Silicon Carbide Shapes By Selected Area Laser Deposition Vapor Infiltration

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Abstract

Selected Area Laser Deposition Vapor Infiltration (SALDVI) is a unique combination of selected area laser deposition, chemical vapor infiltration and layered powder handling techniques that can be used to fabricate silicon carbide (SiC)/SiC composite shapes. This paper discusses a SALDVI process under investigation which selectively infiltrates SiC powder with SiC generated by decomposition of a gas precursor under a scanned laser beam. A general description of the process, including some of its inherent advantages is presented. Experimental results which explore beam interaction, powder size and infiltration time effects are also presented.

Introduction

Selected Area Laser Deposition Vapor Infiltration (SALDVI) is a new solid freeform fabrication (SFF) technique that has the potential to produce both monolithic and composite metal and ceramic shapes. SALDVI uses the selected area laser deposition (SALD) technique [1] to provide material used for simultaneous localized chemical vapor infiltration . The process begins by generating a two dimensional pattern on a powder substrate with a scanned laser. The high temperature at the laser's focal point pyrolitically decomposes a gas precursor(s), and the products of decomposition locally infiltrate and bond the powder particles together. After each two dimensional pattern has been scanned and infiltrated, a fresh layer of powder is spread on top of the previous infiltrated layer using technology developed for selective laser sintering [2]. The process is repeated until the desired three dimensional shape is built. The finished part has a composite structure consisting of the starting powder bonded into a matrix of vapor deposited material.

The SALDVI process has great potential due to several inherent features. These features, discussed below, include the potential to produce fully dense shapes without post processing, a wide materials selection, and the elimination of dimensional constraints associated with traditional chemical vapor infiltration techniques.

High density has been one of the main goals of most SFF processes because of the direct relationship between density and mechanical integrity. Many SFF processes must include secondary heat treatments to produce shapes with satisfactory density. This adds time and cost to production, and complicates the initial build of the shape by forcing consideration of shrinkage effects [3,4]. The SALDVI process has the potential to make fully dense shapes without post processing because it incorporates vapor deposition, a process that can inherently produce theoretically dense material.

The SALDVI process has the potential to produce shapes from a wide range of materials. Gas precursors have been developed, particularly by the microelectronics industry, for the vapor deposition of a variety of elements and compounds including such useful materials as Al, AlN, Al_2O_3, Si, SiO_2, Si_3N_4, SiC, Ti, TiN, TiO_2, Zr, ZrO_2, Hf, HfC, W, WC, Co, Fe, Ni, Cr and Cu [5]. Many of these precursor systems are candidates for laser deposition, and therefore potential candidates for the SALDVI process. With the proper selection of gas precursor(s) and powder bed material(s), a variety of metal/metal, metal/ceramic, and ceramic/ceramic structures could be produced.

The SALDVI process could also eliminate the dimensional constraints inherent to traditional chemical vapor infiltration (CVI) techniques. Traditional CVI processes are limited to small shapes because the outside of the shapes tend to seal and prevent further infiltration before the interior is fully dense [6]. More complex CVI processes are currently under investigation such as forced flow reverse gradient and microwave heating that impose a reverse temperature gradient on the preform, i.e. heat from the inside out, to improve the infiltration of larger shapes [7,8]. The SALDVI process avoids part size limitations by infiltrating consecutive thin layers to build shapes. This makes possible the production of shapes with dimension limited only by machine size.

Silicon Carbide/Silicon Carbide SALDVI

Initial work characterizing SALDVI has concentrated on producing SiC/SiC composite shapes using SiC powders and a SiC gas precursor. SiC powder was chosen because of the material's desirable engineering properties, and its 2560°C decomposition temperature [9,10]. SiC's high decomposition temperature makes it stable under the laser beam, thereby minimizing any melting or decomposition of the powder that would complicate the vapor infiltration process. Tetramethylsilane (TMS) was chosen as the gas precursor because of its demonstrated ability to produce SiC in a laser deposition process [11], and its ease of handling. TMS is a liquid at room temperature with a vapor pressure that is sufficient for vapor deposition processes.

Preliminary tests were run to establish basic process parameters for use in multi-layer shape fabrication. The effect of beam interactions on power delivered to the surface, the effect of powder size on infiltration behavior and the degree of infiltration versus time are reported on in this paper.

