Sains Malaysiana 43(1)(2014): 123–128

Characterization of the Feedstock Properties of Metal Injection-molded WC-Co with Palm Stearin Binder System

(Pencirian Bahan Suapan bagi Pengacuanan Suntikan WC-Co dengan Sistem Bahan Pengikat Stearin Sawit)

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ABSTRACT

Feedstock preparation, as well as its characterization, is crucial in the production of highly sintered parts with minimal defect. The hard metal powder - particularly, cemented carbide (WC-Co) used in this study was investigated both physically and thermally to determine its properties before the mixing and injection molding stage. Several analyses were conducted, such as scanning electron microscopy, energy dispersive X-ray diffraction, pycnometer density, critical powder volume percentage (CPVP), as well as thermal tests, such as thermogravimetric analysis and differential scanning calorimetry. On the basis of the CPVP value, the feedstock, consisting of WC-Co powder, was mixed with 60% palm stearin and 40% polyethylene at an optimal powder loading, within 2 to 5% lower than the CPVP value. The CPVP spotted value was 65%. The feedstock optimal value at 61% showed good rheological properties (pseudoplastic behavior) with an n *value lower than 1, considerably low activation energy and high moldability index. These preliminary properties of the feedstock serve as a benchmark in designing the schedule for the next whole steps (i.e. injection, debinding and sintering processes).*

Keywords: Critical powder loading; metal injection molding; palm stearin; WC-Co

ABSTRAK

Penyediaan bahan suapan berserta dengan penciriannya adalah sangat penting dalam menghasilkan jasad sinter berketumpatan tinggi dengan kecacatan yang minimum. Serbuk logam keras, iaitu karbida terekat (WC-Co) yang digunakan dalam kajian ini diuji secara fizikal dan terma untuk mengkaji sifatnya sebelum proses percampuran dan pengacuanan suntikan dilakukan. Beberapa ujian telah dilakukan, antaranya mikroskopi elektron imbasan (SEM), serakan tenaga sinar-X (EDX), ketumpatan piknometer, jumlah peratusan serbuk kritikal (CPVP) manakala secara terma adalah analisis permeteran graviti haba (TGA) dan permeteran kalori pengimbasan kebezaan (DSC). Berdasarkan nilai CPVP yang diperoleh, iaitu 65%, didapati bagi bahan suapan yang mengandungi serbuk WC-Co yang dicampur bersama bahan pengikat stearin sawit sebanyak 60% dan polietilena sebanyak 40%, beban serbuk yang optimal berada 2-5% di bawah nilai CPVP tersebut. Seterusnya didapati sifat reologi bagi bahan suapan pada beban serbuk yang optimal, iaitu 61% mempamerkan sifat pseudoplastik, dengan menunjukkan nilai n *kurang daripada 1, tenaga pengaktifan aliran yang rendah dan indeks pembolehacuanan yang tinggi. Kesemua ciri awal bahan suapan ini akan digunakan sebagai penanda aras dalam merangka jadual bagi proses yang berikutnya; iaitu pengacuanan suntikan, penyahikatan dan seterusnya pensinteran.*

Kata kunci: Pembebanan serbuk genting; pengacuanan suntikan logam; stearin sawit; WC-Co

INTRODUCTION

Feedstock characterization is one of the most crucial steps in metal injection molding (MIM) technology because the rest of the steps (molding, debinding and sintering) depend on the properties of the feedstock. One of the most important parameters is the optimal solid loading, which is estimated based on the critical powder loading. It is a state where all the spaces between particles are filled with binder and no void exists. The optimal solid loading is usually kept lower by 2 to 5 vol.% than the critical value (German & Bose 1997) to ensure process flexibility and to recognize powder-binder variations. Critical powder loading is important in determining rheological properties and interparticle distances. It is affected by the binder system and by the following powder characteristics: Mean size (fine or coarse), particle size distribution (wide, narrow, monomodal or bimodal), and particle shape (spherical or irregular) (Contreras et al. 2010).

