REVIEW OF THE MIM INDUSTRY: RECENT TRENDS IN POWDER SIZE AND COMPOSITION

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ABSTRACT

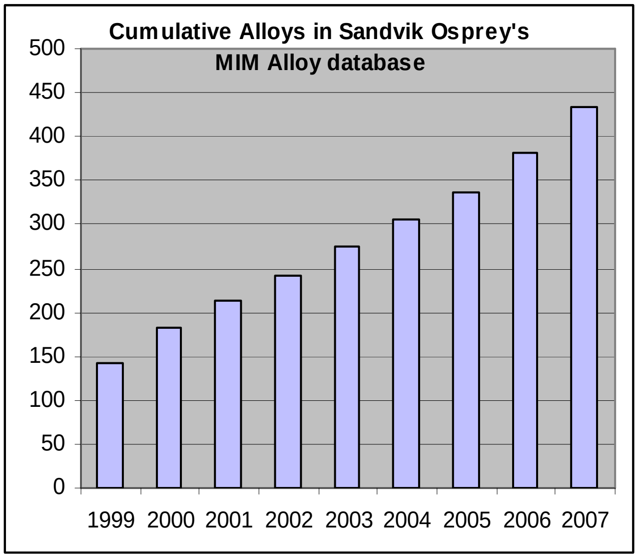
This paper provides a review of the Metal Injection Moulding (MIM) industry in terms of market size and types of materials currently used. Developments are highlighted in new materials for growth sectors, including fine copper powder for thermal management, nickel base superalloys for hot service environments and cobalt alloys for medical applications. An analysis is presented of competing trends in particle size where coarser powders for low cost/high volume applications are gaining popularity and finer sizes are specified in low volume applications demanding superior precision and material properties. The advantages of master alloy powders are considered in relation to control of distortion and consistency of sintering response vs prealloy powders. The benefits of finer average particle size and narrower alloy chemistry ranges are important in achieving high quality components but it is clear that for other applications, coarser powders can be cost effective.

INTRODUCTION

While patterns of geographical activity in MIM may be changing, with a gradual shift of emphasis of activity and investment from North America to Asia, evidence continues to point to well above average growth in the global market. This has been helped by ambitious new entrants in lower cost economies seeking to displace competing manufacturing technologies. With new equipment, rapid design turnaround and relatively low manufacturing costs, Asia is well suited to dominate in the large consumer, communication and computer market sectors. The backdrop of rising metal prices, whilst undesirable in terms of constantly changing cost bases is, in a broader sense, advantageous to all producers engaged in net shape technologies such as MIM. Those producers seeking to push the envelope of applications addressable by MIM are being supported by powder and feedstock producers who are offering a wider range of cost effective products. Likewise, production plant technology is advancing in order to offer closer process control and reduce overall conversion costs, and alloy powders processed are dominated by stainless steels. To compete effectively, western producers are taking advantage of greater automation, robotics and optical quality control facilities and are focusing on high added value applications, including medical and aerospace components.

As MIM technology becomes more pervasive and designers become more confident in the capabilities of MIM, the envelope of alloys, geometrical complexity and application type is expanding steadily. In recent reviews, Bloemacher [1] has highlighted a number of growth areas in the industry driven by a need for greater precision, lower cost and improved material properties. The inherent flexibility provided by a powder manufacturing route means that individually tailored solutions are available. The role of the powder manufacturer is in offering a range of options which can be applied to particular applications development. In relation to established, high volume applications, improved process control to achieve narrow chemistry ranges in sensitive alloys and the use of master alloys gives better control of chemistry and hence sintering characteristics. For complex and large parts, there is an increasing realisation that limitations imposed by part distortion can be reduced by the use of finer powder size fractions and/or master alloys in combination with carbonyl iron. It is also the case that master alloy approaches can improve on operational costs and dimensional control. In other circumstances where cost is the ultimate driver there is a trend towards the use of coarser sieved powders, specifically -32 and -38µm fractions, to reduce material costs.

