Rapid Tooling of AL_2O_3 Parts using Temperature Induced Forming

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ABSTRACT

A novel near-net shape process for the rapid production of complex-shaped ceramic prototypes has been developed. By using a newly developed in situ temperature induced forming (TIF) technology, concentrated ceramic slurries are vacuum cast into a mold, which has been produced in a rapid tooling process. The tooling process enables the production of complex-shaped molds which allow powerless demolding. The TIF technology enables the change of particle potential in the slurry from a repulsive into a weakly attractive particle network during consolidation. In combination of TIF- and rapid tooling technology a green body with a high and homogeneous density without any cracks can be realized.

1 INTRODUCTION

The processing challenge in the forming of advanced ceramic parts is to achieve a defect-free microstructure in the final body. Forming a ceramic powder into an engineering part can be performed with a variety of methods, such as powder compaction, injection molding, extrusion, slip casting, pressure casting and more. In general the choice among these methods often depends on several important factors, such as product volume, size tolerance, cost, short cycle time and more. Due to the high hardness of ceramic materials, it is almost always cost prohibitive to form a component by removing material from a solid ceramic block or shape. Therefore, near-net-shape forming is regarded as the most efficient, economical and reliable pathway for practical applications /1/.

Temperature induced forming (TIF) /2/ is a novel forming method for ceramics via a colloidal processing route. Consolidation of a highly filled ceramic slurry is induced simply by an increase in temperature. No dispersing media needs to be removed and so a nonporous mold can be used. TIF produces not only near-net-shape parts, but also complex shapes limited only by the mold design.

Consequently, a novel rapid tooling process for the production of complex-shaped ceramic prototypes has been developed by combining temperature induced forming with a rapid tooling technology allowing an almost powerfree demoulding. This novel process allows the production of advanced ceramics with a high surface finish, near-net shape and reliability in their mechanical properties.

2 TEMPERATURE INDUCED FORMING (TIF)

Figure 1 presents a flowchart for the TIF process. Al_2O_3 powder (AKP 53, Sumitomo, Japan) with mean particle size of 0.24 μm was used for the experiments. Aqueous Al_2O_3 slurry at a solid volume fraction of 0.55 with tri-ammonium citrate (TAC) (Fluka, Germany) as dispersant was homogenized by ball milling (a planetary ball mill, ZrO_2 balls) for two hours in a Teflon container. As reported by Luther /3/, the isoelectric point of Al_2O_3 shifts from about pH 9 to pH 3 when a large amount of tribasic ammonium citrate is added. The pH value of the Al_2O_3 slurry is stable at about pH 10 due to the dissociation of TAC. Therefore, TAC is a very good dispersant for Al_2O_3 with which a highly concentrated slurry with low viscosity can be realized. Poly(acrylic acid) (PAA) from Polyscience, Inc. was added to a dispersed Al_2O_3 slurry and further ball milled at lower speed. After milling, the dissolved gas in the slurry was removed in a vacuum chamber at about 20 mbar.