In addition, the viscosity of MIM feedstock plays a very important role in MIM because the particles must be allowed to flow into the die cavity. This process requires specific rheological characteristics (German & Bose 1997). MIM feedstock is often rheologically characterized using capillary rheometry, especially at low shear rates. Good rheological properties of binders and feedstock are one of the keys to produce green parts with uniform density and no defects, in addition to obtain successful debinding and sintering and high-quality products (Rhee et al. 1998). Palm stearin has been reported as having a good attribute as a binder system in MIM (Iriany 2002; Istikamah 2010; Nor et al. 2010). Although the role of the binder as a vehicle to support the metal powder is only temporary, especially during mixing and injection molding, it is very important. Its behavior, especially its flow properties during molding, is the most important criterion in developing new binder systems to ensure that no defect occurs during molding. The characteristics of the feedstock are crucial because the part defects can be controlled in subsequent processing steps. The objective of this study was to investigate the feedstock characteristics until the stage of the feedstock rheological properties.

EXPERIMENTAL DETAILS

The powders used in the present investigations were WC (supplied by Eurotungstene.com) and Co powders (supplied by Buffalo Tungsten, Inc.). The elemental WC powder and Co powder were milled in ethanol media to form the WC-9Co alloy. The procedures and results of the wet milling process have been discussed in literature (Amin et al. 2012). The elemental distribution nearest 91% of WC and 9% of Co was checked using energy dispersive spectroscopy (EDS). The microstructure of the ball-milled WC-Co powder was observed using scanning electron microscopy (JEOL JSM-6380LA).

The binder system used in this investigation comprised of palm stearin and polyethylene (PE). The characteristics of the binder components were examined using differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA), based on the ASTM Standard E 473-00. The critical powder loading of the WC-Co powder was tested according to the modified American Society for Testing and Materials Oil Absorption Test, ASTM D-28-31. The rheological behavior of the feedstock was examined using a CFT-500D Shimadzu capillary rheometer, which measures the viscosity resistance when melted materials pass through an orifice. The test was conducted at a constant powder loading of 61%. The capillary temperature was from 130 to 150°C.

RESULTS AND DISCUSSION

Figure 1 shows the EDS graph of the wet-milled powder. The graph shows presence of 86.22% WC and 13.79% Co in the alloy powder, indicating that the nearest value was the ideal one (WC-91% and Co-9%).

The morphology of the milled powder, as shown in Figure 2(a), shows a deagglomerated powder with finer particle sizes compared with the as-received sample (Figure 2(b)). This criterion is favorable because the homogenous feedstock of deagglomerated powders exhibits low viscosity and high flow stability (Suri et al. 2003).

In the DSC analysis, the peak of the graph corresponded to the melting temperature of the binder component. As shown in Figure 3(a) and 3(b), the melting point of the Palm Stearin binder was originally 61°C, whereas that of PE was 127°C. The mixing and molding temperatures should be set above the melting point of the highest melting component of the binder (i.e. PE 127°C) to ensure that all the binders will melt and that the mold will be homogenously filled with the feedstock. Mold temperature should be kept below the melting point of the minor binder (i.e. Palm stearin, 61°C) to prevent the molded part from sticking into the mold cavity.

Figure 4(a) and 4(b) shows the TGA curve for both Palm Stearin and PE, respectively. Palm Stearin decomposed between 398.5 and 598.8°C (Figure 4(a)),

