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Influence of microcrystalline wax on properties of MIM multi-component wax matrix binder①

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[Abstract] The properties of PW-EVA-HDPE binder with the addition of MW were studied. It shows that the addition of MW from 1% to 20% (mass fraction) causes an increase in the tensile strength and a decrease in shrinkage of the binder. After blending PW with MW, the crystallation behavior of wax base changes, which results in fine grain for the binder and more isotropic microstructure for the feedstock. The powder loading capacity increases and homogeneity of feedstock becomes better. The reason of the modification is also discussed.

[Key words] metal injection molding(MIM); binder; rheological behavior
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1 INTRODUCTION

As a typical thermal plastic binder system, wax matrix binder is one of the most competitive binders[1]. PW-EVA-HDPE is a good thermal plastic binder that is successfully applied in practice for its good flow behavior and powder loading capacity. However, the microstructure of commercial crude wax is often composed of coarse flake crystallines[2,3]. It shrinks a lot and leads to internal stress while it cools down from the melt. Shape deformation and cracks are easy to occur when the internal stress releases unevenly in different places of the green parts, especially the place where the size changes sharply[4,5]. Separation of binder from the feedstock sometimes happens which leads to defects in the final product. Microcrystalline wax (MW) is chose to modify the characteristics of wax matrix as well as the properties of the binder in this study.

2 EXPERIMENTAL

Four main polymer components, such as paraffin wax(PW), microcrystalline wax(MW), polyethylene-ran-vinyl acetate (EVA) and high density polyethylene(HDPE), and some additions were used to make the binder. The characteristics of these polymers are listed in Table 1. Metal powder adopted was 4 μm spherical carbonyl iron provided by BASF.

Different mass fractions of MW (from 0 to 30%) were blended with PW. Put MW in and mingle the blend after the paraffin wax was fully melted in the stainless steel double-layer container heated by oil to 90 ℃, take sampling from the container after 30 min, and test the properties of wax blend. Then, appropriate amount of MW was added in paraffin wax according to the former results. EVA and HDPE were added in turn, then the blend is mixed for 120 min. Additions such as stearic acid(SA) were added to enhance the wetting between the binder and metal powder[6,7] in the end before the mixture cooled down. Viscosity was tested by INSTRON 3211 capillary rheometer[8]. Shrinkage in volume was tested by a capillary dilatometer. Mechanical properties were tested so as to evaluate the strength of feedstock. SEM was applied to check the fracture surface of wax blend and the binder after it was coated with a film of gold. The feedstock was observed by SEM to determine the homogeneity.

Table 1 Characteristics of polymer components

Material	Melting point/ ℃	Density/ ($g \cdot cm^{-3}$)	Relative molecular mass(code)	Shape
PW	58	0.911	360~ 540	Flake
MW	80	0.925	550~ 700	Powder
EVA	110	0.942	(E/VAC14-M-D090)	Granular
HDPE	140	0.957	(PE-M-57047)	Granular

3 RESULTS AND DISCUSSION

3.1 Viscosity of wax blend

Fig. 1 shows that with increasing mass fraction of MW(from 0 to 30%) added into PW, the viscosity of wax blend increases accordingly; while it decreases when the temperature increases from 100 ℃ to 140 ℃.

3.2 Strength of wax blend

Fig. 2 shows the obvious effect of the addition of MW on the tensile strength and shrinkage of wax blend. It shows almost a linear increase of tensile strength at 1%~ 10% MW and the increase is much more mild when w(MW) > 10%. There exists a maximum point of tensile strength near 20% MW. The tensile strength of wax blend with the addition of 20% MW is about five times the value of that of the crude paraffin wax.

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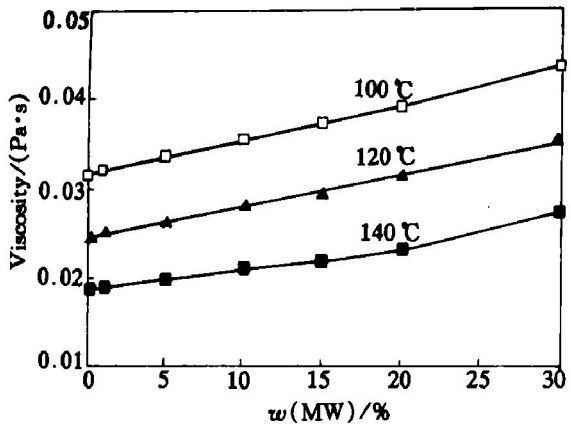

Fig. 1 Viscosity of wax blend as a function of addition of MW at different temperatures

