SALDVI Optimization for the Tetramethylsilane - Silicon Carbide System

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Selective Area Laser Deposition Vapor Infiltration (SALDVI) of silicon carbide powder infiltrated with silicon carbide deposited from tetramethylsilane (TMS) was studied. The effects of deposition time, temperature, and gas precursor pressure are discussed. The discussion centers on the efforts to properly balance these parameters to produce multi-layered shapes with structural integrity, particularly for use as the matrix material for shapes containing embedded devices. This includes optimizing scan speed, deposition temperature, and gas pressure to maximize infiltration to increase density and layer to layer bonding, and minimize excessive deposition to maintain critical dimensions. Initial powder properties are also optimized to minimize bulk motion in the powder bed during deposition, which was observed and identified as a mechanism that reduces inter-layer bonding.

Introduction

The ability to fabricate structurally sound complex shapes quickly and directly in the desired final material without post processing is the ultimate goal of Solid Freeform Fabrication. One possible route toward direct single step fabrication of a variety of metals, ceramics, and composites is the Selective Area Laser Deposition Vapor Infiltration (SALDVI) process[1]. SALDVI builds shapes by selectively infiltrating layers of powder with material thermally decomposed from a reactive gas. The hot spot generated by directing a laser beam onto a powder bed defines both the selective nature and spatial resolution of the process. Scanning the beam across the powder bed defines each two dimensional layer. Each additional layer is produced by spreading a measured layer of powder on the previous layer and scanning the beam in the desired 2-D pattern. Under the proper process conditions, the layer is simultaneously infiltrated and bonded to the previous layer. In this manner a 3-D shape is built consisting of powder particles bonded within a matrix of material deposited from the reactive gas or gas mixture. Because material is added from the gas phase to the powder during the SALDVI process, high densities are possible without post processing or shrinkage considerations.

The infiltration of powder by laser-induced gas decomposition distinguishes SALDVI from other gas phase SFF approaches. For example, Selective Area Laser Deposition (SALD) builds shapes entirely from thermally decomposed gases. Such SALD processes have been used for fabricating rods and fibers of constant diameter[2] and varying diameters[3], springs and helixes[4], in complex micro devices composed of rods of various orientations[5], and in the joining of ceramics[6]. The advantage of SALDVI in producing 3-D bulk shapes from sliced CAD models is that the uninfiltrated powder provides a support structure for producing overhangs. Also, confining the deposition to thin powder layers provides dimensional control in the third dimension, which can be a difficulty in SALD due to unstable growth kinetics[7]. Furthermore, SALDVI provides additional opportunities over SALD to tailor the local chemistry and microstructure.

The SALDVI process as a SFF method was introduced with the infiltration of SiC powder with SiC deposited from the thermal decomposition of tetramethylsilane (TMS, SiC_4H_{12}) gas[1]. This initial effort investigated the effect of some important process parameters involved in SALDVI: gas pressure, powder size, and heating time. The necessary interactions between the laser, reactive gas, and powder bed such as reflectivity, absorptivity, and transmissivity relevant for the SALDVI process were discussed. Multiple layer bulk shapes of SiC were fabricated; however, poor mechanical bonding between adjacent layers was identified as a key factor limiting structural integrity. This paper builds on that initial research in the development of the SALDVI SiC system. Process improvements are discussed including the use of feedback from an optical pyrometer to control the laser power and video monitoring of the powder bed during the deposition process. The focus is the effect of processing conditions on the bonding of adjacent layers. Adequate bonding between adjacent layers is required for structural integrity of the bulk shape and in using the SALDVI shapes in the fabrication of embedded devices[8]. Single and multiple layer samples were fabricated using a scanned laser beam. Characterization includes microscopic observations of the infiltration density and the layer-to-layer bonding. The SALDVI system used in these experiments[9] consists of a 50 watt CO_2 laser and a fully automated powder delivery, closed loop temperature control, and laser scanning system.

