

## Fabrication of Ceramic and Metal Matrix Composites From Selective Laser Sintered Ceramic Preforms

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### Abstract

This paper will discuss the tool-less fabrication of functional advanced composites by infusion of a ceramic or metal matrix into Selective Laser Sintered (SLS) porous ceramic preforms using Lanxide's patented matrix infusion processes. The fabrication of porous preforms of particulate ceramics by SLS at the University of Texas at Austin is described in a companion paper. The PRIMEX™ pressureless metal infiltration process was used to infiltrate aluminum matrices into both SiC and Al<sub>2</sub>O<sub>3</sub> particulate SLS preforms to make metal matrix composites without the use of tooling. Also, SiC<sub>p</sub>/Al<sub>2</sub>O<sub>3</sub> ceramic matrix composites were fabricated using the DIMOX™ directed metal oxidation process to grow an Al<sub>2</sub>O<sub>3</sub> matrix into porous SiC particulate SLS preforms. Measured properties and microstructures of the resulting composites will be presented and compared to similar composites made using conventionally fabricated preforms. The rapid prototyping of a SiC<sub>p</sub>/Al MMC electronic power package to near-net shape from an SLS preform will also be described.

### Introduction

Lanxide's matrix infusion processes for fabricating ceramic and metal matrix composites are near-net shape processes in which a matrix is infused into a porous ceramic preform. The final composite shape is dictated by the shape of the preform; therefore, a critical step in the process is the fabrication of a porous preform to near-net shape. Preforms typically have been made using a variety of standard ceramic processing methods, including tape casting, injection molding, green machining, dry pressing, etc. Many of these conventional processes either do not have the complex shape-making capabilities needed or require tooling that is expensive and time-consuming to fabricate for prototypes or small production runs. The newly-emerging Solid Freeform Fabrication technologies, and in particular Selective Laser Sintering (SLS), appear to be ideally suited to fill the need for a method to fabricate porous preforms to near-net shape quickly and without the use of tooling.

One of the objectives of the current work is to evaluate the feasibility of fabricating Ceramic Matrix Composites (CMCs) and Metal Matrix Composites (MMCs) using preforms formed to net shape using SFF processes. Fabrication of the SLS ceramic preforms is described in a companion paper; the results of work to convert these preforms to CMCs and MMCs via Lanxide's matrix infusion processes are discussed below.

### Lanxide's Matrix Infusion Processes

The DIMOX™ directed metal oxidation process for fabricating ceramic matrix composites (Fig. 1) and the PRIMEX™ pressureless metal infiltration process for metal matrix composites (Fig. 2) are similar. In both processes, a ceramic preform is made from the desired reinforcing material, which can be ceramic particulates or fibers. In the current work, SiC and Al<sub>2</sub>O<sub>3</sub> particles ranging in size from 1 μm to 60 μm, which are typical reinforcing materials, were used (fibers were beyond the scope of this work). The preform is formed to the desired shape of the final part, a barrier is applied to the upper surfaces to stop infiltration and retain shape, and the preform is placed in contact with the growth alloy which is typically an aluminum alloy. In the CMC process,

the alloy is heated above its melting point to temperatures of 900°C - 1000°C in air, and the alloy wicks into the preform while simultaneously oxidizing to form an Al<sub>2</sub>O<sub>3</sub> matrix. This wicking and oxidizing process (referred to as "matrix growth") continues, filling the entire preform until the growth barrier on the top surface is reached. The resulting Ceramic Matrix Composite part, consisting of a reinforcement phase and an Al<sub>2</sub>O<sub>3</sub> matrix with small interconnecting channels of Al, is removed from the alloy. The process for fabricating MMCs is similar, except that the alloy is heated above its melting point to temperatures of only 750°C to 850°C in a nitrogen atmosphere; no oxidation of the aluminum occurs as it wicks into the preform to form an aluminum matrix composite. No pressure is required to aid infiltration of the alloy into the preform.

