Journal of Metals, Materials and Minerals. Vol.17 No.1 pp.53-57, 2007

Powder Injection Moulding of Cemented Carbides : Feedstock Preparation and Injection Moulding

Nutthita CHUANKRERKKUL[1*], Peter F. MESSER[2] and Hywel A. DAVIES[2]

[1]Metallurgy and Materials Science Research Institute, Chulalongkorn University, Bangkok, 10330, Thailand
[2]Department of Engineering Materials, University of Sheffield, Sheffield, S1 3JD, United Kingdom

Abstract

Received Mar. 20, 2007
Accepted May 8, 2007

A composite binder system, successfully developed earlier for powder injection moulding of stainless steels, CoCrMo and NiCrSiB alloys, has been adapted for tungsten carbide-cobalt (WC-Co) cemented carbides. The binder consists of a major fraction of polyethylene glycol (PEG), which can be removed rapidly by leaching with water, a minor fraction of a finely dispersed polymethyl methacrylate (PMMA), which retains rigidity of the components and sometimes with stearic acid (SA) to act as a lubricant. The adjustments made to powder characteristics, binder compositions and process parameters to achieve successful injection moulding are reported. Feedstocks containing a powder loading of the WC-Co up to 60 vol% can be moulded successfully. The moulded specimens had good green strength, in the range 12-17 MPa for three point bend tests.

Key words : powder injection moulding, cemented carbides, tungsten carbide-cobalt, PEG/PMMA composite binder

Introduction

Powder injection moulding (PIM) is an effective process for manufacturing small, complex shaped components for high performance applications. The PIM process involves several steps, including feedstock preparation by mixing the powder with a removable binder, injection moulding of the mixture, debinding and sintering.(1) The powder injection moulding process is illustrated in Figure 1.(2) Tungsten carbide-cobalt cemented carbides are used in applications where materials with high hardness and wear resistance with sufficient ductility and toughness are required, for instance, in cutting tools for machining of metal parts or for a variety of wear-resistant components employed in the mining or textile industries.(3)

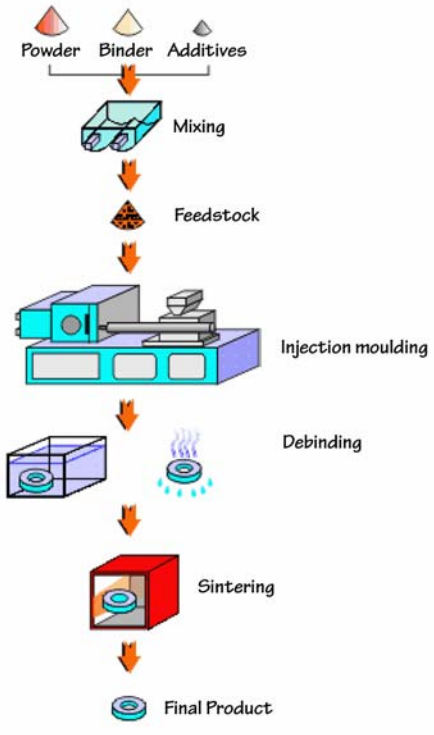

Figure 1. Powder injection moulding process

In PIM process, several classes of binder have been used, but most have long debinding times which can lead to high production cost and high energy consumption. To overcome this problem, a binder system, consisting of polyethylene glycol (PEG) and polymethyl methacrylate (PMMA) was developed at Sheffield University's Department of Engineering Materials.[4] It has been used in a number of previous studies with a range of metallic alloy powders such as stainless steels[4-6] and CoCrMo alloys[7] in addition to ceramic materials.[8] In a preliminary pilot study in this Department, an attempt was made to use the PEG/PMMA/SA composite binder to produce a feedstock with WC-Co powder for injection moulding. However, the feedstocks suitable for injection moulding could not be prepared. Feedstocks, containing ~ 60 vol% or ~50 vol% of the WC-Co mixture, were prepared using a composite binder containing 10 wt% SA. It was found that the binder/powder mixture formed a granulated mass rather than a paste or a mixture in which the liquid separated readily from the solids. As the reasons for these observed behaviours were not understood, the current investigation was undertaken.