Closed Loop Temperature Control

One of the most important processing parameters in the densification of a powder bed by vapor infiltration is thermal history. For a given laser beam power profile, the distribution of temperature in the powder bed initially depends on the optical properties (reflectivity and absorptivity) and thermal properties (thermal conductivity) of the powder. As the infiltration of the powder proceeds, the optical characteristics and the thermal conductivity of the substrate change with time. As the SALDVI process proceeds, the part becomes larger, denser and a better heat sink, especially when depositing a good thermal conductor like SiC. Thus, more laser power is required to maintain a given temperature. To smooth out these temperature fluctuations throughout the infiltration process, feedback from an optical pryometer is used to continually adjust the input voltage to the laser. The pyrometer output is compared to a target temperature parameter set by the operator at the beginning of an infiltration experiment. Any difference in temperature between the pyrometer reading and this target temperature parameter is used to modulate the laser power. Using this closed loop control system, infiltration experiments have been performed at a constant pyrometer value. It is important to note that the temperature profile in the substrate due to laser beam heating is not uniform. Rather, it follows the gaussian-shaped profile of the incident laser beam. So while the pyrometer reading gives an indication of the temperature of the powder bed, it is misleading to describe a temperature distribution with a single number. Hence we use the term target temperature parameter rather than referring to the pyrometer reading as the absolute deposition temperature.

Effect of the Target Temperature Parameter on Infiltration of Single Layers

The sensitivity of the infiltration to the target temperature parameter is illustrated in Figure 1. The cross-sections reveal the single layer deposition and infiltration into a thick powder layer at target temperature parameter values of 975, 1000, and 1050°C. Infiltration density increases significantly when the target temperature parameter increases from 975 to 1000°C. Further increase to 1050°C does not improve the infiltration into the powder bed. Rather, gas phase material deposits primarily at the top surface of the powder layer in a SALD mode, sealing off the powder bed and preventing further infiltration. This effect is due to the strong dependence of the decomposition rate on temperature coupled with the existence of higher temperatures at the powder surface due to laser surface heating. The result is a thick layer of SALD material deposited on a poorly infiltrated powder layer, obviously an undesirable result for SALDVI. Thus, a target temperature parameter value of 1000°C gives the optimum infiltration for SALDVI.

Effect of Scan Speed on Infiltration of Single Layers

Heating time is a critical parameter in SALDVI. For a scanned beam, the time duration that a given area remains heated depends on the beam diameter and the speed at which it is scanned across the powder bed. Figure 2 shows infiltration cross-sections for a 3.5-mm diameter beam scanned at three scan speeds. The infiltration density increases as the scan speed decreases. At a scan speed of 5 μm/s, significant infiltration occurs to depths greater than 400 μm into the thick powder layer.

Effect of Gas Precursor Pressure on Infiltration of Multiple Layers

In order for SALDVI to be useful in fabricating 3-D shapes, processing conditions that simultaneously densify a particular layer as well as bond it to the previous layer must be identified. Figure 3 shows the effect of varying the tetramethlysilane precursor gas pressure on infiltration and bonding of adjacent layers. Each sample consists of two 250 μm thick layers on a thick base layer. The infiltration density is low at a TMS pressure of 5 torr, increases at 10 torr, and increases further at 20 torr. At 20 torr there is significant infiltration and bonding between adjacent layers, although some isolated porosity remains.

