MIM offers increased applications for submicron WC-10%Co

Submicron cemented carbides processed by the conventional powder metallurgy (PM) press and sinter route are already in use in a variety of cutting tool applications. The use of powder injection moulding (PIM) techniques would extend this range because of the increased freedom of product design. This study by Johnny Bruhn of Seco Tools and Björn Terselius of Kristianstad University College examines the basic steps of PIM for a submicron WC-10%Co hardmetal system using a wax-polymer binder and stearic acid as a dispersant.

Powder injection moulding (PIM) is still a relatively new technique for manufacturing complex-shaped articles from a variety of powder filled systems containing powder and an organic vehicle that makes the systems fluid and thermoplastic. One of the primary advantages of PIM is the freedom of product design compared with conventional forming techniques. The basic steps of the PIM process are: powder preparation, mixing of powder and binder in a suitable feedstock processor to a highly uniform suspension, injection moulding to produce green bodies of the desired shape, careful removal of the binder and, finally, sintering. Post-sintering operations may be required to ensure necessary product quality, thus reducing the competitive edge over current manufacturing techniques. Fundamental knowledge of each of the process steps and their interaction is the key to successful use of PIM.

The dominant manufacturing route for hardmetal cutting tools is uniaxial powder compaction and sintering. The powder compaction process is today a highly optimized technique offering both good productivity and the possibility of manufacturing products of complex shape. Thus the application of PIM for hardmetal cutting tools lies primarily in novel product designs adding unique features to the product.

The investigation described here studied the different steps of a PIM manufacturing process using a submicron WC-10%Co hardmetal grade currently produced by Seco Tools AB (Fagersta, Sweden) for a variety of cutting tool applications in the aerospace, automotive and engineering industries (see Figure 1).

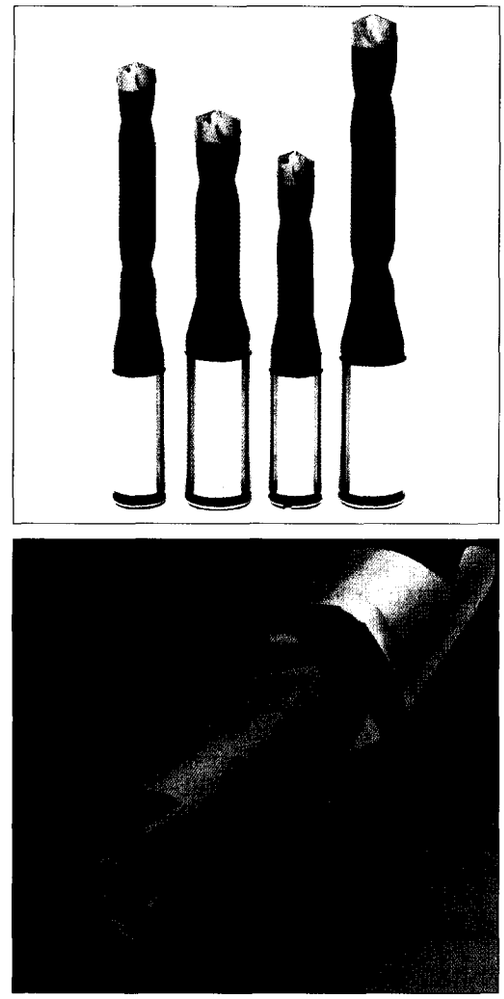

FIGURE 1: Seco Tools already uses submicron WC-10%Co hardmetal powder in a variety of cutting-tool products such as its range of 'CrownLoc' drills (above) and the '3 Fluted Minimaster' endmill (below).

Processing steps

Submicron tungsten carbide powder (89.55 wt%, WC DS80, 0.8 μm, H.C. Starck) was mixed with cobalt (9.95 wt%, Extra Fine, 1.5 μm, Outokumpu) and powdered stearic acid (0.50 wt%) in a ball mill. Micrograin milling balls and an ethanol-water mixture were used and processing lasted 40 hours. The resulting slurry was then spray dried in a Niro atomizer to granules of 10-40 μm in size.

The binder used is a wax-polymer compound with a high polymer content. The components of the binder were dry blended

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in a Turbula Type T2A mixer in a polyethylene vessel for 300 seconds at room temperature.

A twin screw extruder (Werner & Pfleiderer ZSK25) with a side feeding system was used for mixing the feedstocks. The feedstock components were added gravimetrically into the extruder in order to maintain the desired feed rate. The screw speed was kept constant at 225 rpm and the mixing temperature was 170°C. The feedstock was extruded through a multiport nozzle, cut to form pellets and cooled in air on a vibrating chute.

