

Titanium Castings Using Laser-Scanned Data and Selective Laser-Sintered Zirconia Molds

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(Submitted 17 January 2001; in revised form 29 March 2001)

Titanium casting molds, made of stabilized and unstabilized zirconium oxide, were created using a combination of selective laser sintering (SLS) and colloidal infiltration. The mold material system was chosen for its thermal shock resistance and low reactivity with molten titanium. The starting material, stabilized zirconia powder mixed with a copolymer binder, was laser sintered into the desired green shape. The binder was removed during pyrolysis and replaced by unstabilized zirconia. As infiltrant weight gain increased, the density, flexural strength, and surface roughness improved to levels adequate for titanium casting trials. A half-scale casting mold for the ball of a human femur bone was produced from laser-scanned data and cast with Ti-6Al-4V alloy. The castings exhibited an as-cast surface roughness (R_a) of 8 μm and a typical microstructure. This work demonstrates a feasible method of producing complex titanium castings for one-of-a-kind and custom components without the necessity of part-specific tooling or wax patterns.

Keywords selective laser sintering, Ti-6-Al-4V alloy, titanium casting molds, zirconia

Introduction

The advantages of rapid prototyping and titanium casting have been combined in a system to produce custom titanium components from laser-scanned data. The invention of selective laser sintering (SLS) and other rapid prototyping technologies has generated much interest in the biomedical industry because of the potential to build "custom-made" implants and surgical planning models. Data acquired using traditional methods such as magnetic resonance imaging, computerized tomography scanning, or x-ray can be converted to a solid geometric model using standard software. Subsequently, data stored in a drawing format can be converted to a stereolithography (STL) format for use in rapid prototyping machines. Using this method, complicated and exact reproductions and negatives of human structures can be constructed using SLS without specific tooling or a physical pattern. Additionally, design changes and additions can be added to the computer model prior to physical construction. Selective laser sintering was used to create a casting mold for a human femur, based on laser-scanned data from a real bone.

Because of molten titanium's strong chemical activity, only a limited number of materials are resistant to chemical attack and are suitable as casting mold materials.[1,2] Several studies show that zirconia is one of the least reactive materials.[1,3,4] However, zirconia undergoes a destructive phase transformation at 1100 °C that can crack a mold during casting. It converts from a monoclinic to a tetragonal atomic structure, resulting in 3 to 7% volume shrinkage.[3,5] The addition of a small amount of yttria stabilizes the zirconia in a cubic structure that does not change phase at 1100 °C.[6] When a mold based in stabilized zirconia is infiltrated with zirconia in the unstabilized form, a "partially" stabilized mold is created. This partially stabilized structure has better shock resistance than either the unstabilized or fully stabilized forms of zirconia.[3]

Mold "green" shapes were selective laser sintered at low temperature from a powder bed of stabilized zirconia and sacrificial polymer binder. The green shapes were infiltrated with an unstabilized zirconia precursor solution and pyrolyzed to remove the sacrificial binder. The fired molds were cast with Ti-6Al-4V alloy. Both the mold material and the castings were characterized.

Experimental Procedure

Material Preparation

Granulated yttria-stabilized zirconia powder from Tosoh Ceramic Corporation (Tokyo, Japan) was selected as the base material for the casting mold. The Tosoh powder consists of zirconia crystallites, 24 nm average size, granulated into 50 μm size spheres with a water-soluble binder. To minimize post-process shrinkage and prevent granulate dissolution during infiltration, the granulated particles were presintered for 1 h at 500 °C and 2 h at 1250 °C to pyrolyze the binder and densify each particle. The sintering schedule was determined from a sintering map previously developed for zirconium oxide.[7]

The sacrificial SLS binder, an 80:20 molar blend of methylmethacrylate and butylmethacrylate, respectively, was produced by methods discussed elsewhere.[8] This particular copolymer binder was chosen because it completely "unzips" during firing, leaving no contaminating residue. The copolymer binder was spray dried into fine particles using an Anhydro Laboratory 1 Model Spray Drier. The presintered zirconia powder and 30 vol.% copolymer were mixed in a rolling mill for 24 h.

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Material Processing and Characterization

An SLS Model 125 Workstation (DTM Corporation, Austin, TX) was used to build a series of bend test specimens. Layer thickness was set at 127 μm (0.005 in.), laser scan spacing at 76 μm (0.003 in.), and laser scan speed at 40.6 cm/s (16 in./s). A CO_2 laser, operating at a power level of 5 W, sintered the powder. To prevent part curling, the powder bed was preheated to 80 °C.[9]

Five green bend specimens were measured and tested per ASTM C1161-94 using a four-point bend apparatus. Fracture surfaces were examined using scanning electron microscopy. Density was measured using the Archimedes method with ethanol. Surface roughness (R_a) was measured using a Surfanalyzer 5000.