Materials and Experimental Procedure

The WC and Co powders, used in this study, were supplied by Marshall Hard Metals Ltd., Sheffield, U.K. WC powder is denoted WC Powder A and powder of 94wt% WC and 6 wt% Co, produced by milling WC and Co powders together, is denoted Powder Mixture D. The WC Powder A, milled for 60 minutes, is denoted WC Powder B. The powder characteristics, including particle size and size distribution, and apparent and tap densities, were evaluated.

The polyethylene glycol (PEG), used in the present study had an average molecular weight of 1500. It was supplied by Honeywill and Stein Ltd., Surrey, U.K. Polymethyl methacrylate (PMMA) emulsion with finely dispersed 0.1-0.2 μm PMMA particles, was specially prepared by Scott Bader Co. Ltd., Wellingborough, U.K. The molecular weight of PMMA is approximately 10^6. Stearic acid (SA) was supplied by Lancaster Synthesis, Morecombe, U.K.

Various feedstock compositions were prepared as listed in Table 1. In the table, the powder loadings are given in terms of volume percentage of the powder assuming that the feedstocks contained no voids and that the powder mixture has a density of 14.93 g/cm3. This was done in order that comparisons could be drawn between feedstocks made with powders having different densities. Studies were undertaken to establish suitable feedstock formulations for WC-6Co powder mixtures with the PEG/PMMA/SA binder system. Feedstocks were composed as follows: 94 wt% of WC Powder A and 6 wt% of Co (Powder Mixture A*), 94 wt% of WC Powder B and 6 wt% of Co (Powder Mixture B*), and Powder Mixture D, which was pre-mixed/milled 94 wt% WC and 6 wt% Co by the supplier. The binder was initially composed of 85 wt% of PEG and 15 wt% of PMMA. For some feedstocks, part of the PEG constituent was replaced with SA and its effect investigated. In two feedstocks made with Powder Mixture B*, the PMMA content was reduced from 15 wt% to 12.5 and to 10 wt%, respectively, so that its effect on the mouldings could be studied.

Table 1. Composition of feedstocks

Feedstock	A*2	A*5	B*16	B*20	B*8	B*9	B*22	D4	D5	D6	D7
Powder loading(vol%)	54	54	60	60	60	60	60	54	54	56	57
PEG (wt%)	85	83	90	87.5	85	83	81	85	83	83	83
PMMA (wt%)	15	15	10	12.5	15	15	15	15	15	15	15
SA(wt%)	0	2	0	0	0	2	4	0	2	2	2

PEG = polyethylene glycol, PMMA = polymethyl methacrylate and SA= stearic acid

Injection moulding was performed using a simple plunger-type machine, which was originally employed for plastic injection moulding. A diagram of the machine is shown in Figure 2. The temperature used was 100 °C with 44MPa injection pressure for the mould having dimensions of 5 x 5 x 55 mm. The as-moulded specimens were subjected to density measurement and strength evaluation. Densities of the mouldings were measured by a mercury displacement method. Mercury was used in the present study in order to avoid PEG dissolution in the water during the measurement. A Hounsfield universal testing machine was employed to determine the strength of the mouldings.