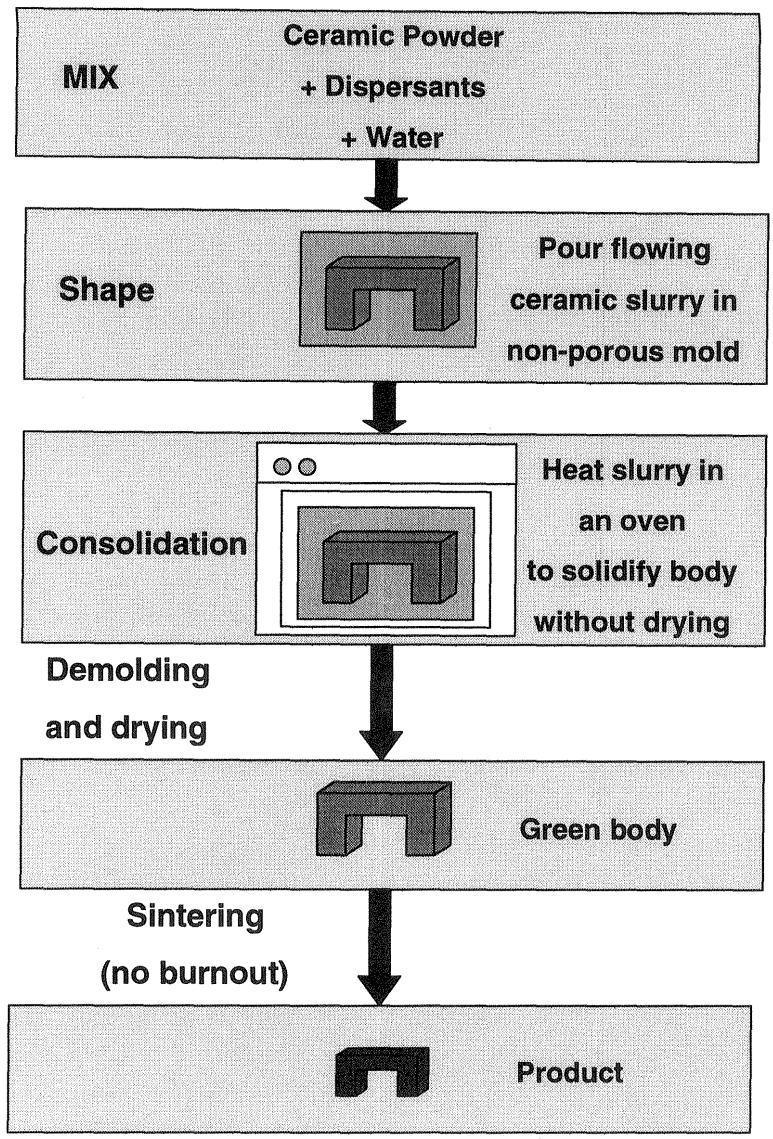

Figure 1: TIF process

This highly concentrated Al_2O_3 slurry is then poured into a non-porous mold. After casting, the slurry together with the mold is sealed and warmed up in a furnace. The temperature treatment at about 60°C yields coagulated slurry in the mold. An elastic wet green body can be achieved without drying. After demolding, drying and sintering, a near-net shape ceramic prototype with a high degree of surface finish and reliable mechanical properties is achieved.

Recent studies indicated that the consolidation of Al_2O_3 slurry results from bridging flocculation of the polyacrylic acid caused by a competitive adsorption of the dispersants that is enhanced with increasing temperature. The rheological properties are a key to monitor this gelation behavior. Figure 2 shows the relationship between the viscosity of Al_2O_3 slurry and temperature. Aqueous Al_2O_3 slurry at a solid volume fraction of 0.34 was used for the rheology measurement. The pH value of the slurry was adjusted to pH 9.12 with KOH. The measurement was performed with a rotational viscometer (SR500, Rheometric Scientific, USA) at a shear rate of 20 s^{-1} as the temperature was increased from 25°C to 60°C. Vegetable oil was placed on top of the slurry to prevent evaporation of water. By increasing the temperature, the viscosity of the slurry increases rapidly because of bridging flocculation. The significant gelation occurs at temperatures between 30°C and 40°C. The plateau region in the viscosity curve is due to the break down of the gelled network by rotation during measurement.

