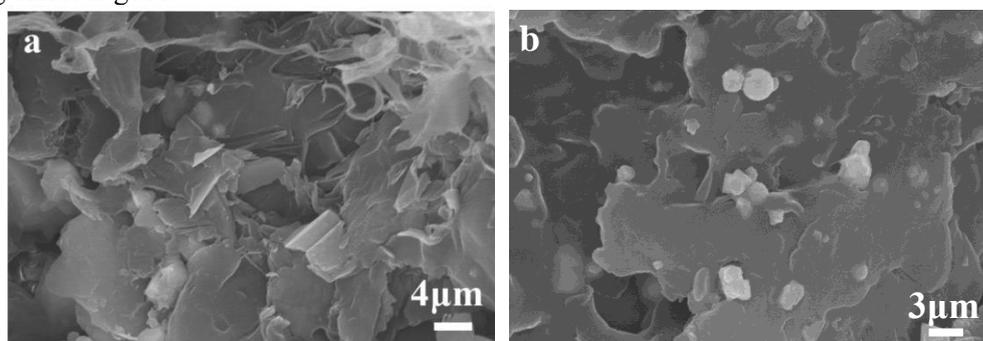


Figure.2 The surface images of W/PPS (W/PPS) by Scanning electron microscopy (SEM)
a) scale bar, 4µm b) scale bar, 3µm

The section microstructure of tungsten powder/polyphenylene sulfide composite sample is shown in

FIG. 3. It can be seen from the figure that there are a small number of 1-2 μm micro voids in the interface between tungsten powder and substrate PPS. On the other hand, PPS material itself has water absorption, and the smaller size of tungsten powder particles themselves will also occur agglomeration phenomenon. During the plasticizing process, the water absorbed by PPS and the gas absorbed by the aggregated tungsten powder will form bubbles in the composite. At the section of PPS/W composite material, most of the tungsten powder particles are completely coated by PPS, while the matrix polyphenyl sulfide is obviously deformed, indicating that the interior of the matrix PPS is weaker than the interface binding area during the fracture process of PPS/W composite material, and the strength of the interface binding area between PPS and metal tungsten powder is better. Therefore, in the fracture of the composite material, the filler tungsten powder in the interfacial bonding area and the matrix polyphenyl sulfide did not peel and split.

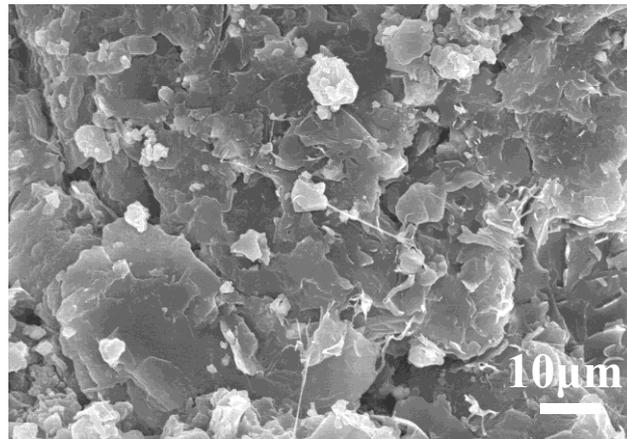


Figure.3 microstructure of tungsten powder/polyphenylene sulfide (W/PPS) at a section of 4000 times, scan bar $10\mu\text{m}$

3.2. XRD analysis

Figure 4 shows the XRD pattern of PPS/W composites. The results show that in the process of thermal compression and heat treatment, the PPS may undergo thermal oxygen cross-linking and oxidation, and form —C=O , —C—O , —S=O , —C—S . By analyzing the XRD curve in figure 4, it is found that the pure PPS material has a higher crystallization state, while the crystallinity of PPS added with lubricant and other additives decreases, which also indicates that the added additives do not react with PPS and generate new phase.