The chief advantages of these processes are:

- near-net shape capability (less than 1% shrinkage compared to preform dimensions)
- uses comparatively inexpensive raw materials and equipment
- no part-specific tooling required after fabrication of preforms

### Experimental Work

As described in the companion paper, Marcus, et. al. supplied Lanxide with both Al<sub>2</sub>O<sub>3</sub> and SiC particulate SLS preforms made using a variety of sintering parameters. Initially, preforms were supplied in the form of 2" x 2" x 1/4" coupons, then after composition and sintering parameters were established, complex-shaped preforms were fabricated. These preforms were evaluated for compatibility with Lanxide's matrix infusion processes. Initially, preforms were made using either 20 μm Al powder or spray-dried PMMA as a binder. We were able to successfully make MMCs and CMCs from both types of preforms; however, the microstructures of the composites made with preforms using the PMMA binder were much more uniform. It was therefore decided that further work would employ the PMMA binder only.

Table 1 gives the properties measured to date for MMCs and CMCs made from SLS preforms. Although SLS preforms were fabricated from both Al<sub>2</sub>O<sub>3</sub> and SiC particulate, work concentrated on SiC because both the CMC and MMC demonstration components were to be fabricated using SiC particulate reinforcement. The reinforcement particle loadings of the SiC preforms ranged from 40 to 46 vol%. At least one preform from each set (specimens fabricated using the same SLS parameters) was infiltrated using Al alloy to make an MMC, and another preform from the set was grown to make a CMC. The SiC preforms generally infiltrated well; no significant differences in matrix infusion behavior for either the CMC or MMC processes were observed for specimens made with the various SLS parameters tested. The main effect observed in preforms fabricated using different SLS parameters was variation in green strength which affected ability to withstand handling. Figure 3 shows a microstructure for the MMC; it can be seen that there is almost no visual evidence of layering of the SiC. However, a relatively large amount of fine porosity (approx. 8 vol% as measured by QIA) was evident, resulting in lower thermal conductivity than would normally be expected. Work is currently concentrating on adjusting infiltration parameters to reduce the occurrence of this porosity.

Figure 4 shows a plot of modulus as a function of particle loading for SiC/Al MMCs made at Lanxide using a variety of preforming techniques; it can be seen that MMCs fabricated from SLS preforms have elastic moduli comparable to MMCs made using other preforming techniques, taking into account the vol% particle loading. Figure 5 shows a similar graph of CTE as a function of loading, and again it can be seen that MMCs made using SLS preforms behave similarly to composites made using conventional preforms.

These results indicate that it should be possible to make fully functional MMC components from SLS preforms. However, many MMC applications require composites with higher reinforcement

loadings than those attained to date. For example, many electronics applications require a CTE of  $\leq 7.0$  ppm/ $^{\circ}$ C, which translates into a particle loading of at least 60 vol%. Less demanding electronics applications require a CTE of approximately 8 ppm/ $^{\circ}$ C, or approx. 55 vol% reinforcement. The most highly loaded MMCs fabricated to date from SLS preforms have had 44 vol% reinforcement. Increasing the reinforcement loading is therefore the most important issue to address in developing functional and commercially viable MMCs made from SLS preforms. Since the reinforcement loading in the composite is a direct function of the preform density, efforts were made to increase the SLS preform density by using a blend of four particle sizes to increase packing efficiency. These initial efforts, described in the companion paper, were unsuccessful; however, it is expected that optimization of the binder system and spray drying parameters may allow the fabrication of higher density SLS preforms.

SLS 15  $\mu$ m SiC particulate preforms were also subjected to the DIMOX<sup>TM</sup> directed metal oxidation process to form a SiC<sub>p</sub>/Al<sub>2</sub>O<sub>3</sub> CMC. Figure 6 shows a photomicrograph of the resulting CMC; it can be seen that the microstructure is quite uniform and there is little evidence of layering. As in the case of the MMCs, most SiC<sub>p</sub>-reinforced CMC applications typically require reinforcement loadings from 55 vol% to 60 vol% or greater. Therefore, increasing the preform density is also important to the fabrication of functional SiC<sub>p</sub>-reinforced CMC components.