The remaining bend specimens were measured and weighed and then infiltrated with a colloidal solution of hydrous zirconia Zr100/20 (Nyacol Products, Inc., Ashland, MA). Wetting was improved by adding a surfactant to the colloid. After a number of infiltration and curing cycles, the samples were pyrolyzed at 500 °C to burn off the copolymer binder and then fired at 1500 °C for 10 h to promote crystallization and grain growth. After firing, each specimen was reweighed and remeasured. Five samples were set aside for Archimedes density measurements, flexural testing, microscopy, and roughness measurements. The infiltration and firing sequence were continued for two more cycles, and material characterization was done after each cycle. X-ray diffraction was used to determine the zirconia phases in the final mold material.

Mold Preparation and Casting

A human femur bone was scanned using a Digabotics three-dimensional (3-D) laser scanner (Digibotics, Georgetown, TX) to produce an 80,000-facet 3-D model in a Drawing eXchange Format (AutoDesk, Inc., San Rafael, CA). The model was converted to an STL format using STL_UTIL (B. Michel Sprimont, Belgium, public domain). Because of the immensity of the file size, the model was decimated from 80,000 to 30,000 facets using Visualization Toolkit software (K. Martin, W. Schroeder, and B. Lorensen, public domain, available through Kitware, Inc., Clifton Park, NY). The femur model was divided into two pieces, separating the femur shaft from the femur ball. The surface normals of the femur ball were inverted and used for the inside of the casting mold. A sphere was constructed for the outer surface of the casting mold, and three cylinders were introduced to create a relief vent and a gate. Boolean operations combined the objects in 3-D Studio Max (AutoDesk, Inc.) to create the complete mold design. Custom software converted the 3-D Studio Max model to an STL format.

The femur ball mold STL file was transferred to the SLS Model 125 workstation. The model was sliced using custom software and constructed using the SLS processing parameters given in the previous section. Two identical molds, scaled to half-size to conserve material, were constructed. After infiltration and firing, the casting molds were transported to Howmet Research Corporation (Whitehall, MI). The molds were attached to a standard casting runner using a proprietary mud and left to dry overnight. The most widely used titanium alloy, Ti-6Al-4V, was cast into the zirconia molds. The titanium castings were returned to The University of Texas at Austin for metallographic examination.

A section of the casting gate was mounted, polished, and etched with a 10% hydroflouric acid solution for microstructural analysis and alpha-case thickness determination. Roughness measurements were done on the as-cast surface.

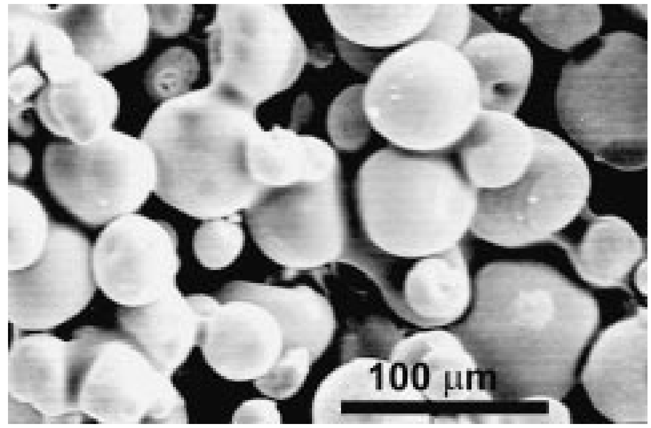


Fig. 1 Fracture surface of SLS part (zirconia-30 vol.% copolymer)

