



12th INTERNATIONAL SCIENTIFIC CONFERENCE
ACHIEVEMENTS IN MECHANICAL & MATERIALS ENGINEERING

Manufacturing of steels and magnetic ceramics by Powder Injection Moulding

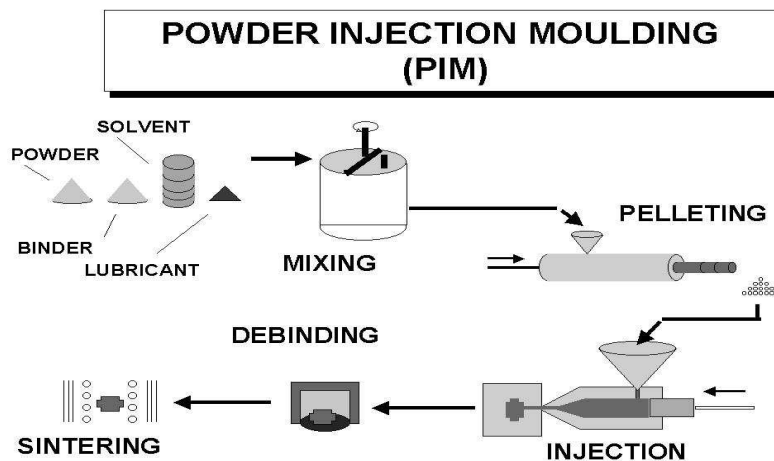
B. Levenfeld

Materials Science and Engineering Department, Escuela Politécnica Superior.
Universidad Carlos III de Madrid, Avda. Universidad, 30. E-28911 Leganés. SPAIN

Powder injection moulding (PIM) is a manufacturing technology that combines the advantages of polymer injection moulding and traditional powder metallurgy (1,2). This process was firstly developed for ceramic powders and is specially appropriate for the production of pieces of small size and complicated forms.

The general steps of the process appears in the figure 1. Essentially it consists on the following stages (1,2,3):

- 1) mixing ceramic or metallic powders with a binder system, usually polymers, to form the " feedstock ".
- 2) pelletizing of the " feedstock " to be able to feed the injection machine.
- 3) injection in a conventional plastics injection machine producing the so-called green part.
- 4) debinding where the binder is removed from the pieces (by chemistry, catalytic or thermal way), obtaining the "brown part".
- 5) Sintering which permit to obtain the sintered part with the required characteristics.



Among the main advantages of the technique stand out the shape complexity, the narrow dimensional tolerances, the big production cadences and the high mechanical properties as consequence of the high densification. Powder injection moulding is an attractive process when the following

component features apply (4): thickness ranging from 0.2 to 20 mm, corner radius greater than 0.075 mm, mass ranging from 0.02 to 1000 g, moderate levels of shape complexity and smooth surfaces. The typical range of tolerances is between 0.1 and 1 mm.

The election of the binder is one of the most critical task in the global process, because it depends on the debinding stage, which normally takes associate a certain technology.

The binders used can be classified in five types: thermoplastic and thermosetting polymers, water based systems and gelation systems.

From the industrial point of view, thermoplastic systems are those most widely used, and these systems include the majority of commercial polymers: polyethylene, polystyrene, polypropylene, etc. In addition to the main component (thermoplastic polymer) the system can contain additives to control lubrication, viscosity, binder adhesion to powder particles, and debinding. Thermosetting polymers imply cross-linking of polymer units at high temperatures, and they are not reversible.

Debinding is also a critical stage. It is a process that can be carried out either in several steps or using one single debinding operation, always depending on the binder system.

The most usual systems are:

- 1) Solvent debinding (water, acetone, toluene,...) (5).
- 2) Catalytic debinding: a gaseous catalyst hydrolyses the binder in its monomers (BASF patent) (6).
- 3) Wick debinding: consists of removing the binder by capillary action, by means of a porous material placed in contact with the green compact (7).
- 4) Thermal debinding: increasing the temperature leads to degradation of the polymer and its elimination (8).