The SLS Al<sub>2</sub>O<sub>3</sub> (15  $\mu$ m nominal particle size) preforms with PMMA binder had Al<sub>2</sub>O<sub>3</sub> loadings of 37 - 38 vol% as determined by green density and TGA measurements, and contained approximately 8 wt% binder. The preforms were successfully infiltrated with two aluminum alloys commonly used for automotive MMC applications. Filler loadings in the composites measured by Quantitative Image Analysis (QIA) were around 35 vol% with 1.4 vol% porosity. Figure 7 shows the microstructure of a typical MMC made using an SLS preform. Only slight evidence of layering of the Al<sub>2</sub>O<sub>3</sub> is visible in the SLS composite; the homogeneity of this microstructure is encouraging. It should also be noted that the particle loadings are within the range of loadings needed for many of the automotive applications.

#### Fabrication of Demonstration Articles

As part of the RAPTECH-CMC program, two demonstration articles were selected for fabrication from SLS preforms. The first article was a generic electronic power package to be fabricated from SiC/Al MMC using an SLS preform. The second article was a turbine engine tip shroud to be fabricated from SiC/Al<sub>2</sub>O<sub>3</sub> CMC using an SLS preform. As discussed in the companion paper, preforms for both articles were successfully fabricated using SLS. The preform for the generic power package was successfully infiltrated to form a near-net shape MMC part (Fig. 8). Lateral dimensions of the infiltrated MMC part were consistently 17% to 18% larger than the specified drawing dimensions. The thickness of the part was within the  $\pm .005$ " specification. These dimensional differences were due to oversized dimensions of the SLS preform; the infiltration process causes a small (<1%) shrinkage compared to the preform dimensions. Since this part was among the first of this design to be fabricated using SLS, it is expected that dimensional accuracy will improve as more preforms are fabricated. Work to fabricate the CMC tip shroud from the preform is currently in progress.

#### Conclusions

The results to date indicate that Selective Laser Sintering may in the future be used as a fast, tool-less route to obtaining preforms for prototypes and small production runs. Furthermore, based on the properties measured to date, if the preform density can be improved it is very possible that the resulting composite properties could be fully interchangeable with composites made using more conventional preforming methods. This would mean that parts for prototypes

and small production runs could be made using SLS preforms, and then if a larger production run is required high volume preforming methods could be used to make essentially the same material.

#### Acknowledgements

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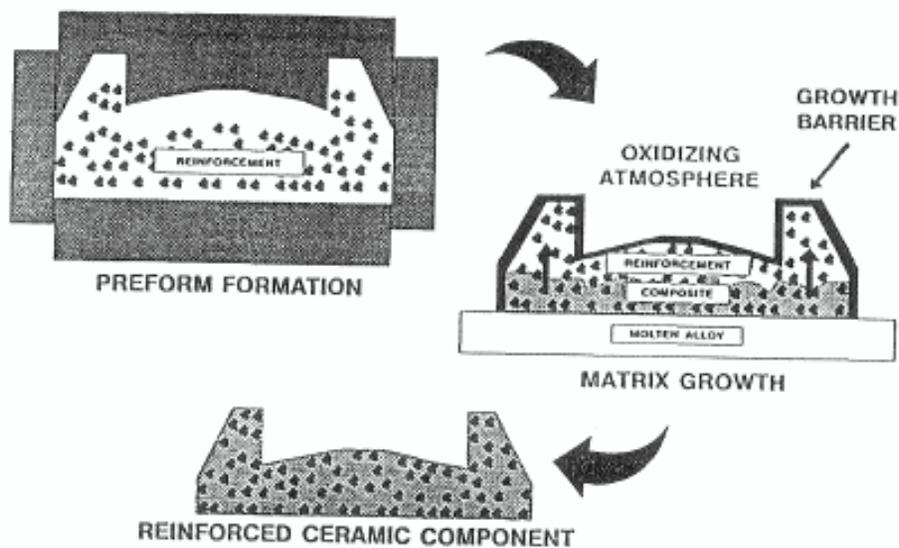


Figure 1. A Schematic of the DIMOX™ directed metal oxidation process for making CMCs

