Powder injection moulding at Canada's National Research Council
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The National Research Council is Canada's leading industrial R&D organisation and its Industrial Materials Institute, based in Boucherville, Québec, is a key centre in the ongoing development of powder injection moulding technology. *PIM International* reports on the evolvment of PIM at NRC-IMI and current areas of activity.

The Industrial Materials Institute (IMI) is one of the 20 institutes that forms Canada's National Research Council (NRC). NRC-IMI works in a number of sectors, including metals, polymers, nanomaterials and biomedical materials.

It is under the "Technology and Industry Support" programme that work is undertaken in the field of metal and ceramic injection moulding to encourage and support the commercial development of this technology in Canada.

Founded in 1916, NRC's earliest function was as an advisory body to Government, a role that changed in the 1930's with the construction of new laboratories in Ottawa. NRC grew rapidly during the Second World War, as it contributed to the Allied effort. As a result of this growth, NRC played a major role during the explosion of basic and applied research in science and engineering during the post-war period. Today, research ranges from life sciences to engineering and the organisation employs 4000 staff across Canada.

NRC-IMI's extensive in-house laboratory equipment list includes Arburg 320S Allrounder and Battenfeld Microsystem50 injection moulding machines, various furnaces for sintering in vacuum and hydrogen and a wide range of characterisation and testing equipment.

Fig. 1 An aerial view of the NRC facility in Boucherville, Québec, Canada

Titanium MIM

A key area of investigation internationally is the powder injection moulding of titanium. Titanium parts are, of course, already in production in some countries around the world. However it is the production of commercially pure titanium (CpTi) and Ti6Al4V products for biomedical and aerospace applications that is the focus of the research team at NRC-IMI.

Careful selection of Ti and Ti alloy powders and the use of debinding methods such supercritical debinding are helping to minimise contamination from binder materials and to reduce debinding temperatures, reports NRC-IMI. Significant work has been accomplished for the determination and the ranking of the source of contaminants.

The effect of sintering supports such as boron nitride, alumina, zirconia and yttria has also been studied in order to determine the best candidate for the sintering of high purity titanium and titanium alloys.

As reported in the September 2008 issue of *PIM International*, NRC-IMI is successfully developing a composite porous/dense dental implant based on a MIM titanium dense core and titanium foam coating for the outer surface. The mould design was done with the aid of full 3D injection moulding simulation, with the feedstock made of plasma atomised CpTi-Grade 1 powder and a wax based binder.

Dr. Éric Baril, Research Officer and Project Leader on Ti-MIM at NRC-IMI told *PIM International* "The major challenge when making titanium parts for

Fig. 2 An Arburg 320 S 55 ton Allrounder injection moulding machine in the NRC-IMI PIM laboratory

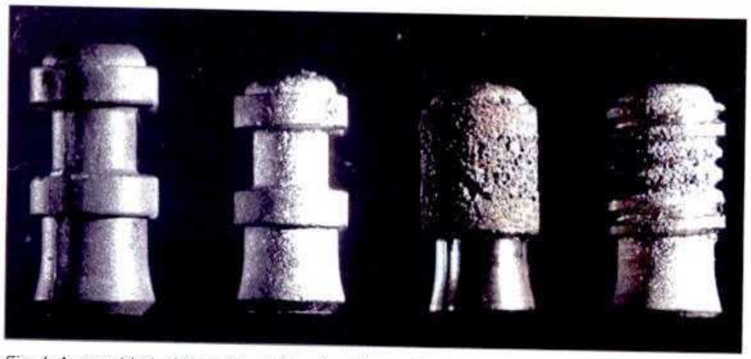

Fig. 4 As moulded, sintered dental implant dense titanium core, coated with titanium foam and machined threads

Fig. 3 Titanium laboratory at NRC-IMI

medical applications is to achieve the required mechanical properties. This is extremely dependant on the chemistry after sintering. The cost of the titanium powder is still high, but it is acceptable for the smallest components. On the other hand, powders with sufficient purity levels can only be found at the highest price range. There is still some development required for low cost, high purity, spherical powder obtained by gas atomisation. Direct electrode melting atomisation seems to be a promising production technology, with the potential to open Ti-MIM applications for larger parts in the medical sector and also in consumer goods sectors."

