HARD METAL TOOLING VIA SFF OF CERAMICS AND POWDER METALLURGY

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ABSTRACT

In the past five years, Solid Freeform Fabrication (SFF) Technologies have been explored for fabrication of functional ceramic components. This presentation describes the use of SFF ceramic components as pre-forms in a Powder Metallurgical process to form net shape metal tooling for manufacturing processes, such as injection molding, die casting, etc. The approach involves metal powder consolidation around a SFF ceramic pre-form. Two different SFF approaches, Laminated Object Manufacturing (LOM) and Selective Laser Sintering (SLS), were used to fabricate ceramic pre-forms. The SFF ceramic pre-forms and the consolidated metal tooling were evaluated for density, dimensional accuracy, and surface finish. Results will be presented on the consolidation of tool steel using both the SFF ceramic pre-forms.

BACKGROUND

Dies and molds constitute one of the most significant costs associated with manufacturing processes such as injection molding, die casting, etc. Currently these dies and molds are fabricated by machining wrought tool steels, such as H13. Machining is extremely costly, time consuming, and generates high amount of scrap. The machined die or mold is then subjected to appropriate heat treatment to impart the desired microstructure and properties. The heat treatment often causes dimensional instability and warpage. Machining also imposes restrictions on the tool geometry that can be fabricated. Incorporation of cooling passages, a critical feature for enhanced performance, is almost prohibited by machining. Use of conformal channels in dies and molds can increase manufacturing throughput by over 25% to as much as 70% [1,2].

Hot Isostatic Pressing (HIP) is a preferred Powder Metallurgical (P/M) process for hard to consolidate materials such as ceramics and refractory metals. A major benefit of using P/M dies and molds is the ability to produce near-net shape cavities directly by HIP; thus, minimizing input material and subsequent machining. Furthermore, HIPed dies and molds, using rapidly solidified pre-alloyed powders, have been shown to out-perform cast and wrought-machined tools [3]. However, widespread use of HIPed dies and molds has been precluded due to the difficulty and high costs in fabrication of pre-shaped HIP cans with complex geometry.

In a prior effort, Crucible Materials Corp. has demonstrated that a ceramic pre-form can be successfully used in HIPing of pre-alloyed tool steel powders to fabricate dies and molds [4]. Use of a ceramic pre-form precluded the need for pre-shaped HIP can and also allowed incorporation of conformal cooling passages in the dies and molds. Widespread use of this approach has been hampered by conventional issues associated with use and insertion of ceramics in industry, namely, high cost and long lead times for ceramic component fabrication.

Solid Freeform Fabrication (SFF) technologies have demonstrated that the long lead times and costs for ceramic prototype fabrication can be cut significantly [5]. Use of SFF technologies to fabricate ceramic components has largely been limited to demanding aerospace type applications. The use of ceramics as pre-forms in the HIP process mandates that the ceramic pre-form be low cost and easy to fabricate to keep the dies and molds affordable.

This article describes the fabrication of ceramic pre-forms using two SFF processes, Laminated Object Manufacturing (LOM) and Selective Laser Sintering (SLS), and their use in the HIP process to fabricate tool steel mold cavities, Figure 1. To keep the fabricated dies and molds cost effective, it is important to keep the time and costs low in fabricating the ceramic pre-forms. Also, to avoid carburization and/or nitridation of the tool steel during HIPing, it is preferable not to use carbide and/or nitride ceramics. Prior work has shown that fully dense or high density ceramic pre-forms are not required for successful HIPing of tool steel cavities. In fact, high density ceramic pre-forms make their removal from the HIPed steel cavity difficult. Conversely, very low density ceramic (<70%) is not suitable for successful HIPing. Therefore, it is necessary to determine the minimum density under which HIPing can be successfully performed. Furthermore, lower density ceramic pre-form fabrication will require low sintering temperature and/or time, which result in lower shrinkage during the process.