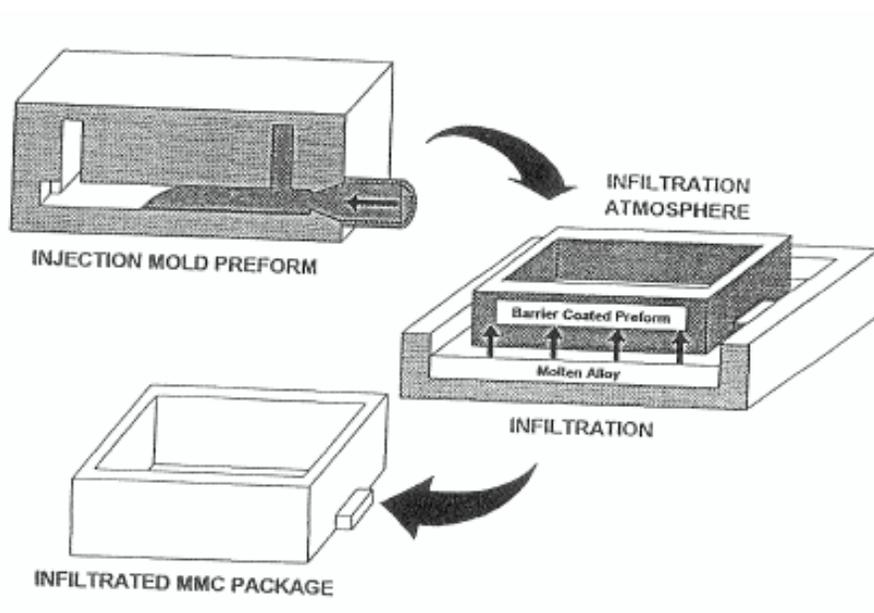


Figure 2. A Schematic of the PRIMEX™ pressureless metal infiltration process for making MMCs

Table 1. Properties Measured for Composites Fabricated from SLS Preforms

	<u>Al<sub>2</sub>O<sub>3</sub><sub>p</sub>/Al MMC</u>	<u>SiC<sub>p</sub>/Al MMC</u>	<u>SiC<sub>p</sub>/Al<sub>2</sub>O<sub>3</sub> CMC</u>
<b>Quantitative Image Analysis</b>			
Reinforcement Loading	38 vol%	45 vol%	41 vol%
Porosity/Other phases	1 vol%	8 vol%	3 vol%
Metal	61 vol%	47 vol%	20 vol% (residual metal)
Al <sub>2</sub> O <sub>3</sub> matrix (CMC)			36 vol%
Density	3.12 g/cm <sup>3</sup>	2.95 g/cm <sup>3</sup>	3.47 g/cm <sup>3</sup>
Sonic Modulus	130 GPa	180 GPa	286 GPa
CTE (25°C - 100°C)	14.5 ppm/°C	9.1 ppm/°C	7.5 ppm/°C
Thermal Conductivity	67 W/m-K (Al - 7 Mg) 83 W/m-K (Al-4.5 Cu-4 Mg)	115 W/m-K	----
Fracture Toughness	----	8.3 MPa-√m	8.0 MPa-√m
Flex Strength (4-point)	----	275 MPa	260 MPa