Beam effects

Absorption of laser energy by the precursor gas is an important process consideration because it directly reduces laser power at the powder surface. Organic molecules such as TMS tend to absorb infrared energy through molecular vibration/photon interactions [12]. The absorption coefficient of TMS was determined experimentally by directing the SALDVI CO_2 laser beam through a chamber filled to various pressures of the precursor gas. Laser power leaving the chamber was measured using a standard power meter and compared to the power entering the chamber. Experimental data fit the Beer-Lambert relationship, which describes the transmission of energy through a medium as an exponential function of gas pressure and distance.

$$I = I_0 \exp(-\alpha p x) = \text{transmitted intensity , in watts}$$
$$I_0 = \text{initial intensity , in watts}$$
$$\alpha = 3.62\text{E-}4 \ \text{Torr}^{-1}\text{cm}^{-1} = \text{absorption coefficient}$$
$$p = \text{pressure, in Torr}$$
$$x = \text{absorption length, in centimeters}$$

Reflectivity of the surface is also an important process consideration because reflected laser power is not available for powder/infiltrant heating. The reflectivities of SALDVI candidate materials were experimentally determined using the SALDVI CO_2 laser and a standard integrating sphere. The beam was directed onto the test material surface, and the power reflected was collected by the integrating sphere and recorded. The ratio of reflected power to beam power is reflectivity.

Material	**Reflectivity**
0.6 -0.8 μm SiC powder	0.42 - 0.50
12μm SiC powder	0.25 - 0.45
100μm SiC powder	0.20 - 0.50
hot-pressed SiC (solid)	0.16 - 0.60

Table 1: Reflectivity for powder and solid SiC surfaces, CO_2 wavelength energy.

Note that the two reflectivity values reported for each material do not represent a continuous range in the data, but rather the high and low values of a periodic fluctuation observed for all materials tested except the 0.6 - 0.8μm powder. The periodic fluctuations in reflectivity, most apparent in the solid SiC data, see Figure 1, are caused by CO_2 laser mode hopping. An unstabilized CO_2 laser has a nominal output wavelength of 10.6μm, but can periodically mode "hop" through several different modes. At each mode the laser produces a different wavelength, with wavelengths ranging from 10.55μm to 10.65μm for the SALDVI laser. SiC reflectivity dramatically increases from 0.25 to 0.8 as wavelength increases from 10μm to 11μm [13]. As a result, the reflectivity of the surface changes significantly as the laser output wavelength changes.

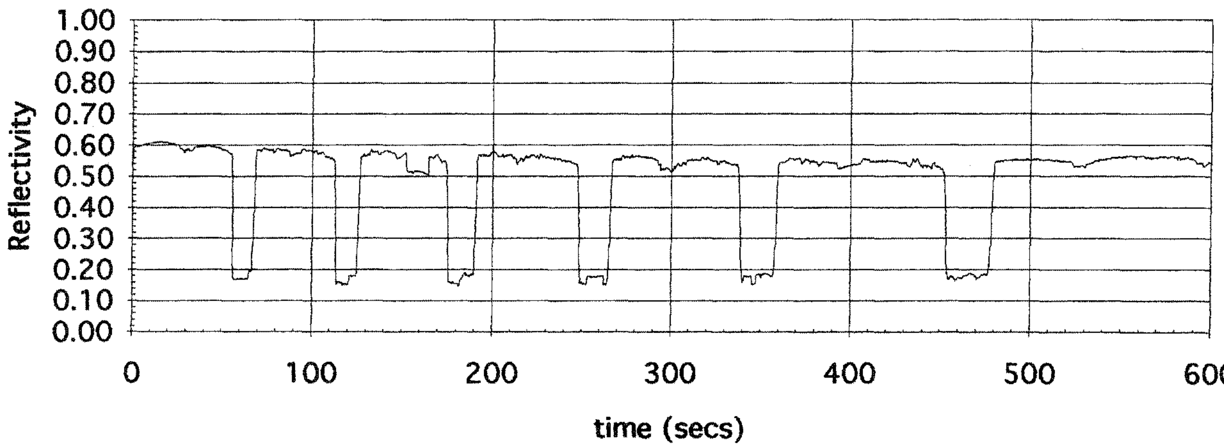

Figure 1: Reflectivity versus time, hot-pressed β-SiC, 10.6μm (nominal) wavelength.