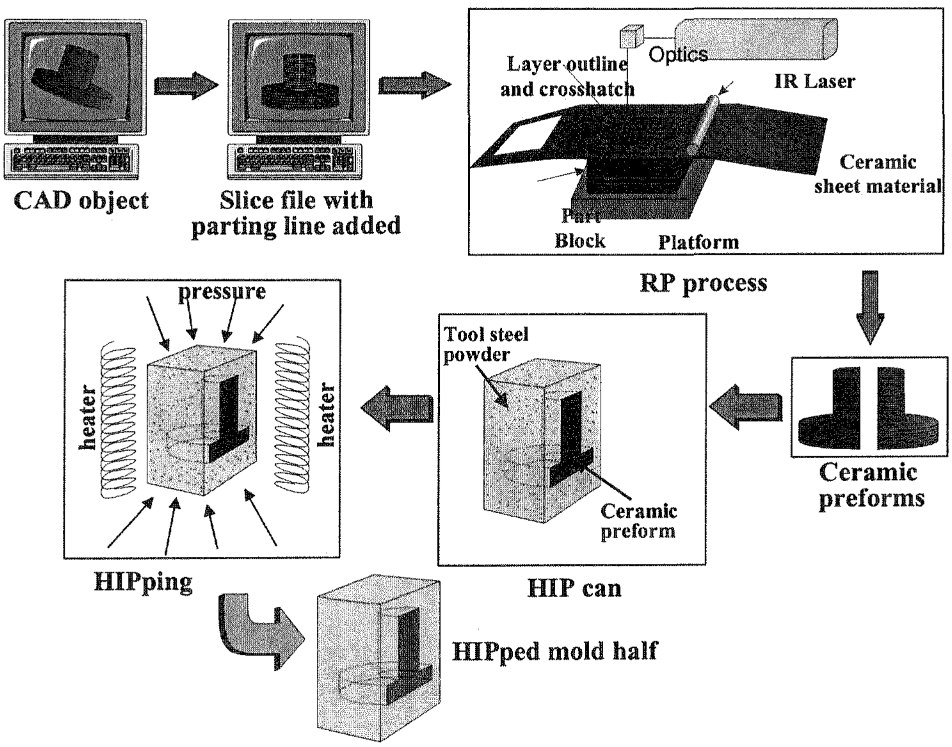

Figure 1: Schematic representation of the entire process employed in this study to fabricate mold and die cavity from SFF ceramic pre-forms.

LAMINATED OBJECT MANUFACTURING (LOM) OF CERAMICS

In the past five years, the University of Dayton and Lone Peak Engineering (LPE), Salt Lake City, Utah, have successfully developed the Laminated Object Manufacturing (LOM) process to fabricate ceramic components using green ceramic sheets as feed materials [6-8]. The LOM process results in a green ceramic part, which is then subjected to conventional thermal cycles of binder burn out and sintering/infiltration to produce a dense ceramic component.

LOM of Alumina:

LOM of alumina has been successfully developed and is commercially practiced by LPE. Continuous rolls of 12" wide and 0.010" thick green alumina tapes, from LPE, were used for this effort. The alumina tapes consist of 55 volume percent Alcoa A16 alumina powder with the balance a thermoplastic binder suitable for LOM processing. The tapes were successfully used in the LOM process to fabricate simple green shapes and were analyzed by Scanning Electron Microscopy (SEM) for inter-layer bonding. The green alumina LOM parts were then used to develop the appropriate binder burn out cycle. The fully burned out LOM alumina parts were also analyzed by SEM. Through a series of LOM trials, binder burn out trials, and analysis, suitable LOM processing and binder burn out conditions were established for defect-free part (1" to 2" cross-section) generation.