Figure 1 illustrates from Sandvik Osprey's own experience that the number of alloys quoted for MIM applications has grown significantly and with it the volume and value of quotations for individual orders. These trends, together with an industry average growth rate of ~15% [1] has been a driver behind Sandvik Osprey's commissioning of a new atomising plant at its Neath facility in the UK.



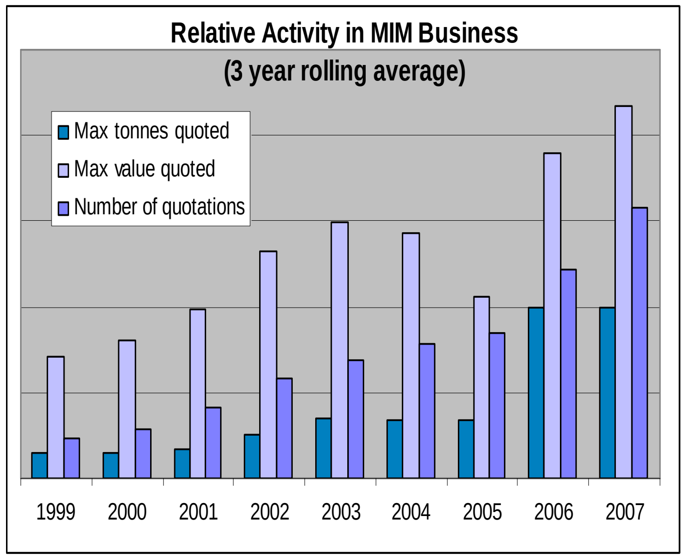


Figure 1(a) Number of alloys in Sandvik Osprey's MIM alloy database and (b) Growing activity in size and number of quotations.

Underlying these growth trends are innovations in atomising processes, product types and sizing. While stainless steels still dominate the MIM market, recent developments in the production of high quality Co, Cu and Ni base alloy powders are opening up new fields of application in medical, thermal management and aero sectors. In this paper we illustrate some notable developments both in these high performance niche sectors as well as higher volume automotive and electronic / consumer markets.

High Temperature Ni-base alloys for Automotive & Aerospace Parts

As designers strive for increasing engine efficiency for transport applications, greater demands are put upon the hot section of the engine and inevitably this means the use of more sophisticated alloys. Often this means that high Ni stainless steels are specified and these include alloys such as 310, HK-30 and GHS-4. A significant proportion of the diesel powered engine market (~50%) has now embraced MIM technology and an array of components is now made by MIM: e.g fuel injection nozzles, fuel line connectors, turbocharger vanes, vane geometry adjustment rings and turbine rotors. Many of these components operate at elevated temperatures and require heat resistant materials including the aforementioned alloys as well as 420 stainless (prealloy or via the Fe50Cr master alloy route) and new developments involving superalloy materials [2].

There is increasing interest from the aerospace industry for powder / net shape manufacturing route in part because of cost considerations but also because increasingly sophisticated multi-component alloys cannot be made via conventional casting methods without gross segregation. Items under consideration include fuel injection components and combustion chamber temperature sensors. Stucky [3] reported development is ongoing for Ni base components for Parker-Hannifin gas turbine fuel injector system in IN625. A powder prealloy has been used and parts were sintered in a hydrogen furnace. Mechanical properties and corrosion performance were reported to be at least equivalent to the cast alloy inferior to wrought properties due to the presence of grain boundary carbides. Further refinement of carbon balance and sintering cycles is required.

Beyond IN625, some of the advanced alloys under development these alloys rely on additions of Ti and Al in order to obtain high strength γ/γ' microstructures and these elements are prone to react with the atmosphere during melt processing. Expensive Ar atmospheres have hitherto been employed to achieve the requisite alloy chemistry and restrict nitride formation, but recent work on melt practices at Sandvik Osprey has established techniques for producing high quality powders with limited use of Ar atmospheres which can make a significant impact on powder costs.