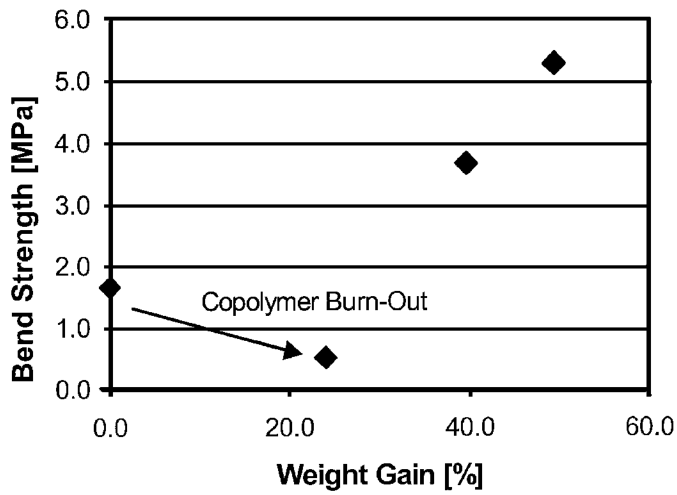


Fig. 2 Flexural strength vs infiltrant weight gain

Results and Discussion

A fracture surface of a green bend specimen (zirconia-30 vol.% copolymer) is shown in Fig. 1. Necks of copolymer tack the spherical zirconia particles together. Initially, the necks provided strength to the green parts. After copolymer burnout, the infiltrated zirconia provided strength. Figure 2 shows the flexural strength of zirconia specimens as a function of infiltrant weight gain. Weight gain was calculated with respect to the original Tosoh zirconia weight (without the copolymer). After copolymer burnout, the strength dropped. However, after multiple infiltrations, the bend strength reached over 5 MPa, three times the reported value for a similar zirconia mold material system used for titanium casting.[2]

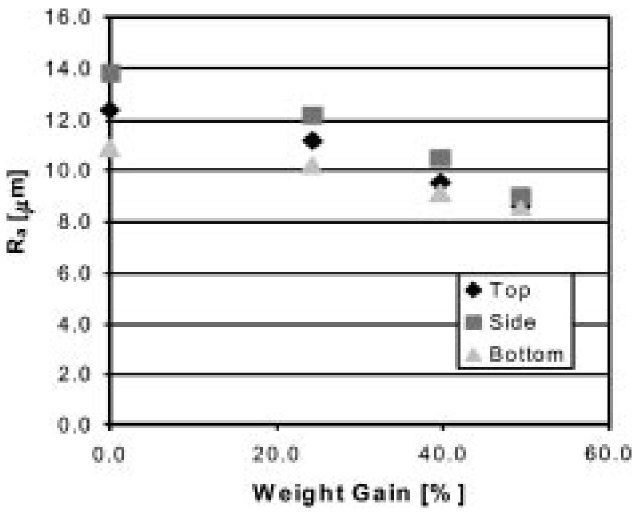


Fig. 3 Roughness vs infiltrated zirconia weight gain

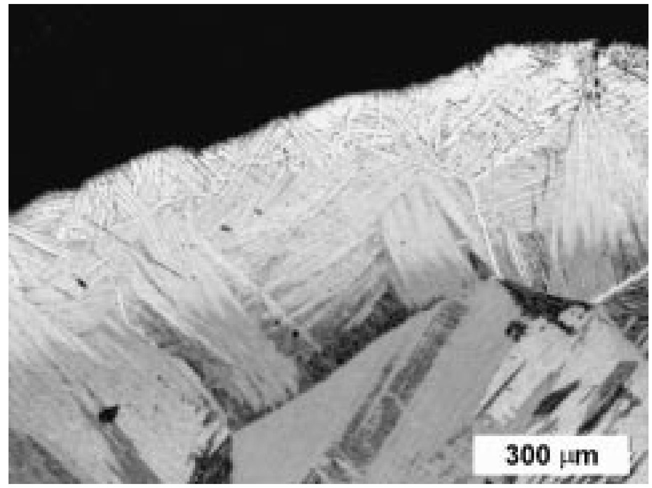


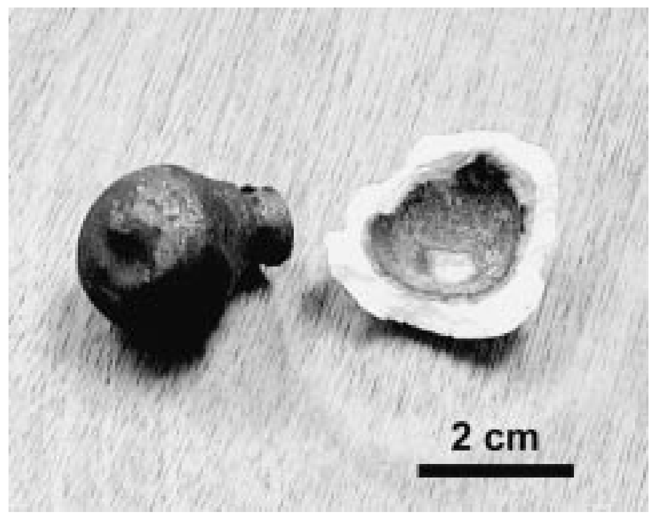
Fig. 4 Microstructure of titanium casting surface

A linear shrinkage of 13% was observed in each direction (*x*, *y*, and *z*), on average, after the first firing. In subsequent firings, little additional shrinkage occurred. This is in accordance with reported shrinkage behavior of porous compacts infiltrated with liquid precursors.[10] The density of the mold material increased significantly with infiltrated zirconia weight gain, to a final value near 60% of theoretical (5.9 g/cm^3). The largest increase in density occurred after the first firing cycle due to particle rearrangement in initial stage sintering. The increase in density was not uniform throughout the part. Scanning electron microscopy revealed that the majority of the colloidal particles collected at the surface of the porous body, resulting in a density gradient.