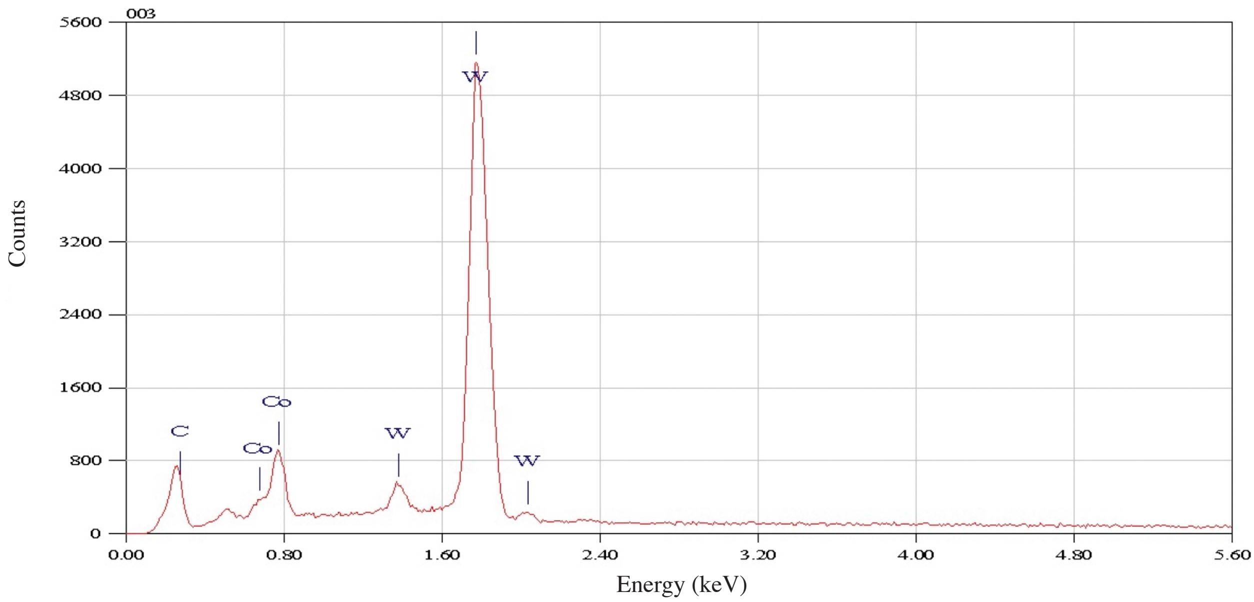
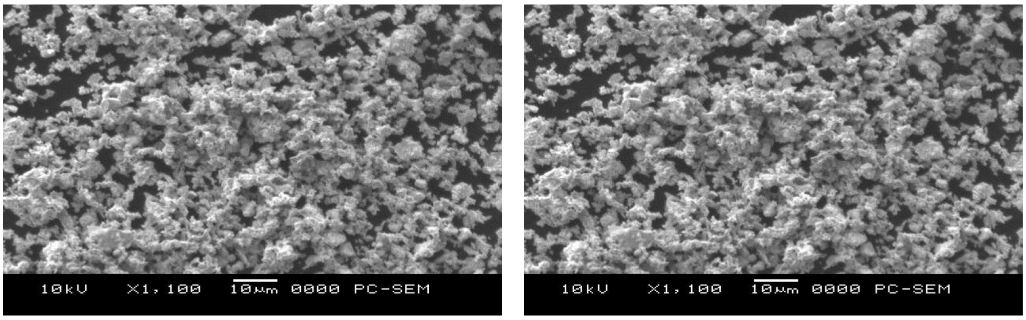
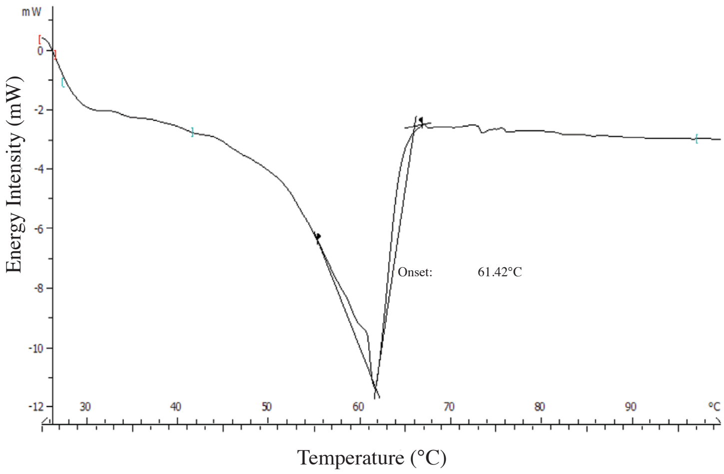