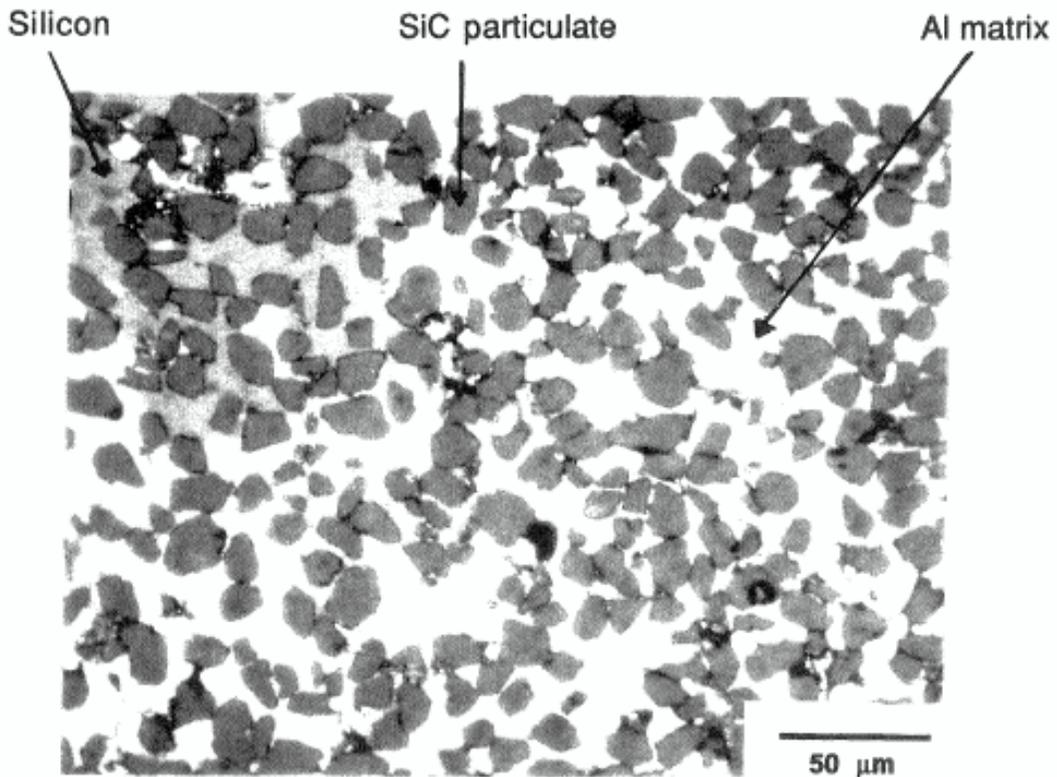


Figure 3. Microstructure of SLS SiC<sub>p</sub>/Al MMC

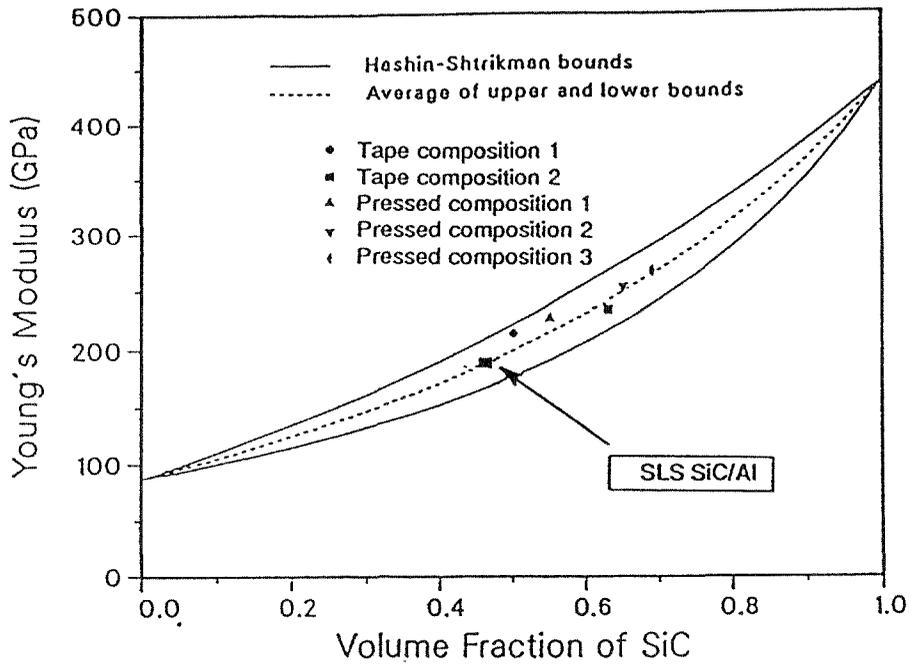


Figure 4. Elastic Modulus as a Function of Loading in SiC<sub>p</sub>/Al MMC