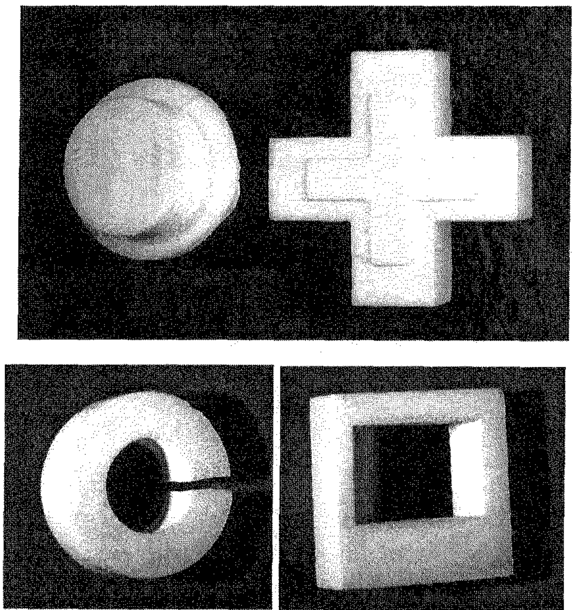

Figure 2: Sintered LOM alumina parts (Size: 1"-2" in X-Y and 0.5" Z)

To evaluate the feasibility of using LOM alumina pre-forms in HIPing, four simple LOM alumina samples (1"-2" dimension) were fabricated and subjected to binder burn out and sintering at 1650°C for 6 hours, Figure 2. The bulk density of these samples was 90%-92%. The average linear shrinkage was: ~17% in X and Y and ~20% in Z (LOM build direction). These samples were then used in the HIPing of tool steel to form mold cavities, discussed later.

A series of sintering experiments were performed with the LOM alumina parts. Through these experiments, sintered density and shrinkage data was established as a function of sintering temperature and time, Table 1. This data will be used in fabricating LOM alumina samples with varying density for evaluation in HIPing of tool steel mold and die cavities.

Table 1: LOM Alumina Sintering Data

Sintering Condition	Sintered Density	% Shrinkage	
	(%)	X & Y	Z
1650 °C, 6 hours	90	15	17
1500 °C, 4 hours	77	10.8	12.7
1450 °C, 4 hours	72	8.9	10
1500 °C, 1 hour	68	7	8.5
1450 °C, 1 hour	65	5.75	7

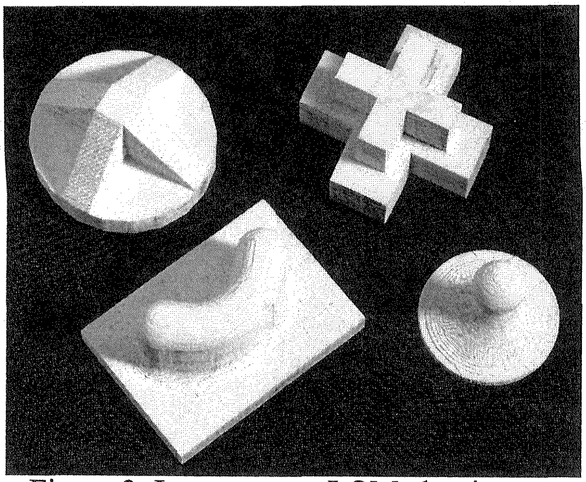

Figure 3: Larger green LOM alumina parts (Size: 3"-4" in X-Y and 1"-2" in Z)

Molds and dies used in manufacturing processes can be from a few inches to several feet in dimension. Fabrication of large ceramic pre-forms are prohibited due to the limited size of the SFF systems and due to difficulty in successfully removing binder from large ceramic pre-forms. The technology in this article is expected to be suitable to fabricate molds and dies with sizes limited to less than 8" – 10". To demonstrate the ability to fabricate larger LOM ceramic pre-forms, a series of four 3"-4" samples were fabricated using alumina, Figure 3. These samples have been successfully processed to 70% - 80% sintered densities and are currently being used in HIPing of steel cavity.