High Quality F75 Powders for Orthodontic & Medical Applications

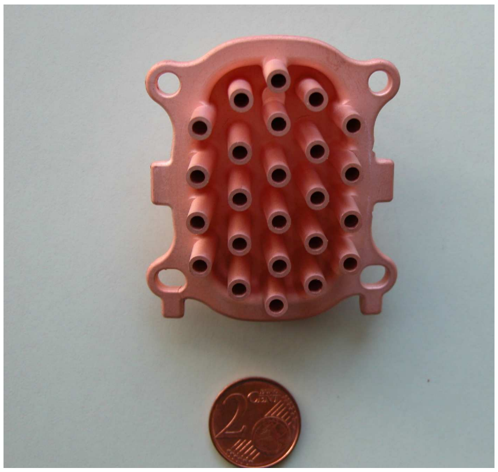
The overwhelming transition from traditional investment cast F75 orthodontic brackets to components made by MIM is rightly heralded as one of the outstanding successes of MIM. In a recent review [4], Williams highlights some of the successes of MIM in the dental sector including a number of past MPIF parts winners. Currently, Ni-free stainless steel compositions are in demand in order to avoid complications with patients prone to Nickel sensitization. Alongside Panacea alloy, which has been a popular choice for several years, there are now alternatives under development based on the FeCrMn system where the aim is to achieve high strength but low modulus. In order to be classified as Ni-free, products must contain <0.1%Ni which is much less than the normal F75 spec of 0.5% max. Sandvik Osprey has collaborated in the development of alloys complying with new strict Ni limits and these have been used successfully already in some cases and are going through final approvals in other cases. In other cases, the alloy powder has been CE marked.

In addition to orthodontics, F75 and stainless steel alloy powders are exploited in other medical and dental applications. For medical products, there is the potential to progress from an established base in small orthodontic brackets and medical instruments towards larger in vivo implants. In spite of a natural conservatism related to lengthy qualification processes, medical parts manufacturers and those who have traditionally relied on investment casting for larger implants are giving attention to MIM options. Increasing raw materials costs and the costs of machining such hard alloys weigh in MIM's favour. Whilst it is unsure whether MIM is capable of delivering satisfactory large scale prosthetics, there are smaller joints and partial implants that could come within scope. Already today, coarser versions of F75 MIM powders are used as feedstock for HIPping and also for rapid prototyping technologies for manufacture of dental bridges, tailored caps and also for bespoke implants made directly from CAD files by laser or electron beam processes.

For CoCrMo alloy powders, C content is critical in determining mechanical properties [5]. At low levels, typical of MIM parts, CoCrMo has a small grain size and high fatigue strength but poor wear resistance. If C levels are raised, wear resistance improves at the expense of fatigue resistance. Cr carbides can reduce ductility if allowed to form at grain boundaries and high temperature solutionizing 1200degC followed by rapid cooling is generally employed. Nitrogenation of low C alloys is an alternative approach to achieving high performance and Johnson et al [5] have reported the use of different sintering atmospheres to control strength and ductility levels with a water atomised powder. Sintering in a nitrogen atmosphere enabled strength levels comparable with wrought F75 to be achieved (0.2% yield strength ~680MPa, UTS ~1000MPa, Hardness ~26HRC and Elongation to failure ~18%) However, initial O levels are high (0.17-0.29%) and there is concern that inclusions in the feedstock will be detrimental to fatigue performance. F75 powders made by gas atomisation typically have oxygen levels <0.02 and satisfactory levels of cleanliness have been achieved for in vivo applications.

Fine, low Oxygen Cu powders for Thermal Management

Another sector where compact performance is critical is in thermal management of electronic devices and the proliferation of LED technology is exacerbating these demands. Zauner et al. [6] recently investigated the potential for use of complex MIMed copper components as heat transfer substrates for LED arrays as opposed to simple Cu parts or bulkier Al substrates. Two fine copper powder grades (80%-22um and 90%-31um) with low oxygen (<1000ppm) were found to be suitable for injection moulding of complex shaped substrates. Both materials reach more than 85% of the thermal conductivity of a pure copper standard.



