Conference Reports

Powder Metallurgy (PM'89) in San Diego

By Rainhard Laag*

1. Introduction

At the PM'89 conference, held 10–14 June 1989 in San Diego, USA, the present state of metal powder injection molding (MIM) in the USA was presented in five sessions, each with 15 presentations where the leading researchers, producers and consumers of MIM parts demonstrated impressive progress in development and application. Currently, the most extended activities in institutional research and development are being made at Rensselaer Polytechnic Institute (RPI), Troy, NY. A considerable number of companies are also developing improved processing, but it is mostly of a proprietary nature.

Before discussing the individual presentations given at San Diego, the general state of the art will be briefly described. The development of new, high performance metal alloys requires rapid solidification process to control the microscale homogeneity of the chemical constituents. Investigations of powder production are, therefore, closely connected with the development of advanced forming and consolidation processes. Two examples of recent developments are Metal Powder Injection Molding (MIM) and sinter-hot isostatic pressing (Sinter-HIP). Both, as concepts, are attempts to conserve the physical properties of powders on a macroscopic scale. Between concept and finished part, however, the processing steps change the initial conditions of homogeneity and freedom from exogenous materials—both solids and gases.

Sinter-HIPing of powder compacts is a pressure assisted consolidation technique and is applicable to a wide size range of parts made from powders. It is beneficial to form a stable green body either by cold isostatic or by uniaxial pressing. Advanced metals like superalloy and intermetallic phase powders do not compact well. Therefore, the powder must be filled in glass or metal containers for consolidation. This encapsulating process increases the production costs (an additional step) especially since the containers are not reusable after HIPing. The advantage of Sinter-HIP consolidation is that relatively coarse powders can be used without any contamination being caused by binders or additives. This simplifies the conservation of the physical properties of the powders and helps to achieve an optimum in mechanical properties.

MIM is a hydrostatic-pressure forming and consolidation process used to produce small and complex shaped parts inexpensively in large quantities. A mixture of approximately 60 vol.-% fine metal powder and 40 vol.-% binder and lubricant can be formed much like a conventional plastic. The use of waxes like polyethylene or other thermoplastics provides the rheological basis allowing the mixture to flow into undercuts and corners in a way that the uniaxial pressing of lubricated metal powders cannot achieve. Dependent on the shape complexity, this process needs very fine powders to achieve homogeneous form filling. The forming process is a combination of thermoplastic pressure casting and conventional powder metallurgical technologies. Like all known processes, MIM has some advantages and disadvantages. The limitations are mainly in the compatibility of the powder/binder interface, the complete binder removal and the pressureless sinterability of the metal powders to near full density.

In order to combine the advantages of MIM with its achievable shape complexity with, for example, the high performance of superalloy and intermetallic phase materials, it is necessary to enhance the ability of MIM either to process coarser powder particles or to develop powder production techniques that allow the inexpensive preparation of fine prealloyed powders on an industrial scale. Because of MIM's potential to produce large quantities of parts, the use of fine powders produced by conventional techniques is currently much more expensive than the use of coarse powders. The savings are twofold, in powder costs and in economies of scale which often result in an exponential increase in cost effectiveness with the quantity of produced parts.

The main disadvantage of MIM is that the savings in processing are connected with an increase in impurity of the part because group III–IV metallic elements and refractory metals are highly reactive towards anionic elements like oxygen or nitrogen. A reduction of the powder particle size far below 100 μm increases the specific surface area of the powder so that the content of impurities can reach 1–2 wt.-%. At the higher temperatures necessary for consolidation, these impurities diffuse to grain boundaries and promote brittleness by forming strong oriented covalent bonds. The me-