Optimisation of this stage is fundamental to ensure that in sintering there are no remains of the binder that could affect the process and the properties of the material. In the case of stainless steels, complete debinding is critical, as any carbon remains within the stainless steel could cause the sensitisation of the steel during sintering. In contrast, in the case of high speed steels the presence of extra carbon can even be beneficial to activate sintering.

The main achievements obtained in the Powder Injection Moulding Group of the Carlos III University are the development of a new process of manufacturing based on PIM which use thermosetting resin as binder and the designing of new binder formulations based on thermoplastic polymers for PIM process. These two achievements has been applied to both metals and ceramic materials.

MOULDING WITH THERMOSETTING BINDER

In our group, a new manufacturing process of metals and ceramic parts using an acrylic thermosetting resin was developed five years ago (9,10). This process was successfully applied to different metals, such as M2 HSS, T15 HSS, 316 stainless steel. and magnetic ceramic. The main difference of this process with the conventional PIM is the way to obtain the green part. In this case, moulding is performed by pouring a slurry (mixture of binder and powder) into the mould at room temperature. The polymer curing is carried out at relatively low temperature (80-90 °C). Later, the debinding and sintering processes were carried out. To establish the best thermal debinding cycle, thermogravimetric analysis of the binder were performed. The main advantage of this method was the simplicity and the relatively short debinding time

One of the most important result was obtained for M2 HSS. The sintering of this kind of materials is problematic from the industrial point of view since it takes place in a narrow temperature range (about 4 degrees). The addition of carbon results beneficial because enhance the sintering kinetics leading to greater densifications at low temperatures (11-16). In this sense, partial elimination of the binder leave carbon, coming from the binder decomposition, homogeneously distributed in the piece. This fact improved considerably the sintering process reducing up to 100 degrees the sintering temperature and enlarging 100

degrees the sintering window. Moreover, the enlargement of the sintering window allow us to study in detail the evolution of the microstructure during the sintering. Different types of carbides have been identified and their morphologies have been established.

This study was also extended to the T15 HSS (17).

Another effect studied was the influence of the sintering atmosphere in M2 and T15 steels (18). Three different atmospheres were used, low and high vacuum and nitrogen-rich atmosphere. The sintering of both steels takes place at lower temperature, compared to the same steels grades manufactured by conventional powder-metallurgy, and they can be interpreted on the basis of a supersolidus sintering mechanism.

The process was also been applied to stainless steels, concretely to 316L austenitic stainless steel (10,19). The obtained densities and tensile strength were higher than those obtained by conventional PM (95% and 450 MPa respectively).

Recently we have applied this process to ceramic magnetic, concretely to commercial Mn-Zn ferrites. The process permitted to obtain small pieces with toroidal shape which presented similar magnetic properties to those obtained by uniaxial pressure compaction.

DEVELOPMENT OF NEW THERMOPLASTIC BINDERS FOR MANUFACTURING OF M2 HIGH SPEED STEELS.

Nowadays our research work has been focused in the design and development of new binder formulation. Specifically we have recently developed a new binder based on high density polyethylene (HDPE) and paraffin wax (PW) which has been used to obtain M2 HSS parts (20). Preliminary results indicate that it can also be used satisfactorily for T15 grade steel. The PE provides mechanical resistance favoring the shape retention, while the wax, with low melting point and low molecular weight, provides high fluidity diminishing the viscosity. Surface treatment of metal powder with stearic acid decreased the blend viscosity allowing the use of lower injection temperatures. The percentages in volume varied from the 80 to 50% of PE.

In a first stage we proceeded to study the compatibility among the two components by means of dinamomechanical analysis, showing that the total miscibility of the two components does not take place, although the mixture is quite homogeneous.

The rheological study of the binder showed that viscosity decreases as paraffin wax content increased and all the binders presented a Newtonian behavior.

Torque measurements and rheological study of the feedstock allowed us to chose the optimum composition to be injected. The maximum amount of metallic load was 70% volume.

The feedstock was also characterized by viscosity measurements, which increases with the metallic load. In all the cases the feedstock viscosity follows a pseudoplastic behavior, diminishing considerably as the shear rate is increased. This behavior is adequate for the injection process due to the viscosity of the mixture diminished when it is approached to the nozzle and when the mould is filled the viscosity increases. The optimized injection molding process leads to high quality green parts.