FIGURE 1. Elemental distribution via EDS after wet milling

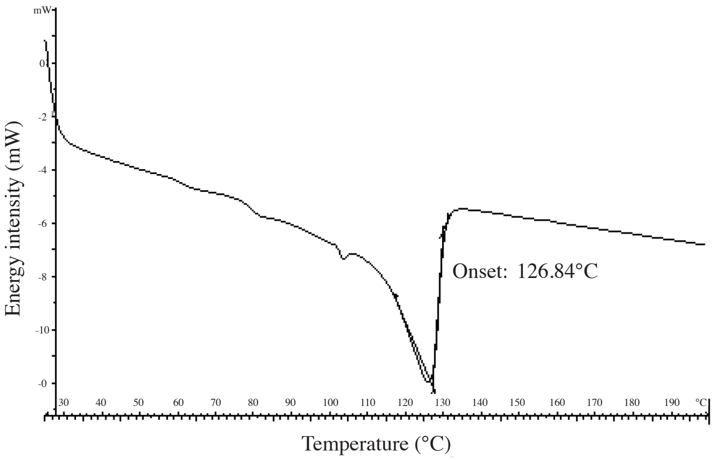


(a) (b)

FIGURE 2. Morphology of (a) wet milled powder and (b) as-received powder



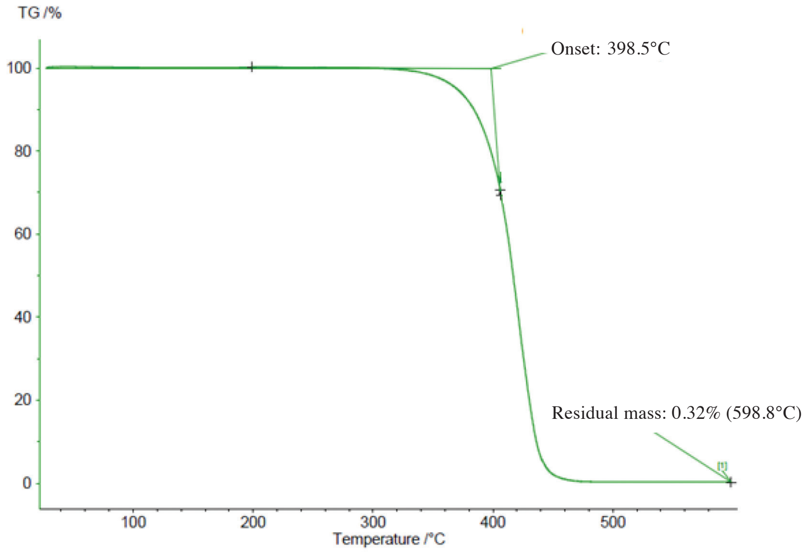
(a) Palm stearin binder



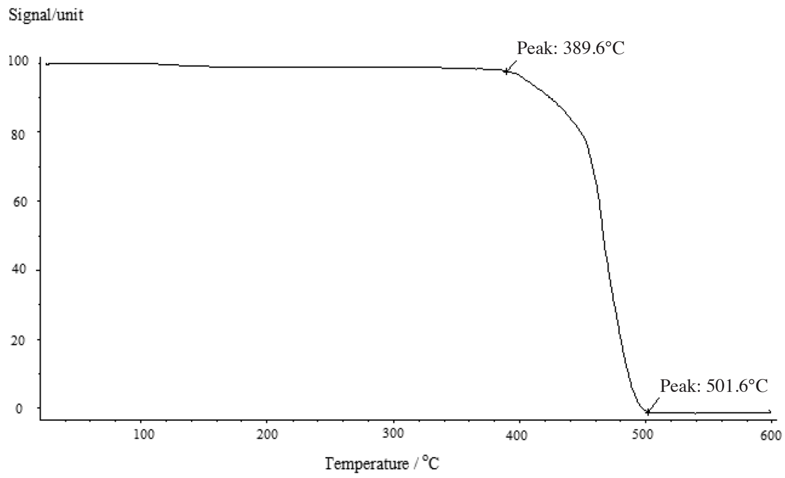
(b) Polyethylene (PE) binder

FIGURE 3. Graph of intensity vs temperature showing the melting point of binders

whereas PE decomposed between 389.6 and 501.6°C (Figure 4(b)). The molding and mixing temperatures must be below the binder decomposition temperature (German & Bose 1997) to prevent binder degradation. According to the TGA analysis, a wide decomposition range is very useful for a fast debinding process and a defect-free product (Youseffi & Menzies 1997). The temperature should not be raised too quickly to prevent defects, such as bubbles and cracks (Luo et al. 2009). The TGA curve was also used to design the thermal debinding cycle, whereby all binders



(a) Palm stearin



(b) Polyethylene

FIGURE 4. Decomposition temperature of binders (a) palm stearin and (b) polyethylene

were removed at above the maximum decomposition temperature of the binders.

Figure 5 shows the torque evolution during the testing of the critical solid loading for the WC-Co powder, with the addition of oleic acid to serve as binder. When the critical solid loading was reached, the torque value decreased with the addition of powder. The reason for this phenomenon is that the excess powder is no