LOM of Silica:

Fused silica is low cost and is the most commonly used ceramic in industry. Therefore, it is most desirable to develop and use silica pre-forms for HIPing of tool steel molds and dies. However, LOM of fused silica has not been explored or developed. The silica powder selected for the process is a refractory grade fused silica. The average particle size of the powder is –325 mesh and its typical chemical composition is: 99.7% SiO_2, 1150 ppm Al_2O_3, 185 ppm Fe_2O_3, 57 ppm CaO, 29 ppm Na_2O, and 58 ppm K_2O.

Green silica tapes for LOM were developed with a binder system used successfully in prior ceramic LOM efforts. 10" wide and 0.008" thick tapes were cast with 84 weight % silica powder and 16 weight % binder. The silica tapes have been successfully used in fabricating simple parts by the LOM process. Simple LOM green silica parts were used to establish suitable binder burn out schedules. Following successful binder burn out, the samples were subjected to firing at temperatures of 1100°C and 1300°C for 2 to 6 hours, Figure 4. The fired part density ranged between 70% and 80% of theoretical density of fused silica (2.2 g/cc). Associated firing shrinkage was 1% - 3% in X and Y and 3%-5% in Z. Simple LOM silica samples are currently being processed (similar to those shown in Figure 2 for alumina) for their use in HIPing of tool steel cavity.

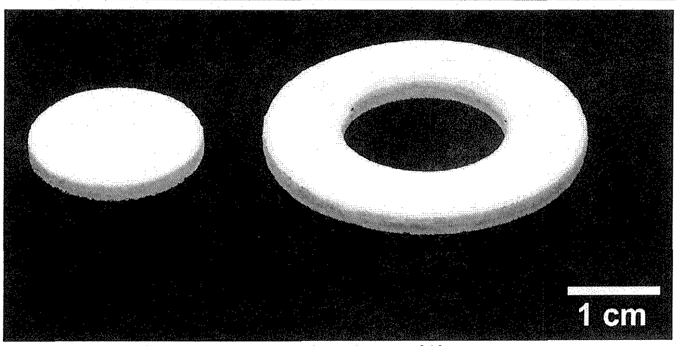

Figure 4: Fired LOM silica parts.

SELECTIVE LASER SINTERING (SLS) OF CERAMICS

Selective Laser Sintering (SLS) is a SFF process, commercialized by DTM Corp., Austin, Texas. Several ceramic systems, with binder, have been explored for use in the SLS process. Due to the low packing density of the powder bed during the SLS process, the green SLS processed parts have relatively low green densities. The low green densities of the SLS processed ceramics have limited their use in many applications. However, two ceramic systems, called SandForm™ Si and Zr, have been successfully developed and commercialized by DTM for use as shell foundry cores in the casting industry. SandForm™ Si and SandForm™ Zr are polymer (thermoset resin) coated silica and zircon sand materials. Under the current practice of SLS SandForm™, the resin partially cures to hold the sand particles together during SLS processing. Following the SLS processing, the resin is fully cured in an oven at 50 to 150°C. The sand (ceramic) particles employed in the process are considerably coarse (140 mesh average particle size). Further, the green density of the parts is extremely low (<50%). These conditions are sufficient for their use as casting cores and match very closely with those used conventionally in foundries. For this study, SLS SandForm™ parts require complete removal of the resin binder followed by firing to achieve the desired density.

Based on thermal analysis of the SandForm™ powders, suitable conservative binder removal cycles were employed to completely remove binder from the SLS processed SandForm™ Si and Zr parts. Under all burn out cycles and conditions, although the binder was completely removed from the samples, due to the low green density of the parts and the coarse particle size, the parts did not retain structural integrity.