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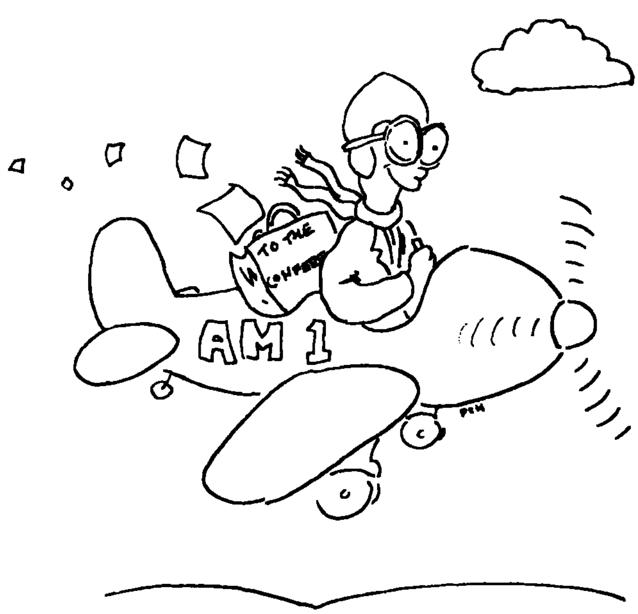
ADVANCED MATERIALS

chanical properties are very dependent on the amount of impurities. Another advantage of using coarse and spherical powder particles is their lower mechanical friction and their higher packing densities. This allows a higher powder loading in the binder. An additional increase in density can be achieved if special particle size distributions like bimodal or trimodal distributions are used. The higher the density of the green shape compared to the theoretical density, the smaller is the resulting shrinkage during sintering.

Today, the use of coarser particles is, however, limited. This has three main reasons:

- Super solidus sintering does not work as well with coarse particles because the surface area decreases with an increase in particle size.
- The internal friction of coarser particles is lower. After debinding, the parts are very sensitive to fragmentation before presintering. Typically the debinding process ends at much lower temperatures than necessary for presintering. This difference is especially noticeable for high temperature alloys.
- Solid state sintering of single phase high temperature alloys like intermetallic aluminides does not work because of the low diffusion rates in ordered lattice structures.

The overall advantage to the processing of fine powders is the superior form filling during molding which allows more complex shapes to be molded and gives a homogeneous distribution of binder and powder in the green body. Spherically shaped particles simplify the critical debinding process, enhance sinterability, and cause uniform dimensional shrinkage which reduces effort in tool design, quality control and finishing of the parts. The application of prealloyed powders achieves the additional advantage of a more homogeneous microstructure after sintering compared to the component or multicomponent powder mixtures.



Injection molding consists of four basic steps: powder-binder-formulation, injection molding, debinding and sintering. Although each step appears independent they are in some ways all related. Currently the interchange between the processing steps is not understood adequately. The procedure of producing parts is based on empirical results which are connected with long and expensive trial and error investigations. Up to now it is clear that the most sensitive factors are the powder properties, the constituents of the binder, the powder binder interfaces and the process variables of debinding and sintering. All these problems were addressed by the presentations in San Diego.

2. Powders

In the section on powders the need for the production of ultrafine powders below 20 µm in diameter was outlined. Three different atomization processes and one high energy milling process were shown. The microatomization of GTE Products Corporation, presented by *M. Pliwal* and *R. J. Holland*, produces spherical powders of tungsten, copper, aluminum and stainless steel by remelting sintered powder mixtures and forming prealloyed powders in a plasma jet. This relatively expensive procedure allows the production of prealloyed powders made from alloys with very high melting points. Another method, ultrasonic gas atomization, provides particles below 45 µm. The volume fraction of powders below 20 µm was small in comparison to the yield of other methods presented. The reason may be the low gas-mass stream that divides the melt stream into the required fine droplets. *K. P. Kao* (Defense Metallurgical Research Laboratory) presented fluid energy milling which he has used to process iron sponges to fine particles of spherical shape. These have a shorter debinding time and a better sinterability in comparison to iron carbonyl powder. With the exception of the ultrasonic gas atomization, all processes described in this session had the same disadvantage in being an additional production step in working with MIM and being expensive when larger quantities are produced.