Powder size effect

Figures 2 and 3 are cross-sections of infiltrated 12μm and 100μm powders. A static beam was used with constant laser power and gas pressure.

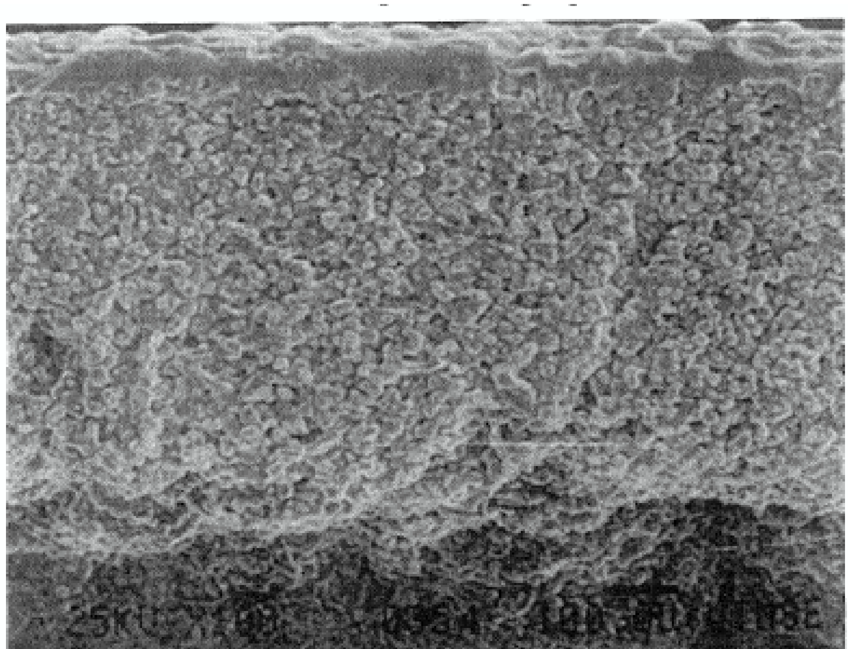

Figure 2: Static beam infiltration - infiltration time 30 seconds, 15μm powder, 25Torr TMS gas pressure, 20watts laser power, cross-section, 200X.

Figure 3: Static beam infiltration - infiltration time 120 seconds, 100μm powder, 25Torr TMS gas pressure, 20watts laser power, cross-section, 150X.

Infiltration time effect

Figures 4 and 5 are cross-sections of material infiltrated different lengths of time. A static beam was used, and gas pressure, laser power and powder size (12μm) were held constant.

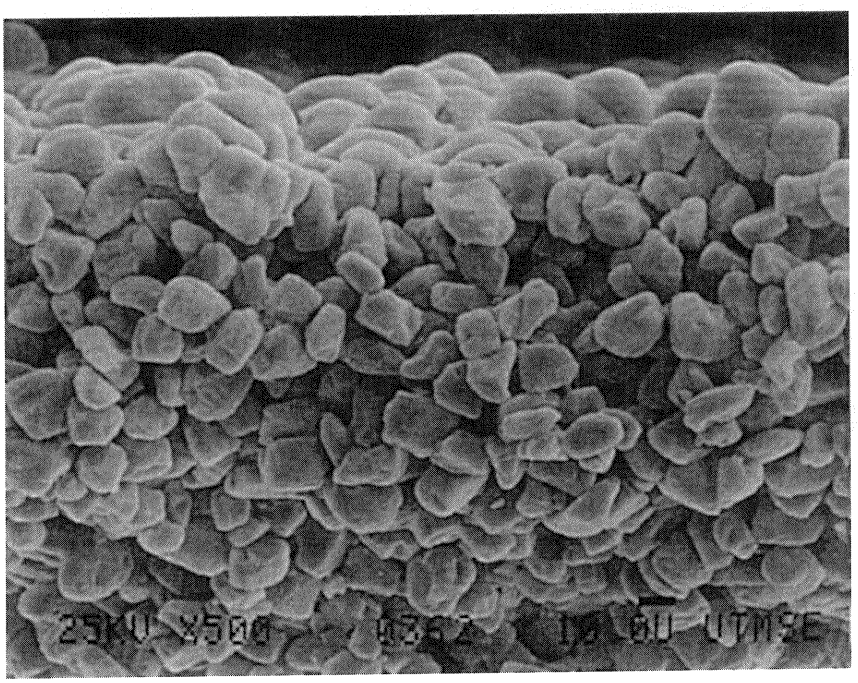

Figure 4: Static beam infiltration - infiltration time 3 seconds, 15μm powder, 10Torr TMS gas pressure, 20watts laser power, cross-section, 500X.