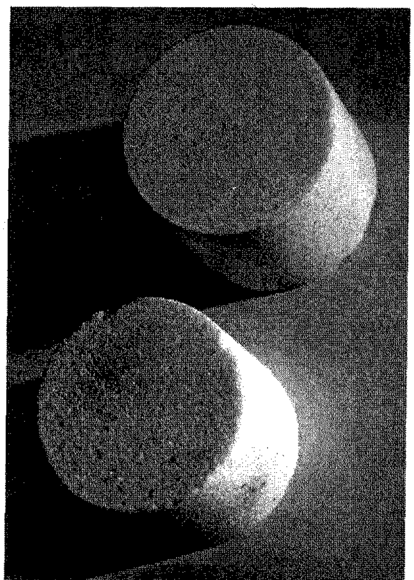

Figure 5: Pressed and fired SandForm silica parts

Improving the green density of SLS processed SandForm™ parts requires modifications to the material and/or the SLS process itself. Therefore, an alternative strategy was explored to first evaluate the suitability of the SandForm™ materials for HIPing of tool steels. This strategy involved fabricating higher green density SandForm™ parts by pressing the powders instead of subjecting them to SLS processing. If it is found that the pressed SandForm™ samples, with improved green density, are suitable for HIPing, then it will form the basis to pursue increasing the green density of SLS processed parts by tailoring the material and the SLS process.

SandForm™ silica powder was pressed in a 1" diameter die in a pneumatic press capable of heating. The pressing temperature and pressure was varied to achieve maximum green density in the pressed samples. The maximum silica green density achieved was 1.88 g/cc, which is 34% higher than the green density of SLS processed silica SandForm™ parts (1.4 g/cc). The pressed silica samples were subjected to binder burn out followed by firing under different firing temperatures and times. The fired density of the samples varied between 65% - 80% theoretical density of silica (2.2 g/cc). The pressed and fired samples, Figure 5, were then used in HIPing of tool steel.

HOT ISOSTATIC PRESSING OF TOOL STEEL WITH SFF CERAMIC PRE-FORMS

Hot Isostatic Pressing (HIP) of tool steel was performed using the LOM alumina samples, shown in Figure 2, and the pressed SandForm silica samples, shown in Figure 5. The tool steel powder used in this effort was a rapidly solidified, pre-alloyed tool steel powder called CPM Nu-Die V that is equivalent to AISI H-13. The chemical composition of the powder is 0.39C, 0.39Mn, 0.010S, 1.01Si, 5.23Cr, 0.43Mo, 0.96V and balance Fe.

The first step in HIPing is the preparation of the "can" (HIP container) containing the powder to be HIPed. In this effort, tool steel cans with 0.25" wall thickness were used. The cans had a 0.25" thick base and lid plates, with the lids drilled to accept a fill/evacuation stem. In addition, the lids were machined to have a stepped diameter fitted into the can ID to facilitate welding without contamination of the powder. After grinding the inside surfaces to a 60 grit finish, the bases and can bodies were welded together.

A 1/8" uniformly distributed layer of tool steel powder was filled in the can and then the ceramic pre-form is placed at the center of the can body. The can was then filled to within 1/8"of the top with tool steel powder. The powder in the can was not packed mechanically to prevent the ceramic pre-form from moving. The lid was then placed atop the can body and welded into place. Additional powder was added to the cans to bring the powder level about ½" up into the fill stems. This entire process is shown schematically in Figure 6. The stem is then topped off with some more tool steel powder. The entire assembly is then evacuated from the fill stem to -10 μm with a -10μm/minute leak rate at 300°F. The stem is then sealed under vacuum. The evacuated and sealed can assembly is placed in a Hot Isostatic Press for HIP operation.