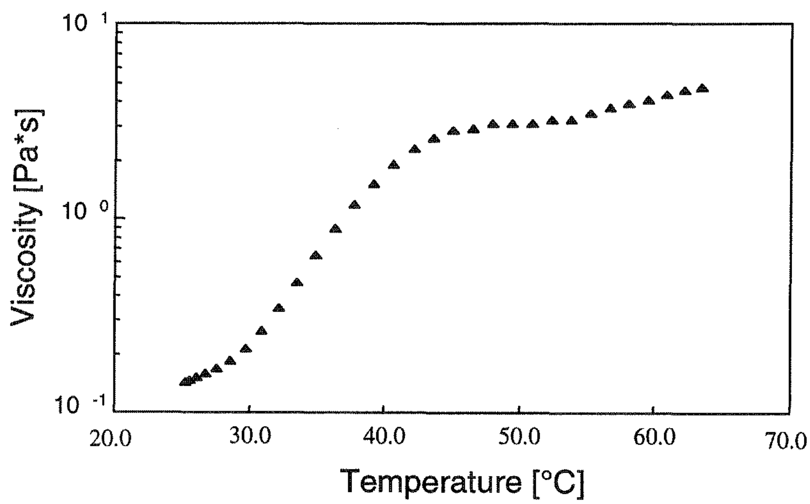

Figure 2: Gelation of TIF-slurry by temperature

Thus, green parts are easily produced by heating alumina slurry at high solid loading in such a way that evaporation is minimized. Once the system is cooled and has sufficient green strength, the body can be removed from the mold and then dried.

The consolidation of the slurry occurs in the mold without any external stress and the shaped parts are dried after demolding. Therefore, a green body with high and homogeneous density (about 65 %) as well as fine surface structure without cracks can be realized.

3 RAPID TOOLING PROCESSES

During the consolidation of the slurry the particle potential changes from a repulsive into a weakly attractive particle network. This weak attraction results in a green body strength comparable with gelatin. To demold simple geometries silicone molds can be used, but in order to obtain complex-shaped green bodies containing undercuts and thin structures an almost powerless demolding process is required. Figure 3 and 4 show the applied rapid tooling processes.

Figure 3 illustrates a process specially for producing small complex-shaped parts. At the beginning of the process a model of an acid-soluble wax has to be manufactured. Applicable techniques are milling or casting in silicone or solid molds. Depending on the melting temperature of the waxes used, the wax model is dipped or immersed in an insoluble wax. Short time dipping or additional cooling of the acid-soluble wax could prevent a blending of the applied waxes. Subsequently the coated acid-soluble wax is immersed and dissolved in an acid-solution.

The TIF-slurry is casted in the hollow wax body generated. The sealed mold is placed in an vegetable oil bath and heated-up to about 60°C to effectuate the coagulation of the TIF-slurry in the mold. As wax is a very bad heat conductor the heating time is relatively long. After the coagulation of the whole slurry the temperature of the oil bath is raised over the melting range of the encasing wax. The wax gets partially dissolved in the oil, the rest is sinks to the bottom of the bin. The oil prevents the ceramic body from air contact and drying at high temperature. The demolded green body remains in the oil until room temperature is reached. Afterwards the part is taken out of the oil and dried under controlled environmental conditions. This way the rapid tooling procedure ensures a powerless demolding and a controlled drying, the requirement to obtain complex shaped, crack free green bodies. If the self weight and the geometry of the ceramic part is under certain limits, the process allows production of green parts without a plane bottom.