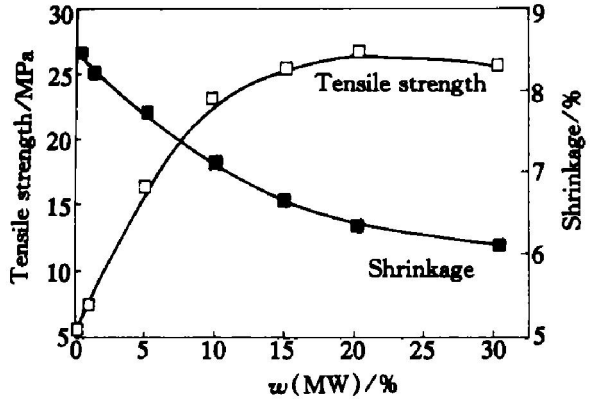

Fig. 2 Tensile strength and shrinkage of wax blend as a function of addition of MW

3.3 Shrinkage

Melt wax blend for sampling will contract when it cools down from liquid to solid and its volume will shrink. Fig. 2 shows a linear decrease of shrinkage at 1% ~ 15% MW and the decrease becomes slower when w (MW) > 15%. The shrinkage reaches 6.44% with the addition of 20% MW and gets three quarters of that of the crude paraffin wax.

3.4 Fracture surface morphology

Once the metal powder is chosen, defects in MIM parts is often relate to the binder. As for thermoplastic wax binder system, the properties of wax base are crucial. A good rheological property is required to suit the flow behavior of the feedstock[5]. It requires good combination of binder with metal powder at the processing temperature with high shear rate and little shrinkage when the feedstock gets into the cool die. Thus, 20% MW is adopted to blends with PW according to the above results.

3.4.1 Wax blend

Fig. 3 shows a brittle fracture surface with a coarse flake microstructure(50 ~ 150 μm) for commercial crude paraffin wax. With the addition of 20% MW, it gets a uniform fine crystal(5 ~ 8 μm), as shows in Fig. 4. The effect of MW on minimizing the crystal size is obvious.

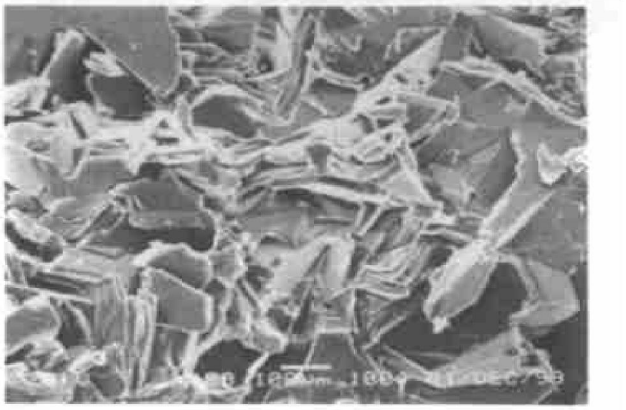

Fig. 3 Fractograph of crude PW

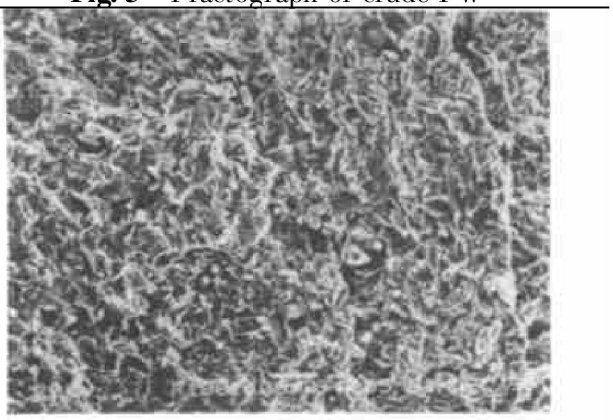

Fig. 4 Fractograph of wax blend

3.4.2 Binder

Fig. 5 shows the crack surface morphology of binder 1(PW-15EVA-5HDPE). The crystal has no sign of deformation and its average size is about 15 μm . The fracture surface morphology of binder 2 with the addition of 20% MW(PW-20MW-15EVA-5HDPE) is shown in Fig. 6. It obviously illustrates a smaller crystal size and strip-like deformation and its average size is about 3 μm.