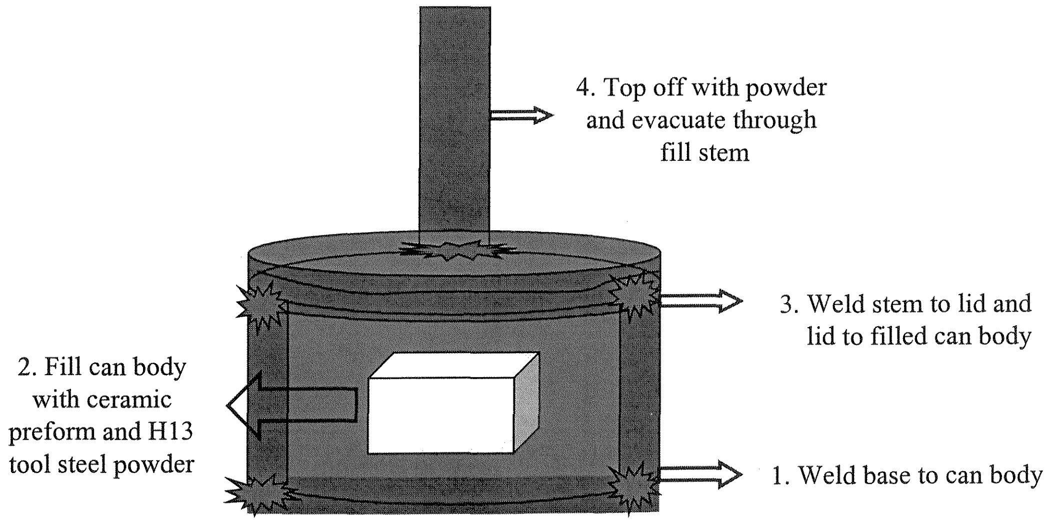

Figure 6: Schematic of the HIP "can" assembly employed in this study.

The HIP cycle used in this study was specifically developed by Crucible for HIPing of the tool steel with ceramic pre-forms [4], shown in Figure 7. In the HIP cycle, the HIP chamber is first evacuated. The temperature is then increased to 2100°F and then the pressure is increased to 1,000 psi. The temperature and pressure are kept constant for 2 hours. The low pressure heating is specifically designed for HIPing when using ceramic pre-forms, to prevent ceramic cracking due to thermal or mechanical shock. The low pressure heating and hold also allows the tool steel to undergo preliminary densification and conform around the ceramic pre-form. The pressure is then increased to the final consolidation pressure of 15,000 psi, with the temperature held at 2100°F. The final pressure and temperature are held constant for 4 hours.

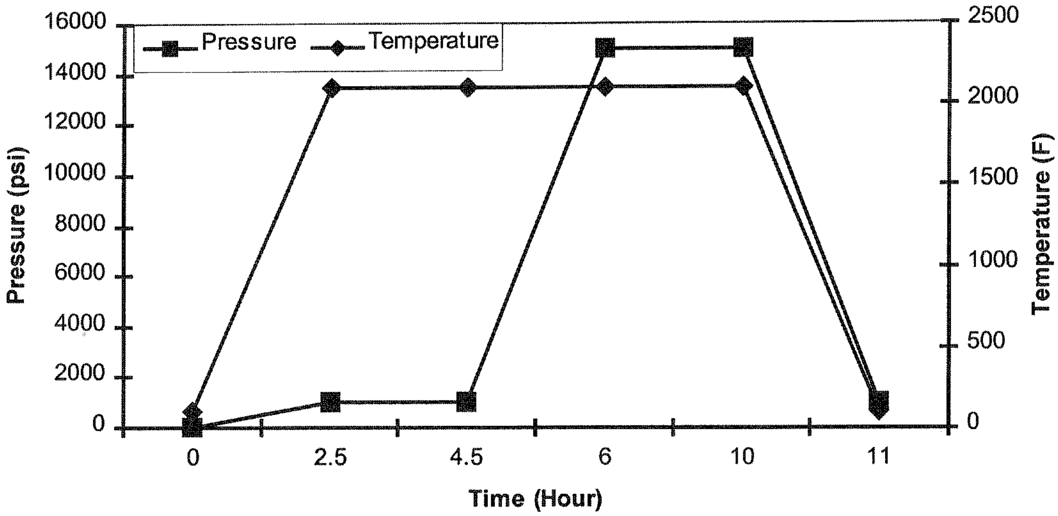

Figure 7: HIP cycle employed in this study

After HIP, the cans exhibited expected shrinkage indicative that the tool steel powder had densified during HIP. The fill stems were cut off flush with the tops of the can lids. The bottom plate in the can assembly is machined to expose the HIPed tool steel layer. The top plate is then machined parallel to the bottom. This is followed by blanchard grinding from the bottom until the ceramic pre-form surface is exposed.