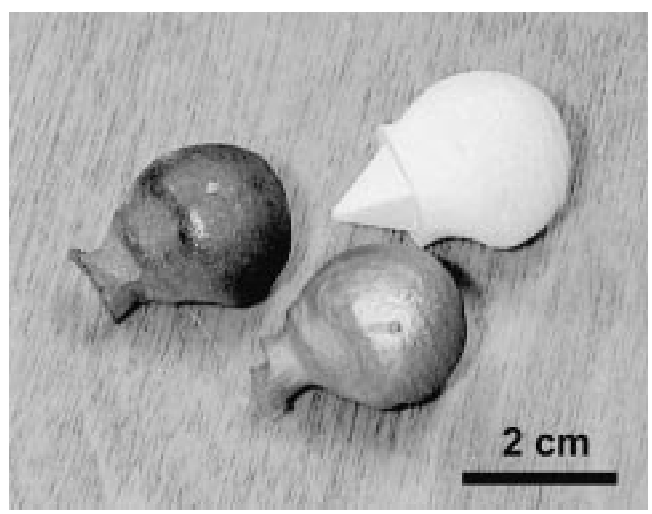
Multiple infiltration cycles smoothed the surface of the SLS produced parts. The roughest surface on an SLS part is on its side, where the stair-stepping effect is prominent. The average roughness, R_a, of the side of the zirconia green part was 14 μm. After multiple infiltrations, the average roughness decreased to 9 μm on all surfaces. The roughness value as a function of infiltrant weight gain is shown in Fig. 3.

X-ray diffraction revealed that the final mold material consisted of yttria-stabilized zirconia and monoclinic zirconia. The monoclinic zirconia formed when the hydrous zirconia precursor crystallized during firing. Hence, a "partially stabilized" structure was created, similar to that previously reported in the literature.[3]

Optical micrographs of an etched section of the casting revealed a surface alpha case (white), shown in Fig. 4. The alpha case consisted of platelike α and intergranular β. The case thickness was approximately 300 μm. The interior portion of the casting exhibited transformed β containing acicular α. This interior microstructure is typical for as-cast Ti-6Al-4V.[11] The average surface roughness of the as-cast specimen was 8 μm. This roughness value lies in the lower end of the range reported for sand casting (6.25 to 25 μm).[12] Considering the molds were created using a patternless free-form fabrication process and that the base zirconia particle size in the mold is 50 μm, the as-cast roughness value is quite good. The roughness value might be improved by using a finer SLS step size or a



(**a**)



(**b**)

Fig. 5 (**a**) As-cast femur ball next to casting mold remnant. (**b**) As-cast and sand-blasted femur ball castings next to nylon model face coat on the mold surface. Photos of the cast femur balls and an SLS-produced nylon model are shown in Fig. 5.

Conclusions

Presintered cubic zirconia powder and a low melting temperature copolymer were selective laser sintered to create titanium casting molds. The SLS shapes were infiltrated with colloidal zirconia and subsequently fired to pyrolyze the copolymer and crystallize the colloidal particles. The resulting mold material consisted of cubic and monoclinic zirconia phases. Increasing infiltrant weight gain improved flexural strength, increased density, and decreased surface roughness of the mold material. Final flexural strength reached above 5 MPa, exceeding values reported for zirconia-based investment casting molds. Average density reached close to 60% of theoretical, but the porosity level varied from the surface to the interior of the part. The majority of shrinkage occurred during the first firing cycle, resulting in approximately 13% shrinkage in each dimension. Roughness decreased from a maximum of 14 μm in the green state to 9 μm in the infiltrated and fired state. Casting molds for the head of a human femur, half-scale, were created from laser-scanned data. Metallographic examination of a casting revealed a thin alpha case and an interior consisting of acicular alpha. The as-cast surface roughness of the titanium casting was 8 μm. This work has demonstrated the feasibility of producing custom titanium components of complex shape from scanned geometric data without part-specific tooling or a wax pattern.

Acknowledgments

We acknowledge Dr. John Kappelmann, Department of Anthropology, The University of Texas at Austin, for the laser-scanned femur data. We also thank Dr. Neal Vail for his guidance and assistance in preparing the copolymer. Finally, we thank Stewart Veeck and Howmet Research Corporation for casting the titanium.

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