As expected the elimination of the binder occurs in a relatively wide range of temperature and in a gradual way. This is as consequence of the two components of the binder with different melting temperature.

Thermal, solvent and wick debinding were performed. The best result was obtained with capillary extraction (wick debinding) where less distortion samples and a easier elimination of the binder was found.

Unlike in the case of samples moulded with thermosetting resin, incomplete debinding did not produce beneficial effects on sintering process, requiring temperature similar to that of conventional PM parts. This fact seems indicate that the decomposition compounds of the binder play a crucial role in the sintering of these steels.

ACKNOWLEDGEMENTS

The author thank CICYT (MAT2000-0417) and “Comunidad Autónoma de Madrid” (3rd Regional Research Programme- “Grupos Estratégicos”) for financial support and the collaboration of the PIM research group of Universidad Carlos III de Madrid.

REFERENCES

1. R.M. German. “*Powder Injection Molding*”. Metal Powder Industries Federation. Princeton, NJ, 1990.
2. R.M.German and A. Bose, “*Injection Molding of Metals and Ceramics*” Metal Powder Industries Federation. Princeton, NJ,1997.
3. P.J. Vervoort, R. Vetter, J. Duszczyk. *Advanced Performance Materials*. Vol. 3 (2), (1996), 123-151.
4. R. M. German. *Advances in Powder Metallurgy and Particulate Materials*, Metal Powder Industries Federation, Princeton, NJ, no. 5 (1998), 71-83
5. S.T.Lin, and R.M.German, *Powder Met. Int.* 21(5), 19-24, 1989.
6. H.J Sterzel., and J.H. Maat,: *European Patent* n° 0 446 708 A2, 28 de Feb. 1991.
7. R. Vetter, M.J. Sanders, L.Z. Zhuang and J. Duszczyk, *Int. J. Powder Metallurgy*, 30 (1) 115-124, 1994.
8. H.H. Angermann, and O. Van der Biest, *Int. J. of Powder Metall.*, 30 (2), 445-452, 1994.
9. B. Levenfeld , A. Gruzza, A. Várez and J.M. Torralba: *Spanish Patent* N° P9802512 (1998).
10. B. Levenfeld , A. Gruzza, A. Várez and J.M. Torralba: *Powder Metall.*, 2000, Vol. 43 (3), pp. 233-237.
11. R.G. Shephard, H.D.L. Harrison and I.E. Russell: *Powder Metall.*, 1973, Vol. 16, pp. 200-219.
12. K.M. Kulkarni, A. Ashurst and M. Svilar: *Modern Developments in Powder Metallurgy*, MPIF Princeton, NJ, 1981, Vol. 13, pp. 93-120.
13. J.S. Oh, Y.L. Kim and H. Moon: *J. Korean Inst. Met.*,1983, Vol. 21, pp. 791-795.
14. W.J.C. Price, M.M. Rebbeck, A.S. Wronski and S.A. Amen: *Powder Metall.*, 1985, Vol. 28, pp. 1-6.
15. C.S. Wright, B. Ogel, F. Lemoisson and Y. Bienvenu: *Powder Metall.*, 1995, Vol. 38, pp. 221.
16. Aguirre, S. Gimenez, S. Talacchia, T. Gómez-Acebo and I. Iturriza. *Powder Metall.*, 1999, Vol. 42, pp.353-357.
17. A. Várez, J. Portuondo, B. Levenfeld, J.M. Torralba. *Mater. Chem. and Phys.*, 67, (2001) 43.
18. A. Várez, B. Levenfeld, J.M. Torralba .G. Matula and L.A. Dobrzanski *Mat. Sci.. Eng. A*, (2003) (in press).
19. L. Castro, S. Merino, B. Levenfeld, A. Várez and J.M. Torralba. *J. Mater. Proces. Tech.* 143-144, (2003), 397-402.
20. G. Herranz, B. Levenfeld, A. Várez, J.M. Torralba, J. Piemme, D. Heaney and R.M. German. *Mater. Sci. Forum*, 426-432 (2003), 4361-4366.