3.4.3 Feedstock

Feedstock is prepared in a double planetary mixer at 130 ℃ for 3 h with a rotation rate of 60 r/min. The same metal powder is employed and powder loading

Fig. 5 SEM morphology of fractured surface of binder 1

Fig. 6 SEM image of fractured surface of binder 2

capacity is 58% for both binders. Fig. 7 gives the SEM image of fracture surface of feedstock made from binder 1(PW-15EVA-5HDPE). There is a thick film among the powders and the wetting of powder is insufficient for the binder conglomeration is observed in some places. For binder 1, the maximum powder loading capacity is 60%. With the addition of 20% MW, the feedstock made from binder 2(PW-20MW-15EVA-5HDPE) shows a homogenous microstructure with thin film of binder coated on the surface, as shows in Fig. 8. The maximum powder loading capacity is 64%.

From viewpoint of Freund[8], PW mainly consists

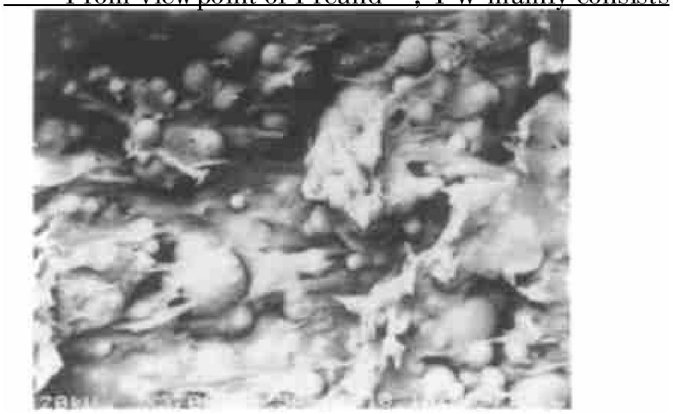

Fig. 7 SEM image of feedstock without MW

Fig. 8 SEM image of feedstock with 20% MW

of regular structure from 22 carbons atom alkane to 36 carbons atom alkane. From 5 ℃ to 8 ℃ below the melting point, there is a crystalline transition($\alpha \rightarrow \beta$) for paraffin wax. α-type crystal is hexagonal while β is orthogonal crystalline. With the transition from α to β, the crystal volume becomes smaller and in a large scale there shows a volume shrinkage. The feedstock contains a very high volume fraction of metal powder and has a higher heat capacity than the pure binder[9]. Furthermore, it is cooled down by cycling water from the injection molding temperature to the die temperature in a very short time. High cooling rate leads to high shrinkage speed, which often results in high remain stress[10]. Crack is often the result of the concentration of high residual stress[11]. MW is an amorphous fine-grain wax, which mainly consists of cyclanes with long side chains and isomer from 37 carbons atom cyclanes to 43 carbons atom cyclanes[8]. With the addition of MW, the new wax blend contains alkane from 22 carbons atom alkane to 43 carbons atoms alkane with regular structure, isomeric cyclanes and cycloparaffins. The properties of wax matrix such as crystal behavior are modified. Amorphous fine-grain MW with a higher melting point comes into being and disperses in the melt blend when it cools down at first. Lower melting point PW crystal wraps on the numerous fine MW core and becomes homogenous fine crystalline wax. There is no transition observed in the experiment for no transition temperature is tested. As a fine crystalline wax, it is more easier to blend with the polymers such as EVA and HDPE. Side chains and cyclo-chains applied is good for incorporates with polymer[12]. The fine wax crystalline also improves the crystal behavior of the binder and fine crack surface is observed. As a result, a thin film coats form on the metal powder after high shear rate mixing. Uniform feedstock with high powder loading capacity obtains. After injection molding, little cracks is found even the most complex parts are used. The variety of piece mass minimizes and a larger tolerance of green parts is achieved[13].

4 CONCLUSIONS

1) With the mass fraction of MW increasing, the viscosity of wax blend increases accordingly at a certain injection molding temperature; while it decreases when the temperature increases from 100 ℃ to 140 ℃.

2) The tensile strength of wax blend with the addition of 20% MW is about five times the value of that of the crude paraffin wax. The shrinkage reaches 6.44% with the addition of 20% MW and gets three quarters that of the crude paraffin wax.

3) With the addition of 20% MW, the wax base alters from a coarse flake grain(50~ 150 μm) crude

PW to a uniform fine crystal(5~ 8 μm) grain wax blend.

4) As for the binder, it illustrates a smaller crystal size and strip-like deformation and its average size is about 3 μm. The feedstock made from binder 2 (PW-20MW-15EVA-5HDPE) shows a homogenous powder group with thin film of binder coated on the surface and gets a maximum powder loading capacity of 64%.

5) After injection molding, little cracks is found even the most complex parts are used. The variety of piece mass minimizes and a larger tolerance of green parts is achieved.

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