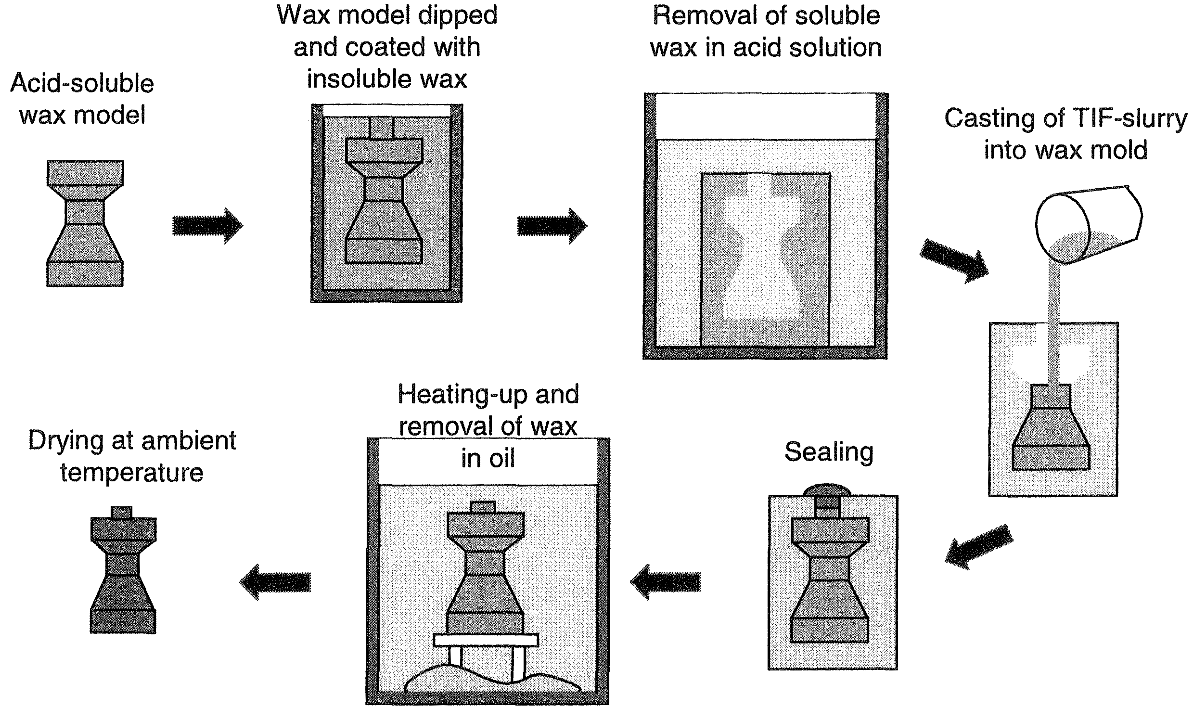

Figure 3: Rapid tooling process using acid-soluble wax

Figure 4 shows a simplified rapid tooling process. The process enables the production of TIF-parts of restricted geometry.

In a first step a silicone part with a plane bottom had to be produced and immerged in a wax. The silicone part is placed on a small substructure that enables the wax to flow around the silicone body. This procedure ensures that the wax is shrinking from all directions on the silicone body and no undesirable warp of the cooling wax occurs. The top side of the solidified wax is milled even and the silicone part is pulled out. TIF-slurry is cast in the wax mold until a meniscus, caused by the surface tension of the slurry, is reached at the top of the mold. A silicone grease coated disk is pushed over the top of the mold sliding away excessive slurry. This procedure ensures that the slurry is airless and covered. Afterwards the mold is turned upsidedown in the oil bath. The remaining process is as described above. The process enables the rapid production of more voluminous green bodies.