Dr Baril continued "With all the developments we have achieved on porous materials for biomedical applications, we believe there is a real opportunity for IMI to merge MIM and metallic foams to make new and high value added products for the biomedical and aerospace sectors. In a few months, we plan to begin new activities on co-injection moulding of titanium alloys and titanium foam for orthopaedic, dental and others biomedical sectors."

In addition to the development of titanium MIM, NRC-IMI is also starting work on the injection moulding of hardmetals (cemented carbides) with a view to new applications serving Canada's wood and lumber sector.

Ceramic injection moulding

Early PIM activities at NRC-IMI were focused on the development of a water soluble feedstock system for zirconia. In 1998 a binder system containing 60% by volume of a water-soluble major constituent was formulated and tested. The main components of the binder phase were a low molecular weight polyethylene glycol, an oxidised high density polyethylene, polyvinyl butyral and stearic acid.

A ceramic feedstock containing these binder components and up to 45% by volume of a submicron stabilised zirconia powder was prepared and characterised. Binder removal was accomplished using a two-stage process. Firstly, the water-soluble constituent, polyethylene glycol, was removed by dissolution in water at 50°C. Tests showed that approximately 90% of the polyethylene glycol could be removed from a 2mm thick part during 2 hour immersion in water. The remaining binder constituents were removed using a thermal treatment to 500°C at a heating rate of 100°C/hour. A final sintering step in air at 1500°C produced parts with a density of more than 99%. Although fully developed, this technology failed to see commercial production in Canada.

More recently, micro injection moulding of fine alumina particles has been undertaken in order to study the effect of sintering parameters on dimensional stability. In addition, the use of miniature ultrasonic sensors in the mould cavity was tested to assess quality diagnostics of micro parts and to improve process efficiency.

Dr Sylvain Pelletier, Group Leader of the Powder Forming laboratory at NRC-IMI told *PIM International* "We have a project in the planning stage for ceramic parts for medical instruments. In the near future, we plan to begin the develop of new parts made from biocompatible materials such as pure alumina and hydroxyapatite (calcium phosphate) for the biomedical sector."

PIM modelling

NRC-IMI's process simulation team has built on 15 years of experience in 3D modeling of injection moulding applications, developing state-of-the-art numerical modelling tools for describing material behaviour during PIM applications.

The institute's simulation software for PIM predicts the flow pattern, temperature distribution, the position of the material interface during filling, particle segregation, shrinkage and warpage of the moulded parts. "This", stated Dr Jean-François Hétu, Group Leader Numerical Modelling at NRC-IMI "allows the identification of various causes of defects, such as jets, flow instabilities, formation of weld lines and particles segregation to finally improve the mould design before its fabrication, thus providing means for testing design, concept and moulding process parameters before the costly

Fig. 5 View of the NRC-IMI PIM laboratory showing the Battenfeld Microsystem 50 micro injection moulding machine