To produce feedstocks with varying powder contents, a master batch with a low powder content was extruded and pelletized and fed back into the extruder. A calculated additional amount of powder was then added via the side feeder system. Thus all feedstocks were processed twice in the extruder with a total residence time of approximately 60 seconds.

Injection moulding of samples was performed in a Battenfeld BA500/200 CDK-SE injection moulding machine. The barrel temperatures were varied between 130 and 170°C and the injection speeds ranged from 10 to 35 $ml.s^{-1}$.

The samples were placed on zirconia covered graphite carriers and put into a furnace in order to thermally debind the samples in a hydrogen atmosphere of 1 bar. This was followed by pressurized sintering in 30 bars of argon at 1410°C for 0.75 h.

Feedstock analysis

Scanning electron microscopy (SEM) was used to evaluate the feedstock homogeneity and the particle dispersion. SEM studies on the master batch compound of low powder content (47.1 vol%) demonstrated porosity because of trapped air, uneven powder particle distribution and agglomeration in the compound after the first extrusion process (Figure 2a). After the second pass through the extruder these inhomogeneities could not be detected (Figure 2b). The density variations were estimated to ± 0.01 $g.cm^{-3}$. Further processing in the extruder did not improve the homogeneity or reduce the density variations.

The behaviour of stearic acid as a wetting agent/dispersant was also documented. Compounding feedstocks without the dispersant stearic acid resulted in very high extrusion viscosity even at very low powder contents (44.7 vol%) and formation of cracks in the extrudate (Figure 3). No rheometry could be performed.

A Shimadzu Flow Tester CFT-500A was used to characterize the rheological behaviour of the compounded feedstocks. The capillary rheometer operates under a constant pressure applied on a piston which moves through a heated cylinder and pushes extrudate out of a die. A cylindrical die with a 1.0 mm diameter and a 10.0 L/D ratio was used. The analysis was performed according to the ASTM D3835-93a standard.

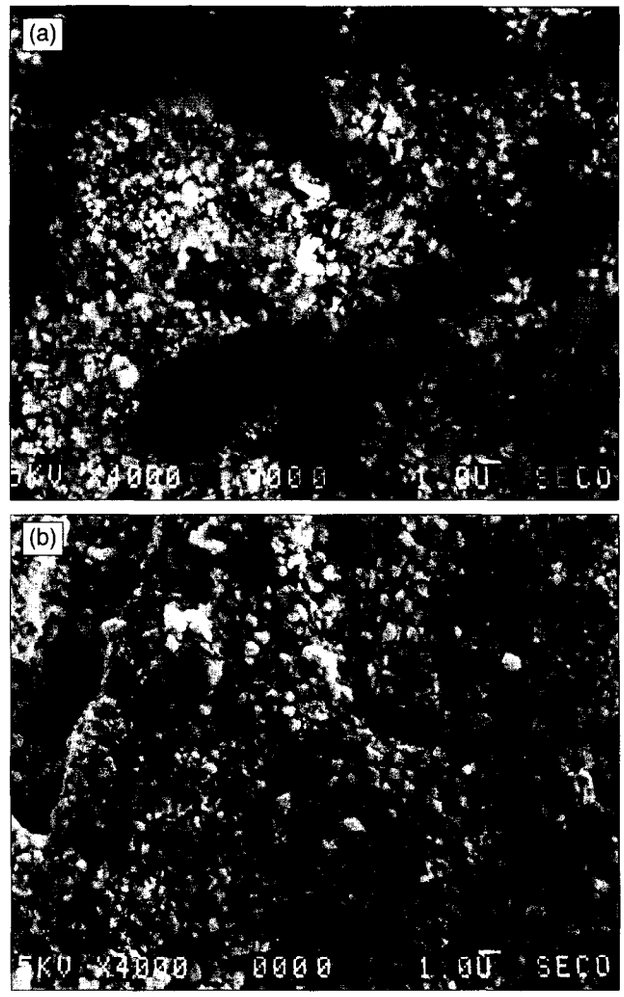

FIGURE 2: SEM images of (a) master-batch compound with 47.1 vol% of hardmetal powder after one extrusion run and (b) compound with 53.5 vol% of hardmetal powder after two extrusion runs (4000X).

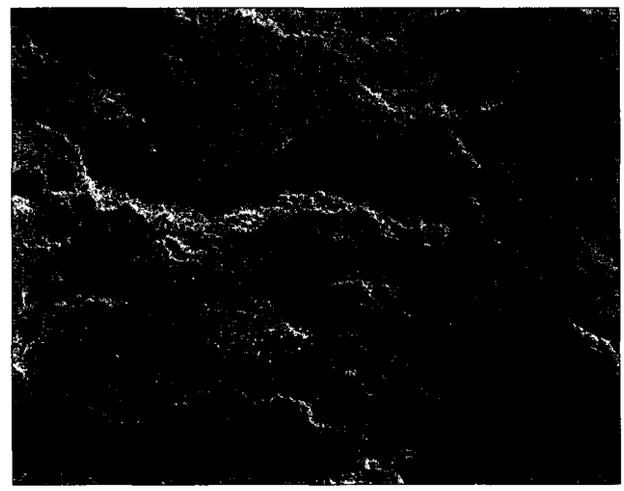

FIGURE 3: SEM image of a compound with 44.7 vol% of hardmetal powder without stearic acid dispersant, showing crack formation (400X).