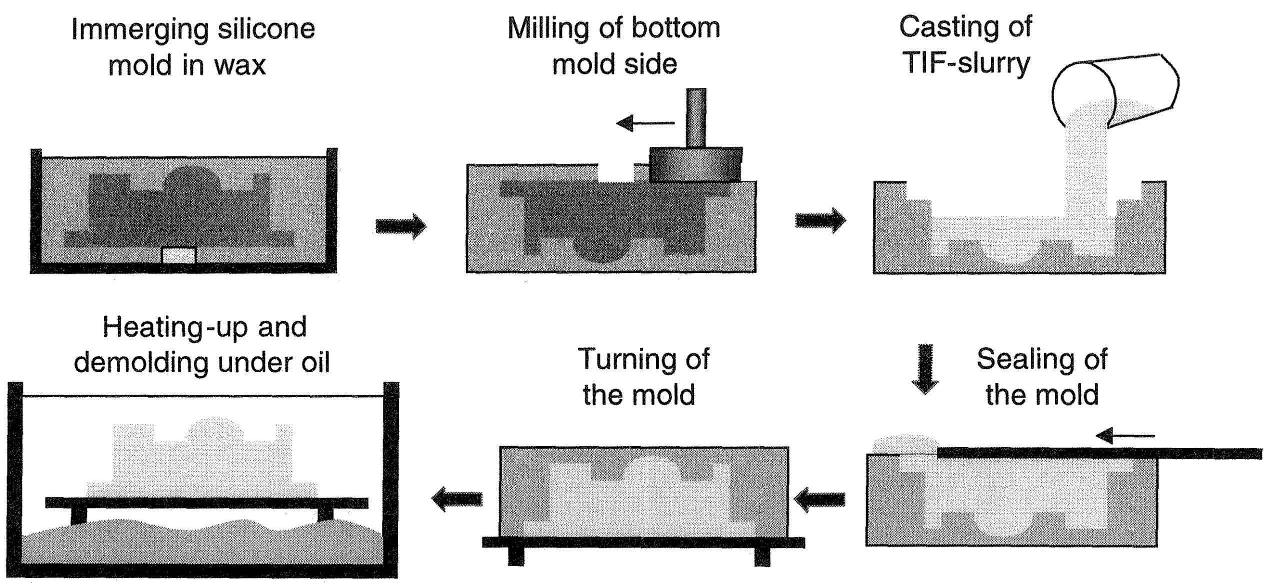

Figure 4: Rapid tooling process using silicone models

4 Results

One example of a stereolithography model and sintered Al_2O_3 is shown in figure 5. Figure 6 shows a complex shaped ceramic tool after removal from the oil bath with wax rests on the surface.

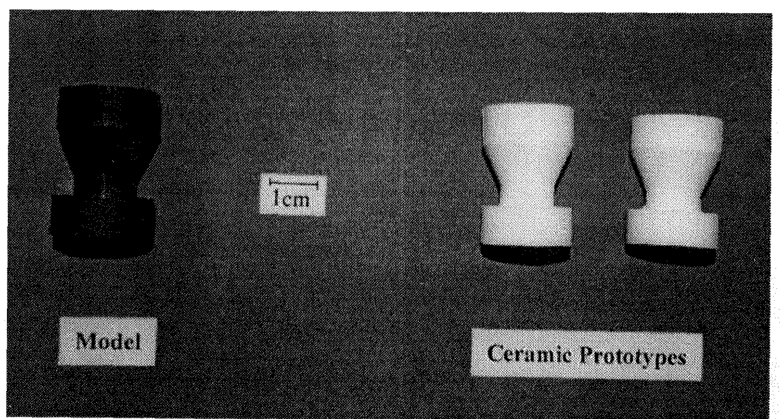

Figure 5: One example of a stereolithography model and the sintered Al_2O_3 parts

The microstructure of the sintered parts is very homogeneous with a mean grain size of about 1 µm (Figure 7). Sintering was performed at 1400°C for two hours in air. Highly densified microstructures are found with only a very small amount of pores in the grains and some abnormal grain growth because of the absence of sintering aids. Therefore, we conclude that this process can achieve homogeneous microstructures.