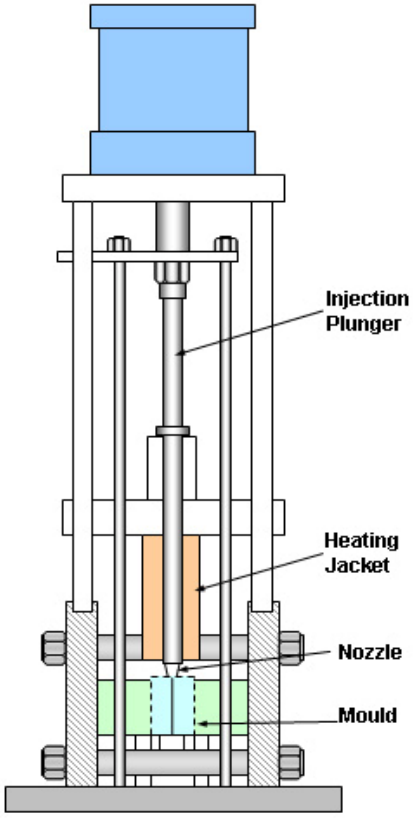

Figure 2. A plunger-type injection moulding machine

Results and Discussion

WC Powder A was composed of aggregates, had a coarse particle size and was not considered to be suitable for sintering.[9] The powder was milled in order to reduce the size of the aggregates. Milling was carried out with a Fritsch planetary mill, comprising a pot, lined with cobalt-bonded tungsten carbide, and milling balls (20 mmϕ) of the same material. The particle size of WC Powder A was reduced after milling and the apparent and tap densities of the resulting WC Powder B were improved. Co and Powder Mixture D had small particle sizes and low values for both apparent and tap densities. The characteristics of powders are presented in Table 2.

Table 2. Characteristic of powders

Code	Powder	Relative apparent density*	Relative tap density*	D_{10} (μm)	Median size, D_{50} (μm)	D_{90} (μm)
A	WC Powder	36%	46%	17	43	101
B	WC Powder	44%	55%	3.6	18	50
Co	Co Powder	24%	37%	1.5	4.9	9.9
D	WC-6Co Powder	15%	25%	3.5	6.8	12.7

* The densities shown are relative to theoretical values of 15.6 g/cm^3 for WC , 14.93 g/cm^3 for WC-6Co and 8.9 g/cm^3 for Co

D_{10}, D_{50} and D_{90} and are the particle sizes corresponding to 10%, 50% and 90% of the cumulative size distribution, respectively.

For a feedstock preparation, the powders were mixed by hand with the required amount of PMMA emulsion. The mixture was dried before further hand mixing with molten PEG or PEG/SA. Stearic acid was added to the molten PEG in feedstocks containing lubricant. Hand mixing has been used in the present study because it would have been costly to use the available high-shear mixer , which had a 1 litre mixing chamber, with these high density powders. This is because of the high cost of WC-Co mixture per kilogram and its very high density of ~ 15 g/cm3. Feedstocks of Powder Mixture D were also prepared, however, the powder loading was not as high as those of Powder Mixture B*.

Feedstocks were injection moulded successfully. No binder segregation was observed during the injection moulding. The properties of the mouldings produced with various proportions of PMMA are listed in Table 3. The as-moulded densities generally increase with increasing PMMA content. This observation cannot be

explained on the basis of any change in the density of the binder as a result of different compositions. It is necessary to assume that the voidage in the mouldings decreases with increasing PMMA content. That is, as the PMMA content increases, the Co particles are pulled together more strongly. This will cause the WC particles to pack more densely and result in increasing densities of the mouldings.

Table 3. Properties of the mouldings produced with various proportions of PMMA

Powder Mixture	Powder Loading (vol%)	Binder Composition (wt%) (PEG/PMMA)	As-moulded Density (g/cm^3)	As-moulded Strength (MPa)
B*	60	90/10	9.34 ± 0.03	15.1 ± 2.4
		87.5/12.5	9.37 ± 0.03	17.6 ± 1.0
		85/15	9.53 ± 0.06	16.8 ± 0.5

(mean ± 95% confidence limit, n=5)

The as-moulded strengths of the mouldings did not show any statistically significant trend,[10] possibly because they were affected by the amounts of both the PEG and PMMA in the mouldings. The contribution to the strength from the PEG would be expected to be reduced by the reduction in its content. On the other hand, the contribution to the strength from the PMMA would be increased by an increase in its content.

In Table 4, data are presented for mouldings made with SA partly replacing PEG. The densities of the as-moulded specimens for a constant powder loading generally decrease with increasing SA content, except for the as-moulded density of samples made with Powder Mixture B* and 2 wt% SA. The observed decreases in density are expected to have occurred because the ability of the PMMA to pull particles together would be reduced with a reduction in the bonding of PMMA to the Co particles. The increase in the as-moulded density of samples made with Powder Mixture B* and 2 wt% SA suggests that the distribution of the Co particles throughout the mass of WC particles is significantly improved with better mixing with the use of SA that acts as a lubricant. This increased the ability of the PMMA to pull the particles together, despite a reduction in the sites to which PMMA could bond.