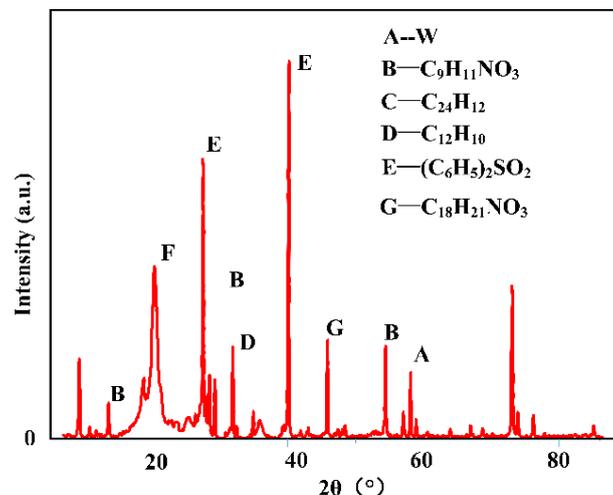
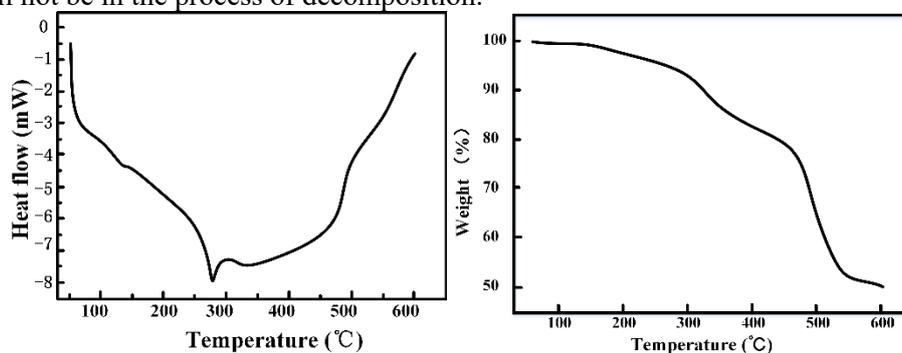


Figure 4 XRD pattern of PPS/W composites

3.3. DSC analysis

We can obtain the phase change decomposition and melting of the material in the process of temperature change, and the decomposition temperature, melting point, phase change temperature and other data of the sample by analyzing the DSC and TG data of the extrusion material.

DSC curves are shown in figure 5-1. From the DSC curve can be learned that the hybrid material melting point is 220 °C or so. In the process of extrusion molding temperature from 260 °C, in turn, reduce the 20 °C to 200 °C, extrusion has been black, mixture burns, carbide. Combined with DSC curve, it should be that recycled materials are used in the raw materials or other mixtures are added. The big difference in melting points of these materials leads to carbonization of the materials. Combined with figure 5-2 TG curve at the same time, the temperature is 250 °C, 5% of the materials have been thermal decomposition. 300 °C at close to 10%. By DSC/TG tests proved that the decomposition of the extrusion temperature is 420 °C, the hot-pressing molding temperature is lower than the temperature, molding materials will not be in the process of decomposition.



1) DSC curves of PPS/W composites 2) DSC curves of PPS/W composites
Figure 5 Thermal decomposition curve of PPS/W composites

4. Conclusion

In this experiment, PPS/W composites were successfully prepared by means of vacuum hot-pressing sintering, and the well-formed products were obtained.

1) It can be seen from the SEM photos that the two phases are well combined. The tungsten powder particles filled are evenly distributed in the PPS matrix, and most of the tungsten powder particles in the composite section are completely coated by the substrate PPS.

2) The temperature, pressure, mold temperature and other conditions of PPS/W composites was explored in the molding process, and determined that extrusion injection molding is the optimal program for processing.

3) Modification of tungsten powder adds make PPS glass transition temperature increased to 100 °C.

4) The XRD curve indicates that the addition of modified tungsten powder does not change the composition of PPS, and the excellent performance of PPS itself remains unchanged while other properties are improved.

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