Although the ceramic pre-forms did not exhibit any deformation or change in size, surface cracks were observed in the LOM alumina pre-forms. However, close examination of the HIPed tool steel powder and the alumina surface cracks indicated that there was no penetration of tool steel powder into the cracks. This observation suggests that the ceramic pre-forms cracked during the HIP cool down stage, after complete consolidation of the tool steel. The cracks occurred during the cooling process due to the thermal expansion mismatch between alumina and steel. No such cracks were observed in the pressed SandForm silica samples.

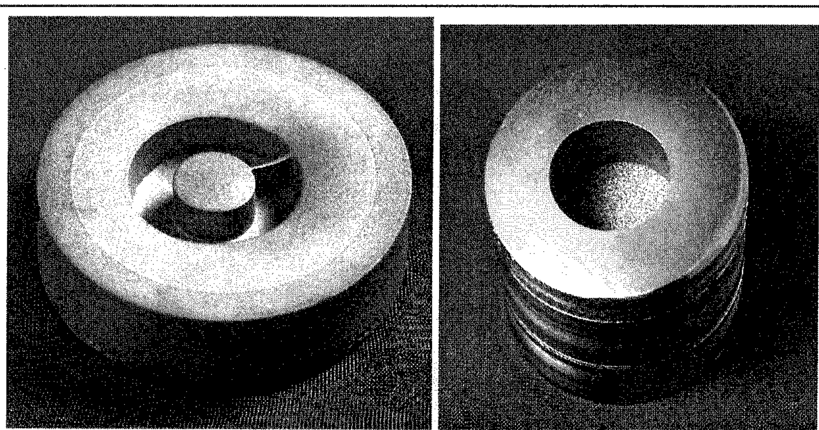
Figure 8: HIPed tool steel cavity fabricated using LOM alumina (left) and pressed SandForm silica (right)

Removal of the LOM alumina pre-form from the HIPed steel cavity has proven to be difficult. Due to high density of the alumina pre-forms, destructive techniques using diamond drilling and other techniques resulted in partial removal of the pre-forms. Grit blasting resulted in complete removal of the ceramic pre-form. However, grit blasting caused some wear of the HIPed steel, Figure 8. Removal of the pressed SandForm silica pre-forms was much easier. The silica pre-forms were removed by blowing away the ceramic using pressurized air, Figure 8.

The dimensions of the tool steel cavities formed using LOM alumina as well as SandForm silica were within 1% of the dimensions of the ceramic pre-forms used in forming them. The surface finish of the cavities formed using LOM alumina reflected the layered finish present in the LOM alumina pre-forms. Similarly, the surface finish of the cavities formed using the pressed SandForm silica reflected the coarse surface finish arising due to the coarse particle size of the SandForm powder.

FURTHER EFFORT

LOM of alumina and silica and SLS of SandForm™ ceramic pre-forms are being further developed for HIPing of tool steel mold and die cavities. This effort involves:

- Optimizing the alumina and silica LOM process for larger parts (8").
- HIPing with lower density alumina and silica LOM samples.
- Efficient ceramic removal approaches, including coating of the pre-forms.
- Tailoring the SandForm™ material and the SLS process for higher green densities.
- Fabrication of demonstration injection mold inserts and their performance evaluation in the molding process.
- Incorporation of conformal cooling channels during HIPing in the mold inserts, using the SFF ceramic pre-forms.

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