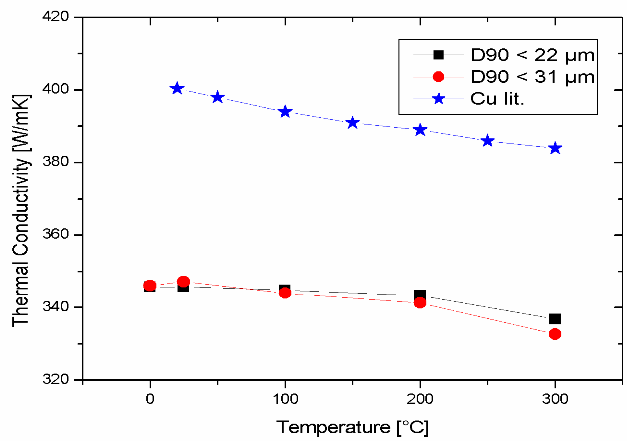


Figure 2. (a) Complex demonstrator substrate for thermal management of LEDs in MIM'ed copper, (b) thermal conductivity vs temperature of MIM'ed copper compared with pure, annealed copper (Ref. 6).

For the LED heat sink demonstrator which was the subject of the study a four-fold increase in energy dissipation could be achieved compared with a pressure die-cast solution in aluminium. Therefore, copper MIM offers huge potential for further development in the future as thermal management becomes more and more of a bottleneck when it comes to increasing the power density of LEDs. However, this comes at a cost. For the heat sink design shown in this study, the initial solution which is suitable for one LED mounted on it costs around 0.70 € each in numbers of larger than 100,000 per year. In comparison, for the optimized material fabricated using MIM it was calculated that the costs could be up to 2.20 € each for such volumes but could be significantly less in a suitably equipped MIM facility. Therefore, this solution is only justified when the power density of LEDs further increases. There are indications that this trend is progressing and it is just a question of time before the current material-manufacturing combinations will reach their limits.

HIGH VOLUME APPLICATIONS IN STAINLESS STEELS

Stainless steels still represent the large majority of MIM products and alloys such as 17-4PH, 316L, 420 and 440C remain among the most popular grades As MIM is being applied increasingly to production of high volumes of larger parts, there has been a trend towards the use of continuous furnaces in larger producers' premises. A recent estimate of the number of continuous sintering furnaces operational today is 55 worldwide [1] equivalent to 3,000t capacity per annum if fully utilised. Batch furnaces remain most versatile for processing a wide variety of products, but unit costs for furnacing can be higher depending on the throughput required.

Refractories used in continuous furnace units are high temperature oxides based on alumina (Al_2O_3) and, like all refractories, may be susceptible to chemical attack from species liberated during sintering. Volatile elements from common alloy systems include e.g. Cu and Mn from 17-4PH. Elements will evaporate at a rate which is dependent on the alloy content, the furnace environment (composition and pressure) and the sintering temperature. There are two possibilities to reduce the problems associated with elemental loss: first to use a powder with lower Cu, Mn levels (e.g. 15-5PH) or second to provide a powder which will sinter at lower temperature. The composition can indeed be controlled to a degree using master alloys as explained below. Alternatively, the use of finer prealloy powders as reported by Merz et al (7) can enable lower sintering temperatures to be employed.

The evaporation of Mn poses a different problem in that the Mn can react with the refractory to form spinel phases $Mn(Al)_2O_4$. These occupy a larger volume than the parent alumina and this leads to stresses in the refractory which ultimately leads to cracking and spalling of refractory. It is costly to refurbish a furnace and the downtime involved is also significant therefore prolonging life is important. Master alloy powders can help alleviate this problem by reducing the levels of Mn in the finished stainless steel. Table I shows the impact of dilution of the concentrated master alloy with carbonyl iron (1 part MA vs 2 parts carbonyl) on final Mn levels. The carbonyl Fe has no significant Mn, so the Mn in the master alloy is effectively one third of that in the master alloy which is likewise approximately one third of the level in a prealloy alternative.