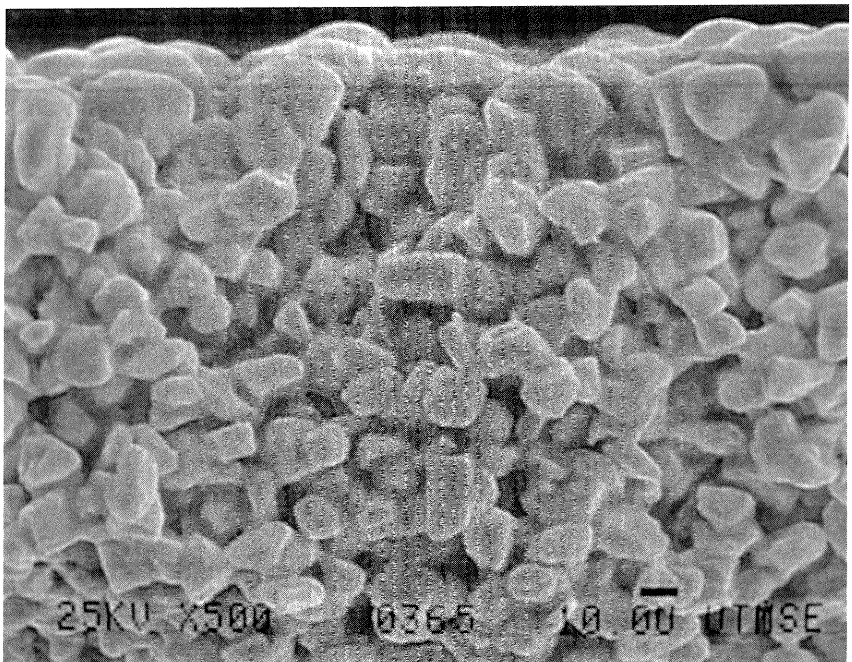

Figure 5: Static beam infiltration - infiltration time 5 seconds, 15μm powder, 10Torr TMS gas pressure, 20watts laser power, cross-section, 500X.

Production of multi-layer parts

Multi-layer tests were run to determine if multiple layers of material could be infiltrated and bonded together to produce a dense multi-layer part. Figures 6 and 7 are cross-sections of one of the test cases.