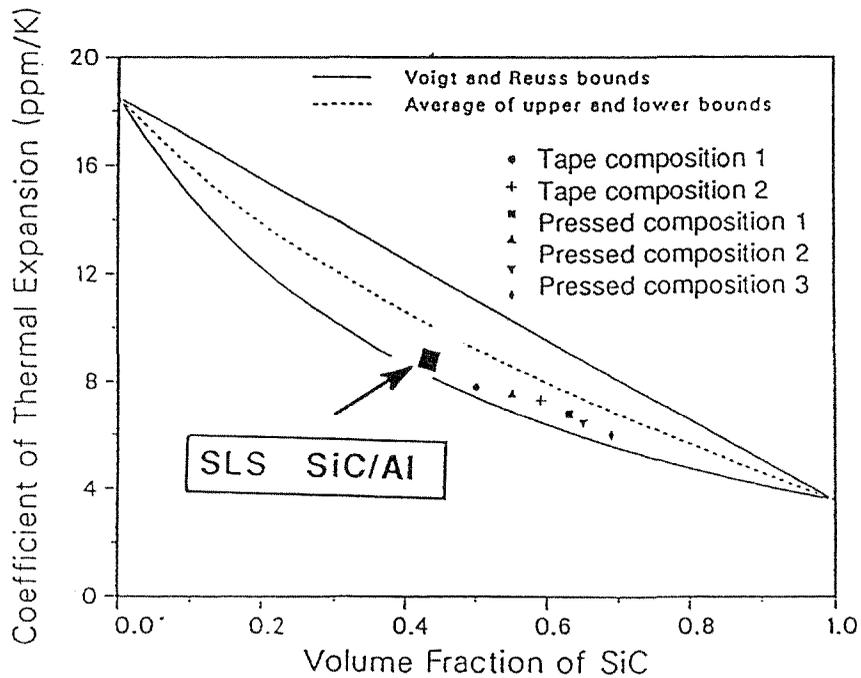


Figure 5. CTE as a Function of Loading for Lanxide's SiC<sub>p</sub>/Al MMCs

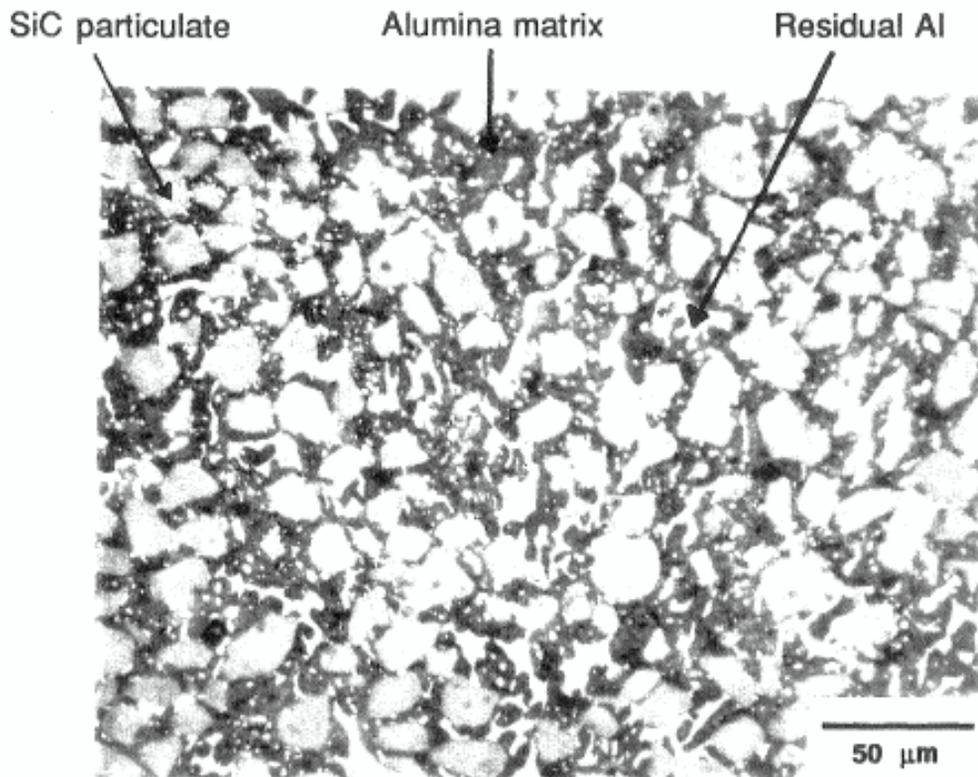