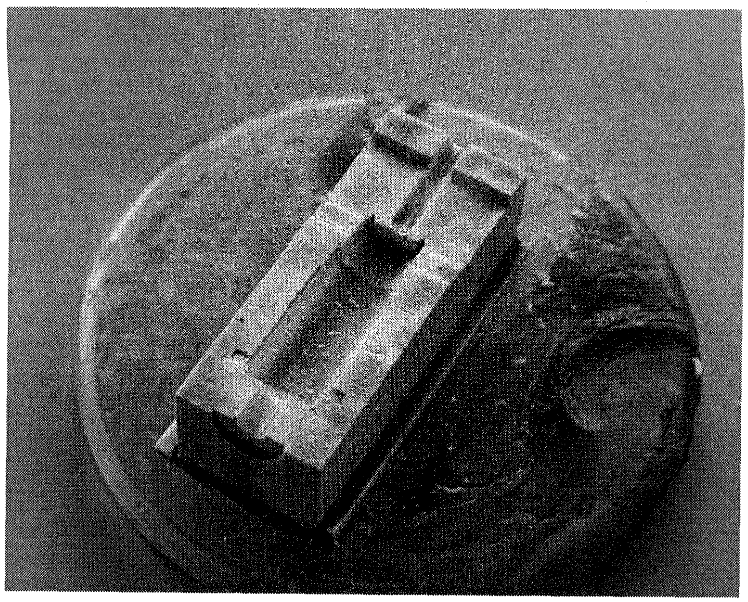

Figure 6: TIF-Ceramic after removing of the oil bath

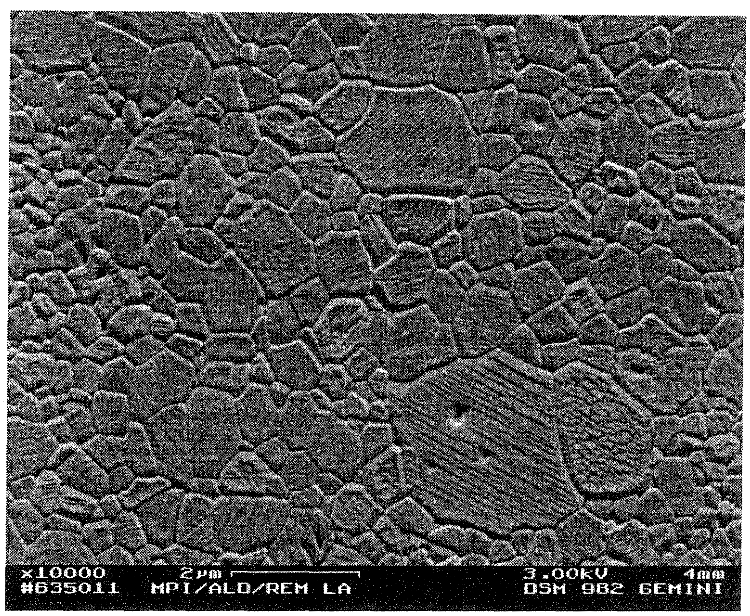

Figure 7: Sintered Al_2O_3 made by the TIF process showing fully dense microstructure with grain size between 0.5 and 2 μm.

The advantage is obvious if the mechanical properties of sintered Al_2O_3 are considered for the TIF process in comparison to conventional cold isostatic pressing (CIP) at 200 MPa. The bending strength at room temperature (RT) was tested on a 20/40 mm four-point fixture with bend specimens of 3 x 4 x 45 mm³ followed by cutting and polishing from sintered Al_2O_3 bars at 1400°C for 2 hours in air without sintering aids. The bending strength for the TIF processed

Al_2O_3 bars (5 specimens) was 455 ± 30 MPa, whereas for CIP processed Al_2O_3 bars only 269 ± 28 MPa were achieved. The significant increase in the bending strength can be attributed to a very high degree of deagglomeration and good dispersion of the originally strong agglomerated sub-micron Al_2O_3 powders. It also indicates homogeneous consolidation as well as stress-free drying by TIF. Further improvements of the mechanical properties by optimizing the TIF processing with filtration to remove heterogeneities and introducing sintering aids, such as MgO are under investigation.

5 CONCLUSIONS

In this work a novel process for the fabrication of advanced ceramic prototypes has been developed by combining rapid tooling technology and temperature induced forming to produce reliable and complex-shaped ceramic prototypes quickly. Complex-shaped molds with high surface quality can be produced. Highly concentrated Al_2O_3 slurry is consolidated in non-porous molds simply by mildly increasing the temperature. Therefore, fine and dense green parts without any cracks can be realized. Apart from the near-net shape, the process also has significance for production of advanced ceramics with reliable mechanical properties. Higher bending strength at RT is achieved for the TIF process in comparison to conventionally processed (CIP) powder.

This novel near-net shape technique offers the opportunity to significantly shorten the time to market for the production of new ceramic parts. TIF enables the forming of advanced ceramic prototypes and the process also seems suitable for small series production. This new forming method has many advantages, such as rapidly produced complex-shaped ceramic parts, a more homogeneous density distribution of the green body and consequently more reliable mechanical properties than conventional pressing techniques offer.

6 ACKNOWLEDGMENT

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