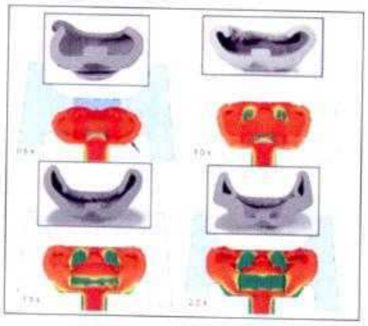

Fig. 6 Partial mold filling compared with simulation results

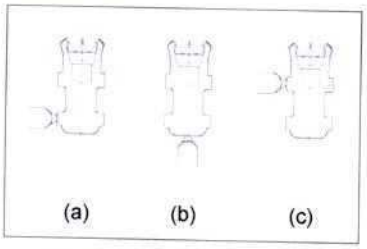

Fig. 7 Investigated gate configurations

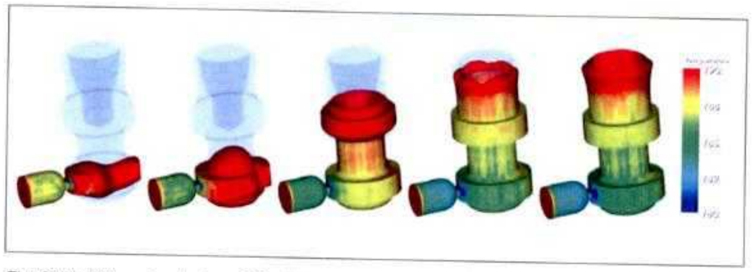

Fig. 8 Die filling simulation: 0.02s filling time of the gate configuration (a)

step of manufacturing a mould".

NRC-IMI's 3D finite-element simulation tool was used to investigate mould filling through a diaphragm gate. The work, in collaboration with Honeywell International, used the numerical simulations to understand experimental observations indicating that the flow of a MIM feedstock through a diaphragm gate can be symmetrical and stable or unstable and severely distorted, depending on the moulding conditions.

The simulation predicted many observed features of the flow (see Fig. 6) - an annular jet, void formation, asymmetrical, unstable flow, and temperature differences indicating eventual unstable flow.

The simulation work indicated that the design and operation of diaphragm gates should (a) exclude combinations of parameters that fall in the unstable flow region and (b) establish early contact of the flowing compound with the external wall surrounding the gate

Fig. 9 NRC-IMI's PIM research laboratory

to prevent formation of an annular jet.

Analysis of the simulation results indicated that the annular jet and associated defects could be reduced by the use of a thicker gate, ensuring contact with the outside wall during gate filling. Subsequent moulding in a tool with a thicker gate showed no voids or exterior weld lines.

Simulation tools can also be used in the mould design stage to improve the filling pattern and avoid mould filling defects. One example is the design of a mould for making dental implants using a titanium powder. Several gate configurations were proposed initially (see Fig. 7) and were validated using the modelling software before machining the optimised configuration. Fig. 8 shows the die filling simulation of the best gating scenario (configuration a).

Other gate configurations show important jetting and flow folding (configuration b) or a weld line in an important load bearing area (configuration c). The mould cavity was machined according to the optimal configuration.

Other simulation related work at NRC-IMI merge the activities of the PIM and Numerical Modelling teams. A 3D numerical solution algorithm for the simulation of free surface flows of dense suspensions, including particle migration phenomena, has been recently developed. Segregation of the solid phase in processes such as PIM affects the rheology of the mixture and therefore the filling pattern. Segregation affects the final properties and characteristics of moulded parts as non-uniform particle distribution leads to non-uniform shrinkage, warpage and non-uniform mechanical properties.

Parallel activities are oriented toward the validation of segregation modelling. NRC-IMI x-ray micro-CT is being used to measure the solid fraction distribution in injection moulded parts as a function of the moulding conditions.

The outlook for PIM in Canada

The team at NRC-IMI believes that there is significant potential for metal and ceramic injection moulding in Canada.

Dr Pelletier commented *PIM International* "In Canada, there are only few companies producing MIM parts. The more active companies are Columbia Plastics in Vancouver and Maetta Sciences in Boucherville, near Montreal. There are also a number of companies doing PIM in-house for their own products, including firearm components and carbides components."

"The largest potential markets for PIM in Canada are however the aerospace and medical sectors. We believe that there is a great potential to start companies on PIM in Canada and it is hard to understand why there are so few Canadian companies involved in PIM when the annual growth rate of this technology in USA is always between 10 and 15%! We at IMI believe that more needs to be done to show the real benefits of this technology to Canadian industry and the design community."

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