longer part of the powder-binder mixture, which leads to reduced cohesion mixture (Contreras et al. 2010). Our calculation showed that the critical powder loading in this study was 65%. Thus, optimal solid loading was at 60, 61, 62 and 63 (i.e. 2 to 5% lower than the critical value). This value greatly differs from that of others, such as Yunn et al. (2011), who obtained 46 vol.% of critical solid loading because the powder used in their study was not milled. This result is supported by Hezhou et al. (2008), who reported that milling enhances the maximum powder loading of the feedstock and by Shengjie et al. (2006), who reported that critical powder loading is inversely proportional to the size of the powder particle. Nano powders have greater surface area and are easier to aggregate than micro powders. This hypothesis is confirmed by Yang and German (1998), who obtained 59 vol.% of critical solid loading for the nanophase cemented carbide that they used.

Figure 6 shows the viscosity as a function of shear rate for the feedstock at 61% powder loading, shear rate at 1000 s^{-1} and temperatures ranging from 130 to 150°C. In general, the feedstock exhibits pseudoplastic behavior, whereby the viscosity decreases as the shear rate increases (shear thinning). This behavior can be due to the breakage of particle agglomerates with the release of the fluid binder (German & Bose 1997). Table 1 summarizes the important rheological properties of the feedstock: Flow behavior index