Table I. Comparison of final alloy compositions via Master Alloy and Prealloy Routes

Alloy element	17-4PH Alloy			316L		
	Typical Prealloy	Master alloy MA range	Prealloy from MA	Typical Prealloy	Master alloy MA range	Prealloy from MA
Cr	15.5-17.5	49.0-52.0	16.3-17.3	16.0-18.0	51.6-53.4	17.2-17.8
Ni	3.0-5.0	13.0-15.0	4.3-5.0	10.0-14.0	37.0-39.0	12.3-13.0
Mo	-	-	-	2.0-3.0	6.6-7.9	2.2-2.65
Cu	3.5-5.0	12.0-12.7	4.0-4.25	-	-	-
Mn	0.7	2.0	0.67	2.0	1.0	0.33
Si	1.0	3.0	1.0	1.0	0.5	0.17
C	0.07	0.07	0.027	0.03	0.03	0.01

The master alloy approach for 17-4PH, 316L (and 420 for that matter, where 3 parts carbonyl iron are mixed with 1 part Fe50Cr master alloy) can also have potential benefits in terms of final product consistency. For example, for 17-4PH, the typical Cu level in the master alloy ranges between 12.0-12.8% which means that in the final alloy, the range of Cu is between 4.0 and 4.25%Cu. This is a much narrower range than normal for the prealloy (3.0 – 5.0%Cu) and this translates into more consistent sintering response, part uniformity and mechanical properties [8]. Clearly, the narrower the processing temperature range, then the narrower should be the scatter of properties from batches of parts.

The automotive sector has shown perhaps the highest growth rate in recent years [1], particularly in Europe and Asia. The industry is characterised by demand for high volume product and process consistency and cost pressure. Close control of chemistry is also a feature of particular alloys which are known to be sensitive to variable sintering performance. An example is high hardness corrosion resistant stainless steel 440C where tight carbon control is usually required to give consistent part properties and to avoid coarse grain boundary carbides that otherwise impair corrosion resistance. Sandvik Osprey specialises in controlling C level in its alloys and can make homogeneous batches up to 3,000kg in size to give high volume process control. Control of batch to batch repeatability of +/- 0.02C is possible and large batch sizes favour consistent properties. In the 440C system, an alternative approach which has been reported by Wohlfromm et al. [9] is to use a 3%Nb addition to the alloy to control sintering response. This enables continuous sintering in a N_2 atmosphere at +/-10degC. The authors recommend C is controlled to <1.25% to avoid formation of coarse carbides.

Furnace running costs are related to the sintering temperature, not only because of the cost of energy but also because the rate of degradation of furnace consumables is dependent on temperature. Lowering sintering temperature would be advantageous but this requires either greater percentages of liquid phase or a finer overall size distribution with greater surface area driving the sintering process. The use of master alloys offers some potential in this respect because blending gas atomised master alloy powder (see typical size ranges in Table I below) with fine carbonyl Fe (median typically 4 microns) gives an overall finer average particle size which sinters at lower temperature. The finer overall particle size also offers other advantages in terms of reduced part distortion. Some part geometries are more prone to distort than others and it is found that the higher surface area provided by finer powders leads to more internal friction and rigidity both in the green and debindered state.

Table II. Typical range of commercially-available particle size distributions for MIM applications. The finer grades offer better surface finish and greater precision but these benefits need to be weighed against the extra cost of such powders whose atomising yield is progressively reduced.