3. Process Variables

The presentation by *D. Lee, K. F. Hens* and *L. A. Najmi* (RPI) was focused on the understanding of the conditions under which the injection molded parts could be produced. They have developed software packages which allow the examination of the form filling conditions with respect to the powder/binder mixture. The basic concept of the calculations is known from plastic injection molding. Additional difficulties arise due to the variation in molding temperature. If the binder shows a rapid change in viscosity, and segregation occurs inside the die, then density variations occur in the molded parts. The optimization of process parameters by extensive computer calculations demonstrates the need for careful process control. The second presentation of this session (*D. A. Issitt*, European Advanced Materials Institute) illustrated the influence of powder characteristics, binder selection, rheology of the mixture, green strength, debinding, shrinkage and tolerance on the properties of the finished part. Here, the audience was told that only the carefully adjusted optimization of each individual process parameter will result in a product with the properties required for advanced technological applications. In the last presentation *T. Tonomura* (Multimaterial Japan Limited) illustrated factors that affect size tolerance and physical and mechanical properties. The high performance of injection molding was demonstrated even for cases where elemental powders were used.

4. Binders

This session dealt with the key problem of successful injection molding, the evaluation of binder systems and their compatibility to the surface chemistry of the powders. The presentation "Optimization of the Powder–Binder Mixture for Powder Injection Molding" given by *R. M. German* (RPI) was the highlight of the injection molding sessions. Based on his long term practical experience, he gave the audience an assessment of the powders and binders currently used in injection molding. A body of mathematics was described which is used to predict and optimize the design of injection molding powder/binder mixtures. Several factors were included in calculating the inherent packing density of a powder. It was shown how the packing characteristics are dependent on particle size, distribution and shape, agglomeration, surface active agents, internal powder porosity, mixture homogeneity, interparticle friction and the size of the mold. Modeling of the viscosity of the powder binder mixture, using several equations developed for concentrated suspension behavior was demonstrated. The method resulted in a predicted optimal mixture for injection molding for a given set of powder and binder attributes.

After the discussion of particle characteristics, the second presentation "Requirements of Binder for Powder Injection Molding" by *C. I. Chung* (RPI) tried to illuminate some of the secrets in binder development. As expected the specific requirements of a binder depend on the complexity, size and metallurgy of the desired part. The performance of the binder can be enhanced by mixing or blending the binder with other materials. The molecular length of all components of the binder must be smaller than the interstitial free path length between powders in order to obtain a microscopically uniform mixture.

5. Research

The purpose of this session included the evaluation of new materials applicable for injection molding in the future. Highly sensitive materials like titanium powder were shown to be moldable if the binder does not violate the surface chemistry of the powder. In the presentation given by *K. Ameyama* (Ritsumeikan University) on injection molding of fine titanium powder it was reported that binder consisting mainly of polybutylmethacrylate can be completely removed after molding and the titanium sintered to a density of about 94% theoretical density. Impact properties were measured and fracture was characterized in order to evaluate the processing (molding, sintering). Correlations between processing parameters and mechanical properties were established.

"Supersolidus Sintering of Coarse Powders and Its Application to Injection Molding", given by *P. F. Murley* (RPI) was an attempt to evaluate the applicability of injection molding for coarse powders. Here, obtaining a homogeneous distribution of binder, and debinding were the most critical steps of the process. Experiments with higher volume fractions of metal in the mixture showed that the disadvantages caused by a homogeneous binder distribution—pore formation during molding and incomplete debinding even after long cycle times—counteract the advantages expected from higher metal fractions. Microstructure development during super solidus sintering of bronze and nickel alloy powder was found to be highly sensitive to local temperature variations.

6. Applications

Three presentations covered the evaluation of profitable applications of MIM. Nickel–Steel parts for the automotive industry, office equipment, oil drilling and camera components were shown. Other profitable examples made from stainless steel are parts for computer equipment, dental instrumentation, orthodontic devices, chemical components, marine equipment, aerospace equipment and a Fender bass guitar. Parts made from some exotic materials like Kovar, Tungsten, Invar and Molybdenum were supplied to the electronics industry in the form of hermetically sealed packages—headers, eyelets, flanges, heatsinks and bases.