Table 4. Properties of the mouldings with various amount of stearic acid

Powder Mixture	Powder Loading (vol%)	Binder Composition (wt%) (PEG/PMMA/SA)	Density (g/cm^3)	Strength (MPa)
			As-moulded	As-moulded
A*	54	85/15/0	8.76 ± 0.05	15.9 ± 1.7
	54	83/15/2	8.58 ± 0.06	11.5 ± 0.3
B*	60	85/15/0	9.53 ± 0.06	16.8 ± 0.5
	60	83/15/2	9.63 ± 0.01	14.7 ± 0.7
	60	81/15/4	9.21 ± 0.02	12.1 ± 0.7
D	54	85/15/0	7.82 ± 0.02	19.4 ± 1.1
	54	83/15/2	7.48 ± 0.13	10.9 ± 1.7
	56	83/15/2	7.69 ± 0.11	13.4 ± 1.9
	57	83/15/2	7.84 ± 0.06	14.7 ± 2.4

(mean ± 95% confidence limit, n=5)

For the same powder loading, the as-moulded strength decreases with the replacement of PEG by SA. This can be understood by considering that the contribution to the strength from the PEG is reduced because a) the PEG content is reduced and b) bonding of PEG to the particle surfaces is reduced by SA, which is a well-known surface active material, occupying sites to which PEG molecules would otherwise bond. In addition, a reduction in the as-moulded strength should occur because the bonding of PMMA to Co particle surfaces would be reduced by SA molecules occupying sites that would otherwise have been occupied by PMMA molecules.

Conclusions

In the present study, it has been shown that mixing and moulding of WC-Co powder mixture containing a powder loading up to 60 vol% is possible with and without the use of SA. Preparation of feedstocks of a finer particle size of WC-Co powder mixture is possible up to a powder loading of 57 vol% with an addition of 2 wt% SA.

This amount of SA is smaller than the amount employed in the previous pilot study.
.

It is probable that a higher density might be obtained if the feedstocks made with Powder Mixture D and the PEG/PMMA/SA binder could be prepared with higher powder loading, using a mechanical high-shear mixer.

References

1. German, R. M. and Bose, A. 1997. *Injection Moulding of Metals and Ceramics*, Princeton, NJ. : Metal Powder Industries Federation.

2. Alcock, J. and Stephenson, D. The Powder *Injection Moulding Process*. (Online) Available at : http://www.azom.com/ Details.asp?ArticleID=1080 (accessed 15/3/2007).

3. Upadhyaya, G. S. 1998. *Cemented Tungsten Carbides : Production, Properties, and Testing*, Westwood, NJ. : Noyes Publications.

4. Anwar, M. Y., Messer, P. F., Ellis, B. and Davies, H. A. 1995. Injection moulding of 316L stainless steel powder using novel binder system. *Powder Metall.*, **38(2)** : 113 - 119.

5. Omar, M. A., Davies, H. A., Messer, P. F. and Ellis, B. 2001. The influence of PMMA content on the properties of 316L stainless steel MIM compact. *J. Mater. Process Tech.* **113** : 477-481.

6. Newell, M. A., Davies, H. A., Messer, P. F. and Greensmith, D. J. 2005. Metal injection moulding of scissors using hardenable stainless steel powders. *Powder Metall.* **48(3)** : 227-230.

7. Bhero, S. W. 1999. *Powder injection moulding of Co-Cr-Mo alloy and type 440 martensitic stainless steels.* Doctoral The University of Sheffield.

8. Wong, S. H. 2003. *Injection moulding of anorthite/mullite porcelain*. Doctoral The University of Sheffield.

9. Chuankrerkkul, N. 2006. *Powder injection moulding of WC-Co hardmetals using a PEG/PMMA binder*. Doctoral The University of Sheffield.

10. Hamburg, M. and Young, P. 1994. *Statistical Analysis for Decision Making*. Fort Worth, TX. : The Dresden Press.