Size range	Median, D50	Separation method	Applications
80% -5	2.8	Classification	MicroMIM: medical, mechanical
90% -10	5.0	Classification	Medical
90% -16	8.0	Classification	Medical, auto
90% -22	10.5	Classification	Master Alloys, mechanical, consumer
80% -22	11.3	Classification	Auto, electronics, consumer
-32um	12.0	Sieving	Auto, consumer
-38um	13.0	Sieving	Auto

Binder and feedstock formulation need to be adjusted according to size of powders in order to achieve adequate melt flow index and injection performance but it is generally found that during debindering and sintering, finer distributions give better processing characteristics in terms of resistance to distortion [10]. Zauner et al. [11] investigated the role of size and powder type on dimensional variability in 316L and found that water atomised powder gave lower dimensional variability vs gas atomised powders which they attributed to greater interlocking of particles. Likewise a 90%-16um gas atomised powder gave better stability compared with a 90% -22um powder. In a later study of the effects of powder type, Stucky et al. [12] found that for 316L and 17-4PH, lowest distortion could be obtained using a gas atomised master alloy plus prealloy gas atomised mix. This could be significant in the drive towards larger MIM parts and/or those which have high aspect ratio or significant bending moment which are most prone to slumping and setter drag. It is not uncommon now for parts producers to seek an optimum blend of gas and water atomised powders to give the best balance of flow, distortion, shrinkage and cost.

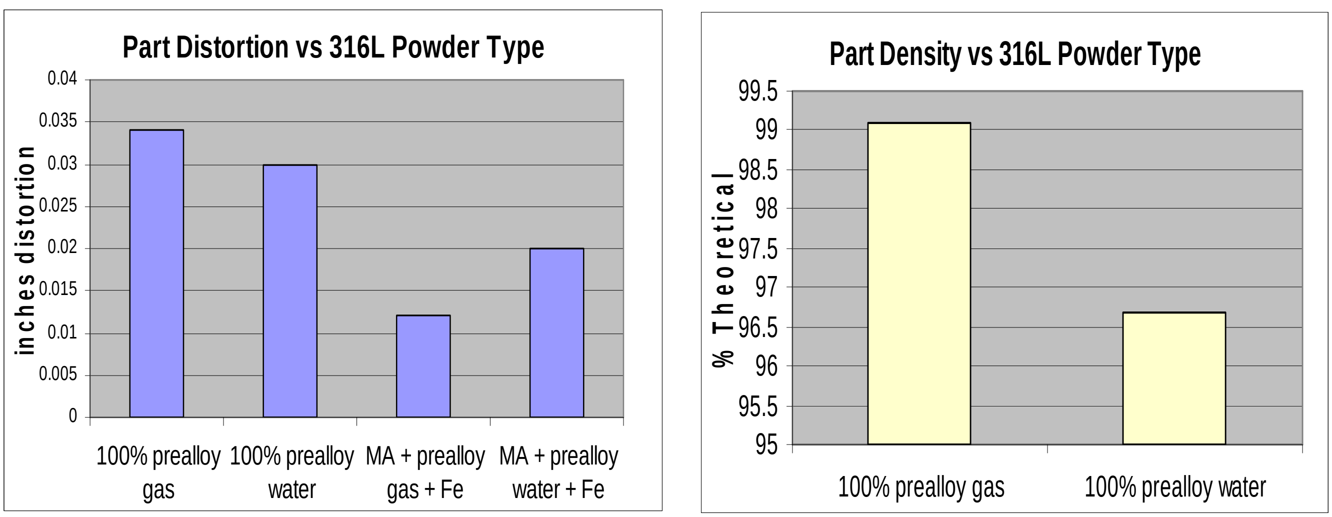


Figure 3. (a) Effect of Powder Type on 316L part distortion and (b) Effect of Powder Type on Theoretical Density (After Ref. 12)

SUMMARY & CONCLUSIONS

The MIM industry is growing in volume and diversity limited in some instances by genuine technical barriers (e.g. in achieving desired tolerances for large parts) but also by ambition and creativity in challenging established manufacturing norms. Some of the often-cited concerns – raw materials availability and prices – continue to be eroded by the actions of suppliers who have consistently invested in capacity and product development. MIM's true value is apparent in high performance niches where performance is critical and examples include Co alloys for medical, Ni base super alloys for high temperature service environments and Cu for thermal management applications.