Figure 6. Microstructure of SLS  $\text{SiC}_p/\text{Al}_2\text{O}_3$  CMC

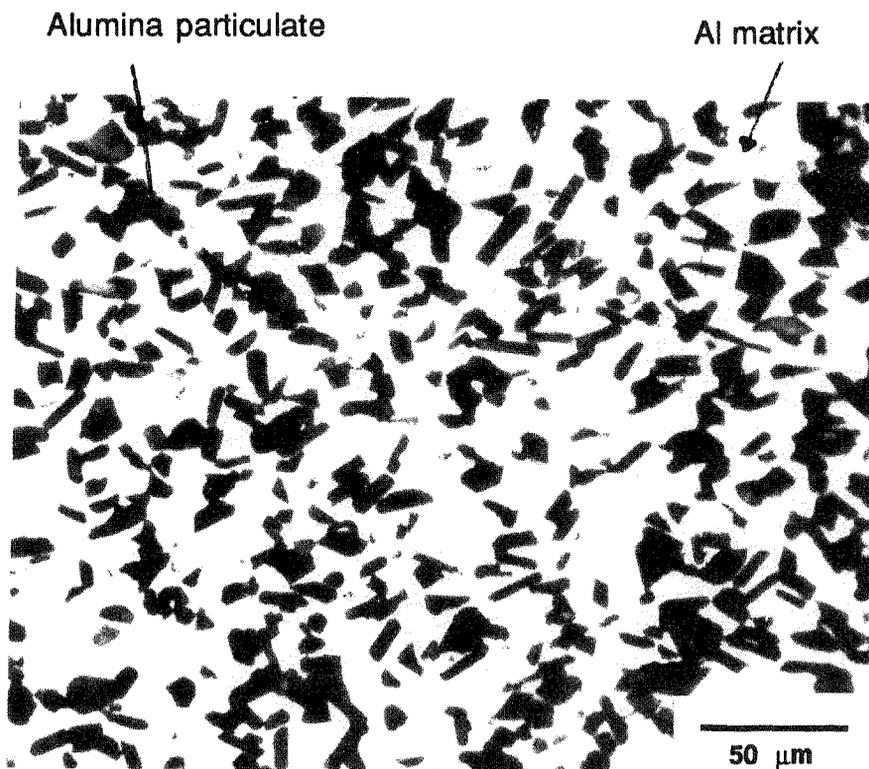


Figure 7. Microstructure of SLS  $\text{Al}_2\text{O}_{3p}/\text{Al}$  MMC