Using the capillary rheometer, the viscosity of feedstocks with different powder loadings was measured at shear rates from 0 to 5000 s^{-1} and in the temperature range 130 to 170°C. Feedstocks with a high powder content could not be analysed with the rheometer at the lower temperatures or at high shear

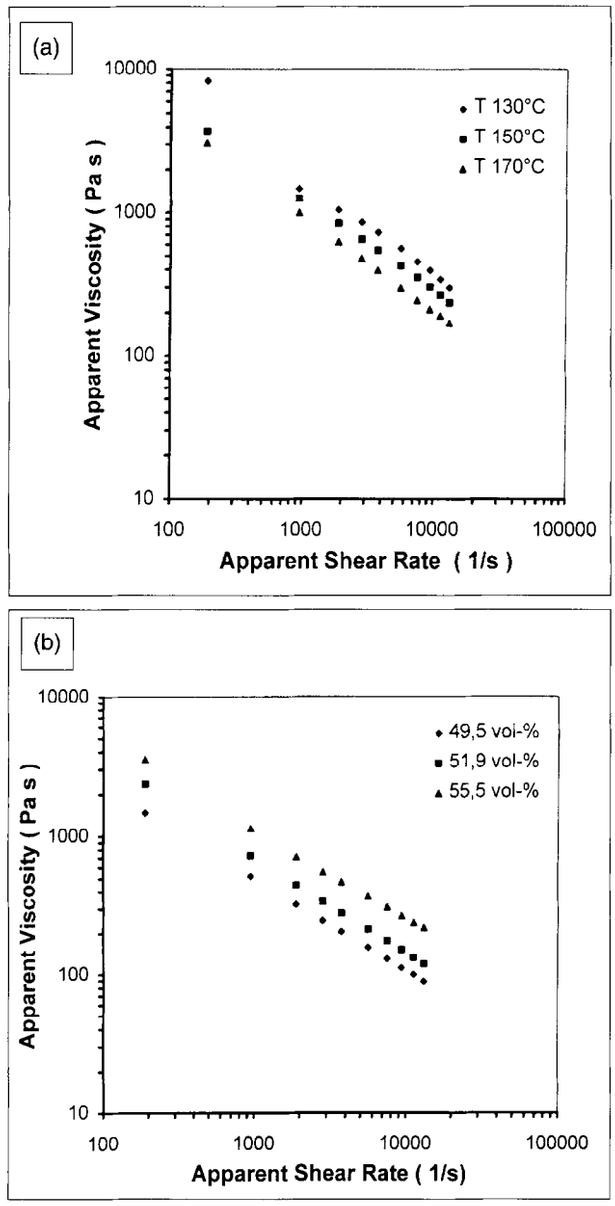

FIGURE 4: The effect on flow data of (a) temperature for a compound with 53.9 vol% hardmetal powder, and (b) powder content at a temperature of 170°C.

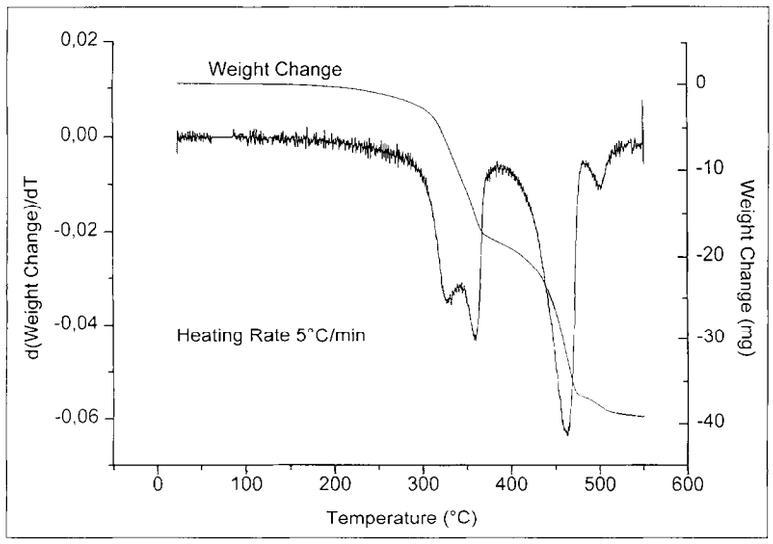

FIGURE 5: TG and DTG analysis of the green body during debinding in flowing hydrogen at 1 bar.

rates, above approximately 1000 s^{-1}, because of the very high viscosity. This was further documented in a study by R.M. German *et al.*[1] using a Dynisco high force model Kayeness Galaxy V Capillary Rheometer.