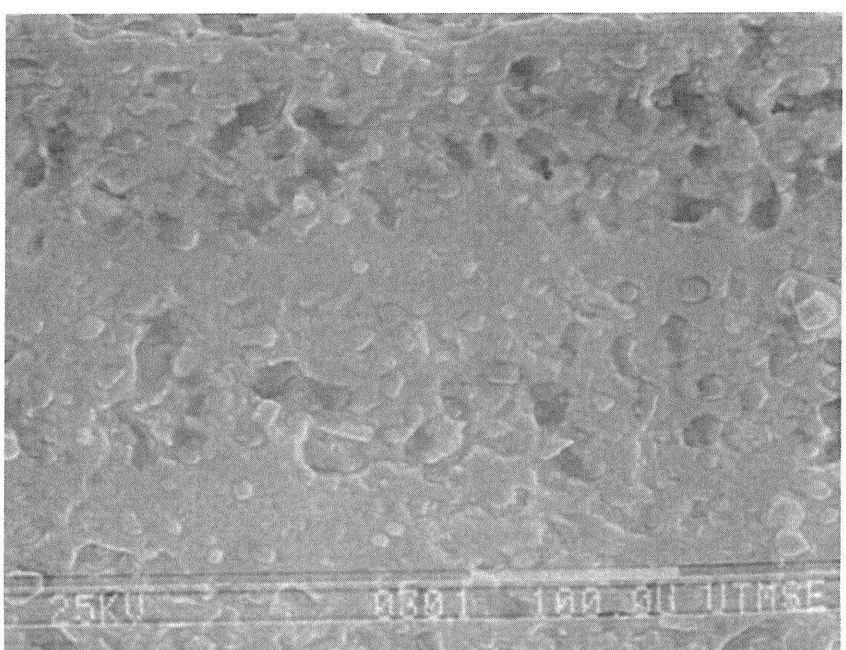

Figure 6: Multi-layer cross-section, top three layers, layer thickness 110μm, 15μm powder, 20Torr TMS gas pressure, 20watts laser power, static beam.

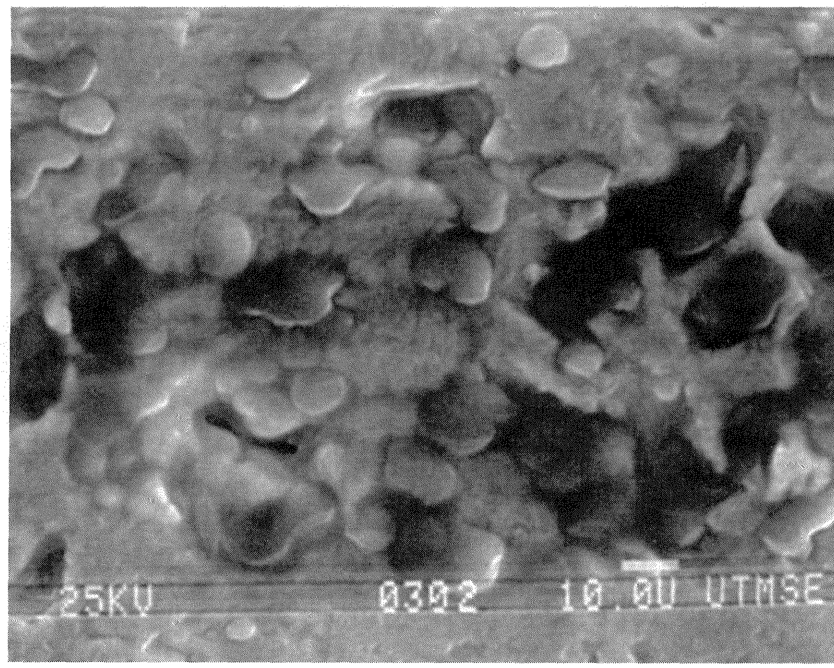

Figure 7: Multi-layer cross-section, top layer including interface, 15μm powder, 20Torr TMS gas pressure, 20watts laser power, static beam.

Discussion

The rapidly changing reflectivity of SiC in the 10.6μm wavelength range makes it difficult to control temperature under the CO_2 laser beam. The immediate solution will be to use a Nd-YAG laser (1.06μm wavelength). This laser should make temperature easier to control because the shorter YAG wavelength is not absorbed by the TMS, and the reflectivity of SiC in this wavelength range is essentially constant. A much more robust solution to the temperature problem will be to use a closed-loop control system like the one currently under development by Benda [14]. That system controls temperature under the beam by measuring surface temperature at the beam focus and then using that information to control laser power. A similar technique using a two colored optical pyrometer is currently under consideration for the SALDVI process.

Powder size has a large effect on infiltration behavior. The smaller powder infiltrates much more rapidly, probably due to increased surface area driving the decomposition reaction. Further testing is required to determine optimum temperature, gas pressure and powder size to achieve maximum infiltration density.

Significant infiltration of the material under the beam occurs in the first five seconds. This represents a part build rate on the order of $2mm^3$/minute, assuming a single 1mm diameter beam is used. It will be possible to speed up the process using variable focus or multiple beam techniques.

Conclusions

Preliminary studies documenting the effects of beam interactions, powder size, and infiltration time have been presented. A multiple layer "proof-of-concept" consisting of a SiC/SiC composite structure produced by the SALDVI process was also presented. Future work will concentrate on maximizing part density and build rate by optimization of gas chemistry, gas pressure, process temperature and powder size. Additional precursors to SiC and other materials will also be investigated.

Acknowledgments

Special thanks to the Office of Naval Research for supporting this work. ONR contract N00014-92-J-1514.

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