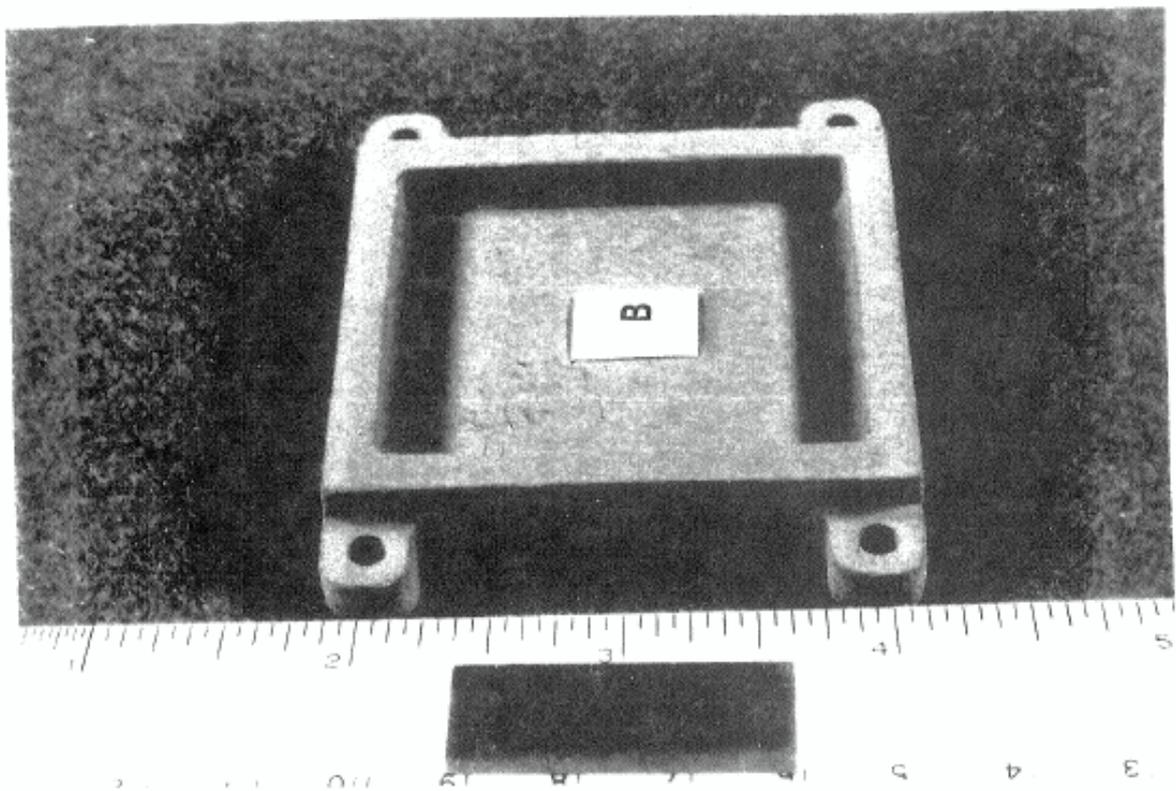


Figure 8. SLS Preform for Electronic Power Package

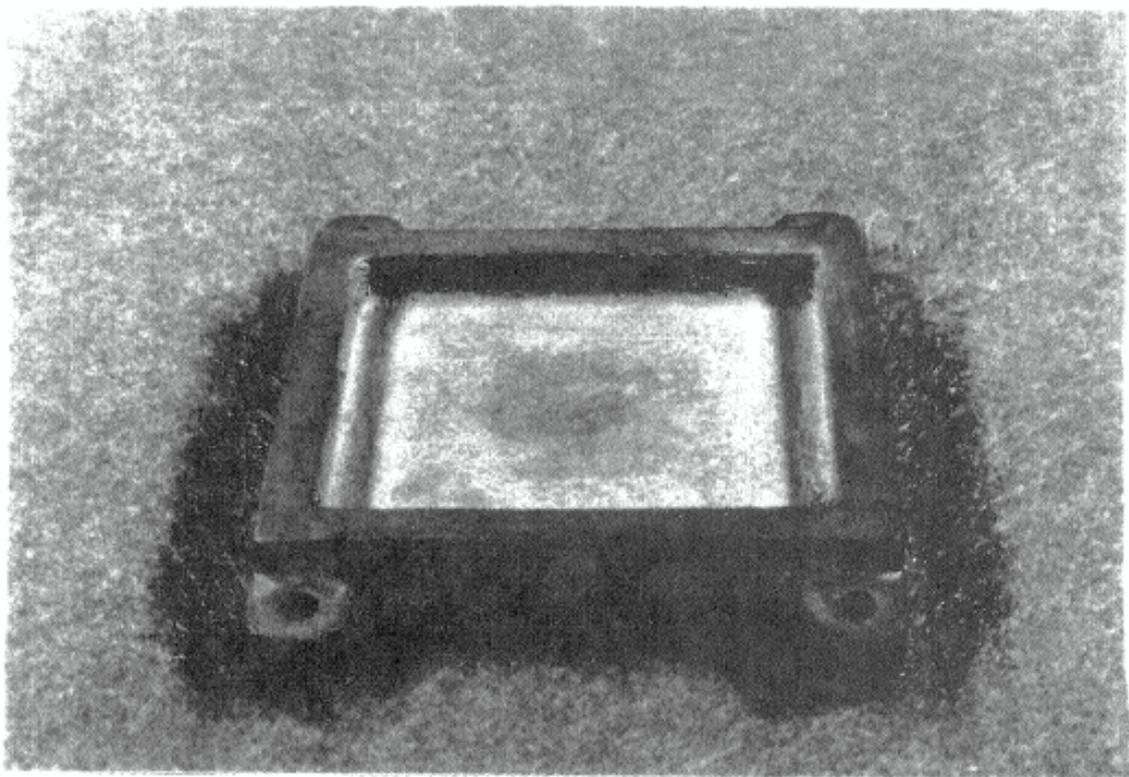


Figure 9. SLS Preform Shown in Figure 8 Infiltrated to Form MMC