The problem of building inexpensive prototyes of mold forms for experiment and process optimization was sketched. A minimum production quantity of about 20,000 pieces per year was thought necessary to amortize tooling costs.

The PM'89 conference in San Diego covered the present state of the research, development and production of MIM. Much effort was made to develop processing and new applications for parts which should be very competitive with other powder metallurgical routes and castings. Standard processing known from plastic injection molding has been successfully transferred to the production of small parts made from carbonyl iron and nickel powders. The transition to more complex materials and the systematic evaluation of new compatible binder systems needs a lot more scientific work towards understanding the principles and dependency of the process. An ideal process would use inexpensively produced, gas atomized, fine and small sized powders of about 10 μm diameter with a compatible multicomponent binder system which provides a mild decomposition at relatively low debinding temperatures. Debinding, presintering and consolidation of injection molded parts in a Sinter-HIP furnace offers the possibility of a one step heat treatment which avoids handling of the debinded parts. Full density can be achieved by pressure assisted final stage HIP. In the USA, a multimillion dollar project, part of the Advanced Powder Processing program at Rensselaer Polytechnic Institute, is the beginning of a move from empirically based process development to a systematic development of the theoretical background.

The basic elegance and simplicity of the injection molding process must not lead to the assumption that fully dense parts of predictable final dimensions are produceable as easily as in plastic injection molding. Up to now the main problems arise after the actual molding, during debinding and sintering. Only complete control over all process parameters will result in high quality reproducable parts.

Macromolecular Physics in Lodz

By Hugh J. Byrne *

To the French Cistercians who founded the Abbey in Sulejow Lodz, Poland, the terms charge transport and activation energy had little meaning, and seldom were they to be seen meandering through the grounds digesting the importance of bipolarons and multiphoton resonances. However, for one week in September, the serenity of this decaying 12th century house of learning was disturbed as it played host to the 21st Euro-Physics Conference on Macromolecular Physics, held from 4–8 September.

The conference was focused on the topic of "Electrically and Optically Active Polymers", a field which has attracted the expertise of a diverse range of scientists including polymer and solid-state physicists as well as physical and synthetic chemists. Interest in these materials was generated by the discovery that it was possible to make thin films of polyacetylene, and was increased by the subsequent discovery that the conductivity of this material could be increased to metallic values by chemical doping. Since then, a great deal of activity has produced a large range of processible organic polymers whose properties have been studied. Of similar interest to the electrical properties of these materials are the optical and, in particular, the nonlinear optical properties. The highly delocalized π-electron backbone of these polymers should give rise to large nonlinear susceptibilities which may render these materials a possible alternative to inorganic materials in the developing fields of all-optical switching and signal processing. This conference was designed to review the progress and problems of understanding the nature and origins of these materials properties.

The key-note address was delivered by the conference chairman, *Prof. M. Kryszevski,* from the Polymer Institute of the Technical University of Lodz, who outlined the objectives of the conference. Particular importance was attributed to the necessity for a greater understanding of the dependence of the electrical and optical properties of these materials on their structure and morphology. In a field where the range of materials available is ever increasing and where the polymer morphology may vary from that of single crystal to that of solution, an understanding of this structure-properties relationship is as important as understanding the microscopic molecular properties. Indeed, the problems facing "homo polymerius" were effectively portrayed by *V. Enkelmann* from Mainz, FRG. In his presentation he described the number of possible inter and intra chain effects contributing to a bulk measurement of any polymer parameter. He stressed the need to control the macroscopic conductivity by means of molecular parameters. The approach taken was to move towards single crystal materials in which interchain hopping is controlled by the size of the molecular structure. In polythiophene and polyparaphenylene, substitution was employed to systematically control the interchain distance by steric effects. The temperature dependence of the conductivity was found to increase as the size of the substituent increased, indicating an increasing activation energy although the conductivity itself decreased. The transition from the dominance of interchain effects to that of intrachain effects was thus shown, and an empirical relationship between the activation energy and the interchain parameter was presented. A similarly strong correlation between the

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