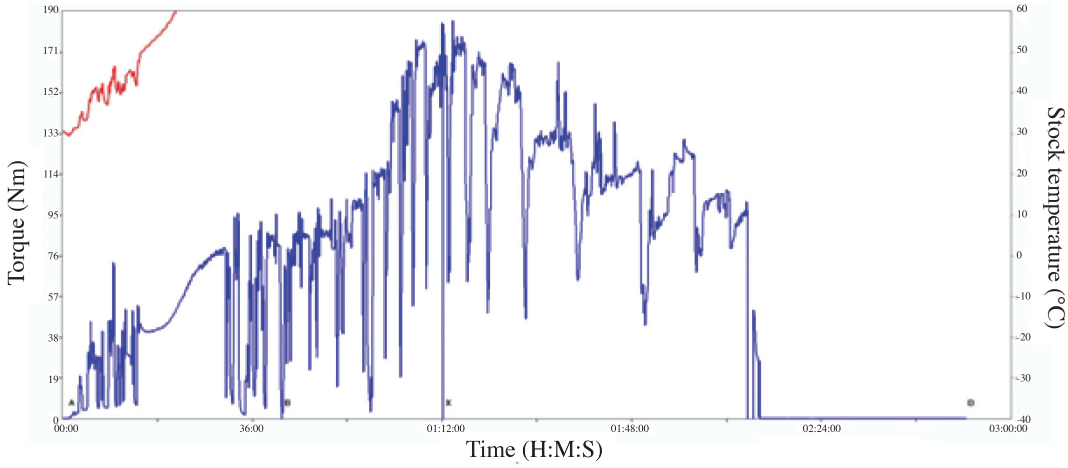


FIGURE 5. Torque evolution of WC-Co powder for CPVC test

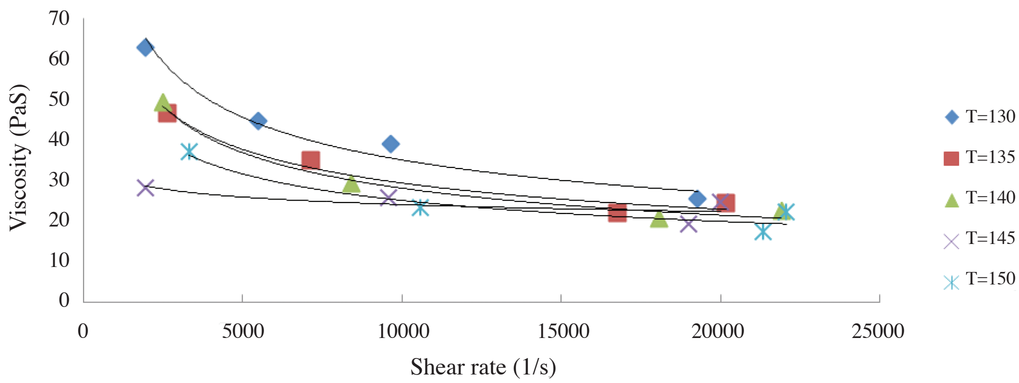


FIGURE 6. Viscosity of PIM feedstocks as a function of shear rate at powder loading 61%

TABLE 1. Rheology properties of feedstock at shear rate 1000 s^{-1}

Powder loading (%vol)	Temperature (°C)	Flow behaviour index n	Activation energy E (kJ/mol)	Apparent viscosity η (Pa.s)	Moldability index α
61	130	0.62	47.3	84.19	792.45
	135	0.64	47.3	67.03	942.93
	140	0.608	47.3	69.07	996.32
	145	0.892	47.3	30.69	617.85
	150	0.665	47.3	54.2	1085.13

n, activation energy E and moldability index α. The flow behavior index, n, indicates the degree of shear sensitivity. The value of n should be smaller than 1 to represent the shear thinning behavior of pseudoplastic materials. A low n value indicates that viscosity is more dependent on shear rate. The n value of the feedstock at 61% powder loading indicates that the feedstock is greatly dependent on the shear rate. However, a very low n value is undesirable because it can lead to the slip flow phenomenon that can cause molding defects. In this case, the feedstock at 145°C showed the best properties because it has the highest value of n or the lowest sensitivity to shear thinning behavior.

The activation energy E, indicates the degree of the dependence of temperature to viscosity. Thus, at high values of E, any small fluctuation in temperature and pressure during molding results in sudden change in viscosity. In contrast, feedstock with less activation energy (less sensitivity to temperature) will minimize stress concentration, cracks and distortion in the molded part. Thus, the feedstock exhibits less sensitivity to temperature and is suitable for injection molding.

Barring any problems, such as jetting or high residual stresses, high values of α are desirable because the feedstock with low α values will be prone to powder-binder separation. As shown, all the feedstock used showed high moldability indices, indicating that minimal compact defects will be produced.

CONCLUSION

The thermal and physical analyses of feedstock have been discussed thoroughly in this study. The critical powder loading spotted for the WC-Co powder is 65%. Thus, the rheological properties of the optimal feedstock with powder loading of 61% show good pseudoplastic behavior, which is suitable for injected molding. All information will serve as benchmarks in designing the subsequent route for processing cemented carbide through PIM.

ACKNOWLEDGEMENTS

The financial support for this project was provided by Fundamental Research Grant Scheme, FRGS (UTM) 78641, Ministry of Higher Education, Malaysia while the equipments and facilities was supported by the Department of Mechanics and Materials, Universiti Kebangsaan Malaysia. Deepest appreciation also goes to Universiti Tun Hussein Onn Malaysia and Ministry of Higher Education, Malaysia for the PhD scholarship.

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Received: 30 March 2012
Accepted: 18 Jun 2012