In other respects, for high volume, mass produced items, low cost and consistent properties are essential where the benefit to the customer is a consistent, high yielding process with low quality control costs. Specific examples shows that gas atomised powders are capable of delivering narrow carbon ranges in ferrous alloys and low oxygen contents. Other advantages stemming from a master alloy approach to MIM include the ability to achieve greater chemical and therefore processing consistency, lower distortion, higher part precision and potentially greater longevity of refractories in continuous furnace operations. By adopting a master alloy route it may prove possible to further extend the size range of components with control of distortion and narrower property bands achieved.

REFERENCES

1. (a) & (b) M. Bloemacher, "Key Trends in the Global MIM Industry", PIM2007 Orlando, Publ. MPIF, Princeton, NJ, USA and EuroPM 2006, Ghent, Publ. EPMA.
2. P.A.Davies, G.R.Dunstan, A.C.Hayward & R.I.L.Howells, "Development of Master Alloy Powders including Nickel-based Superalloys for MIM", Advances in Powder Metallurgy and Particulate Materials, Las Vegas, Publ. MPIF, Princeton, NJ, USA, Part 8: 1-12, 2003.
3. M.Stucky, M.Bulger, H.Zhang, T.Stephenson, B.Hartley, "Comparison of MIM Prealloyed and Elemental Inconel 625"cdcd, Euro PM 2006, Ghent, 91-95, Publ. EPMA.
4. B.Williams, "Powder Injection Moulding in the Medical and Dental Sectors", Powder Injection Moulding Intl., 2007(1), 12-19.
5. J.L Johnson, L.K.Tan, "Processing of MIM Co-28Cr-6Mo", Advances in Powder Metallurgy and Particulate Materials, Montreal, Publ. MPIF, Princeton, NJ, USA, Part 4 13-21, 2005.
6. R. Zauner, R. Nagel, E. Neubauer, K. Portschy, P.A Davies, M.A.Kearns, "Development of Copper MIM Powders for Thermal Management Applications", EuroPM 2006, EPMA, Ghent, 71-77, Publ. EPMA.
7. L.Merz, S.Rath, G.Oerlygsson, R.Ruprecht & J.Hausselt, "Improvement of Microstructures & Properties of Micron-sized PIM Parts", Advances in Powder Metallurgy and Particulate Materials, Chicago, Publ. MPIF, Princeton, NJ, USA. Part 4: 11-22, 2004.
8. P.A.Davies, G.R.Dunstan, D.F.Heaney & T.J.Mueller, "Comparison of Master Alloy and Prealloyed 316L Stainless Steel Powders for MIM", Advances in Powder Metallurgy and Particulate Materials, Chicago, Publ. MPIF, Princeton, NJ, USA. Part 4: 23-32, 2004.
9. H.Wohlfromm, P.J.Uggowitzer, M.Bloemacher, J.ter Maat, A.Thom, A.Pistillo & A.Ribbens, "MIM of a Modified 440C-type Martensitic Stainless Steel", EuroPM 2005, Prague, 333-340.
10. H.R.Lee, G.J.Shu & K.S.Hwang, "Effect of Particle Size on Debinding of PIM Parts", Advances in Powder Metallurgy and Particulate Materials, Chicago, Publ. MPIF, Princeton, NJ, USA. Part 4:147-153, 2004.
11. R. Zauner, D.F Heaney, J.C.Piemme, C.Binet, R.M.German, "The Effect of Powder Type and Powder Size on Dimensional Variability in PIM", Advances in Powder Metallurgy and Particulate Materials, Orlando, Publ. MPIF, Princeton, NJ, USA, Part 10: 191-198, 2002.
12. M.Stucky, H.Zhang & M.Bulger, "Powder Effects on Distortion of MIM 316L", PIM 2006, Tampa, Publ. MPIF, Princeton, NJ, USA.