In order to complete the rheological analysis for all feedstock powder contents within the desired temperature range and using shear rates up to 10 000 s^{-1}, the injection moulding machine was equipped with a pressure transducer close to the nozzle. By using a corrected L/D ratio of 9.2 the obtained curves could be shifted to match those obtained on the Shimadzu Flow Tester.

Plots of apparent viscosity versus apparent shear rate for different temperatures (Figure 4a) and powder contents (Figure 4b) indicate a stable and predictable shear thinning flow behaviour within the tested shear rate and temperature ranges.

Debinding and sintering

The thermal stability and the weight change of the green body during the debinding process were monitored by differential thermal gravimetric analysis (DTG) and thermal gravimetric analysis (TG) from room temperature to 600°C in flowing hydrogen at 1 bar.

The results of the TG and DTG analyses (Figure 5) indicate the presence of two main temperature regions where the polymer decomposition rate is high. In these regions the debinding process must be closely controlled to reduce the formation of defects.

Based on the TG and DTG results, SEM observations of partially and fully debound articles, and practical debinding tests, it was possible to formulate an optimized debinding procedure that produced articles free of macrodefects. The temperature distribution within the debinding furnace was found to be important in finding the correct temperature ramp rates and soak temperatures in the procedure.

The debinding procedure was revised to be slightly more decarburizing because of the high carbon content in the sintered articles. This increased the magnetic saturation level (see Table 1) by 10 units to a minimum of 164. This resulted in the desired microstructure in the sintered article (Figure 6). A PIM part of 17.2 mm diameter was successfully debound in a procedure lasting 34 hours.

The density of the sintered body was measured by the water displacement method. The magnetic properties were determined using a Förster Koercimat coercivity instrument and a Sermag Setaram magnetic saturation instrument. Optical microscopy was used for microstructural analysis and detection of remaining porosity. The hardness was measured using a Leco M-400DT Hardness Tester.

The results of these mechanical and metallurgical tests are shown in Table 1. The corresponding grade specifications for a conventionally produced hardmetal are given for comparison. As can be seen, the mechanical

Table 1: Mechanical and metallurgical properties

	Sintered density ($g.cm^{-3}$)	Coercivity ($kA.\ m^{-1}$)	Magnetic saturation ($\mu T.m^3.kg_{Co}^{-1}$)	Hardness Hv10 ($kg.\ mm^{-2}$)
Grade specification	14.48	20.5	173.7	1550–1600
PIM WC-10%Co	14.47–14.50	19.5–21.0	164.0–180.0	1570–1600

and metallurgical properties of the PIM processed material match those of conventionally produced (pressed and sintered) articles of the same submicron WC-10% Co hardmetal grade.

To conclude, this investigation of the powder injection moulding of a submicron WC-10%Co hardmetal has shown that it is possible to produce articles of complex shape with no residual porosity and with precisely controlled carbon content in the sintered body. Grade specifications are fully met by the PIM processed material.

The MIM project at Seco Tools is on-going and embraces an increasing number of new products and hardmetals. In 1999, all MIM-manufactured products will be offered in a selection of different hardmetals and coatings, which will extend the possible applications even further. The MIM process has been successfully implemented into cutting tool production, with a healthy volume growth expected as products leave the development stage.

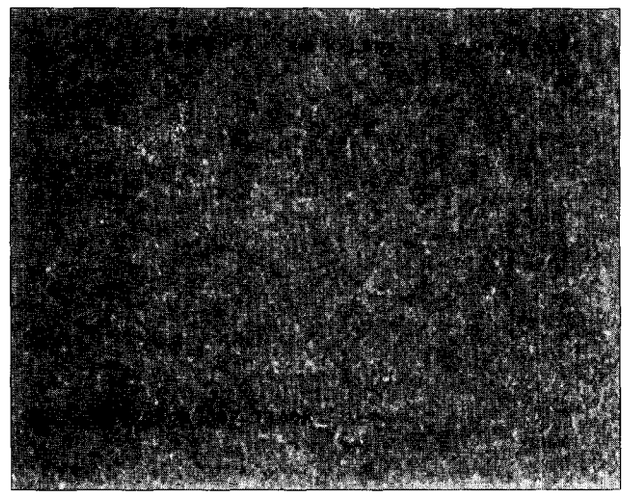

FIGURE 6: Microstructure of sintered submicron WC-10%Co hardmetal (1100X).

Acknowledgement

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Further reading

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