Effect of Scan Geometry on Interlayer Bonding of Multiple Layers

The scan geometry used in the target temperature parameter, scan speed, and gas pressure experiments discussed above consisted of simple straight line scans in which the laser was turned on, scanned at a given speed for 10 mm, and then turned off. Using the best combination of these parameters, we obtain cross-sections showing good infiltration and bonding between adjacent layers as in Figure 3c. Larger and more complex shapes, such as squares, rectangles, and disks, are fabricated by scanning a series of lines separated by a given scan spacing. Figure 4 shows a cross-section of a multiple layer rectangle scanned at a beam spacing of 3 mm. The infiltration density within individual layers is high (Figure 4a), however, a significant separation is apparent between adjacent layers (Figure 4b). Video observations of the powder bed during the SALDVI process reveal a powder bubbling effect occurring in the powder bed. Gas appears to be bubbling up through the surface powder layer from near the submerged previous layers. Although not yet completely understood, possible explanations of this flow may be volume expansion of the gas in the powder bed due to decomposition of the TMS molecules or natural convection effects due to the large temperature gradients. The powder bubbling has been minimized by using larger SiC particles in the powder bed, and by adjusting the composition of the gases in the reactive gas mixture. It is possible that this powder levitation phenomenon is the cause of the separation between layers observed in some multiple layer shapes.

Conclusions

SALDVI of SiC has been shown to depend strongly on three parameters: temperature, heating time, and gas pressure. Multiple layer deposits of low porosity have been built by optimizing these parameters. The primary challenge in building SiC shapes without post processing is the gap between layers, believed to be caused by bulk motion of powder during infiltration. Several approaches are under consideration to overcome this challenge, including closer examination of the effects of gas mixture, and powder size.

Acknowledgements

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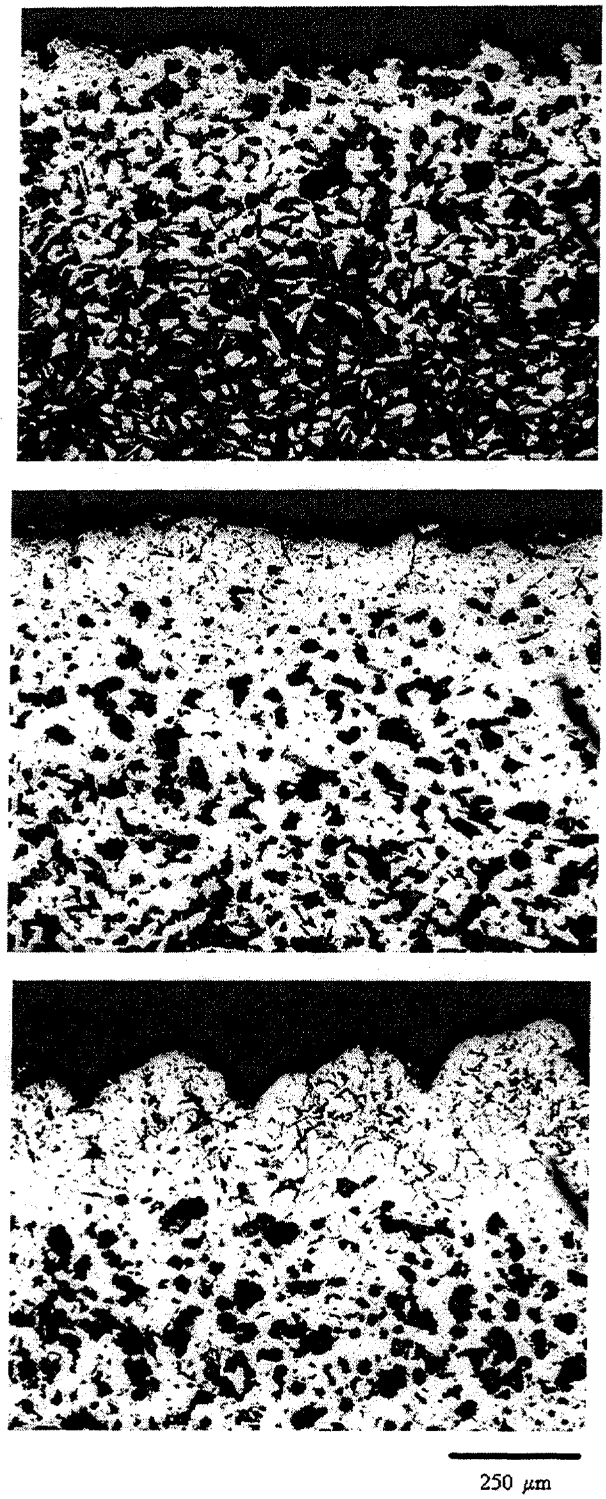

Figure 1. Effect of Target Temperature Parameter
(a) 975 °C (b) 1000 °C (c) 1050 °C
10-20 μm SiC powder, 25 torr TMS + 25 torr H_2, 5 μm/s scan speed, single layer into thick powder

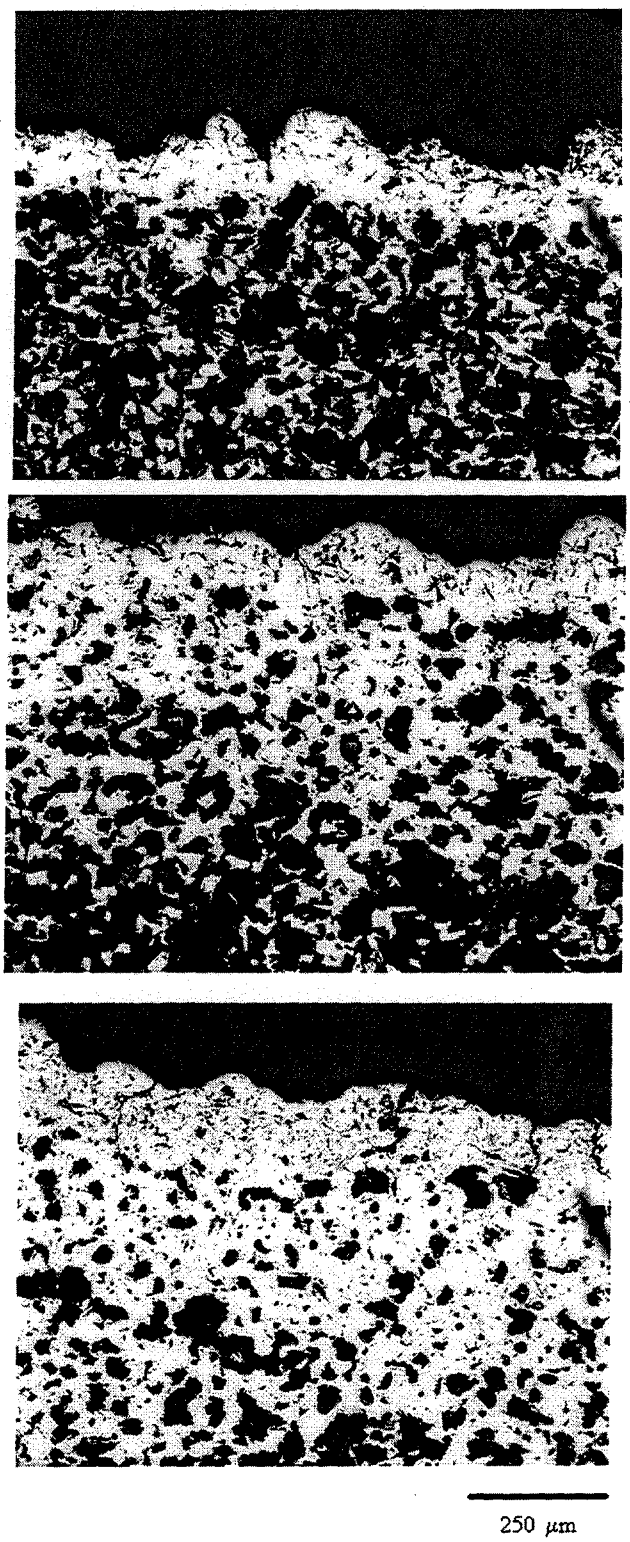

Figure 2. Effect of Scan Speed
(a) 20 μm/s (b) 10 μm/s (c) 5 μm/s
10-20 μm SiC powder, 1100 °C target temperature parameter,
25 torr TMS + 25 H_2, single layer into thick powder

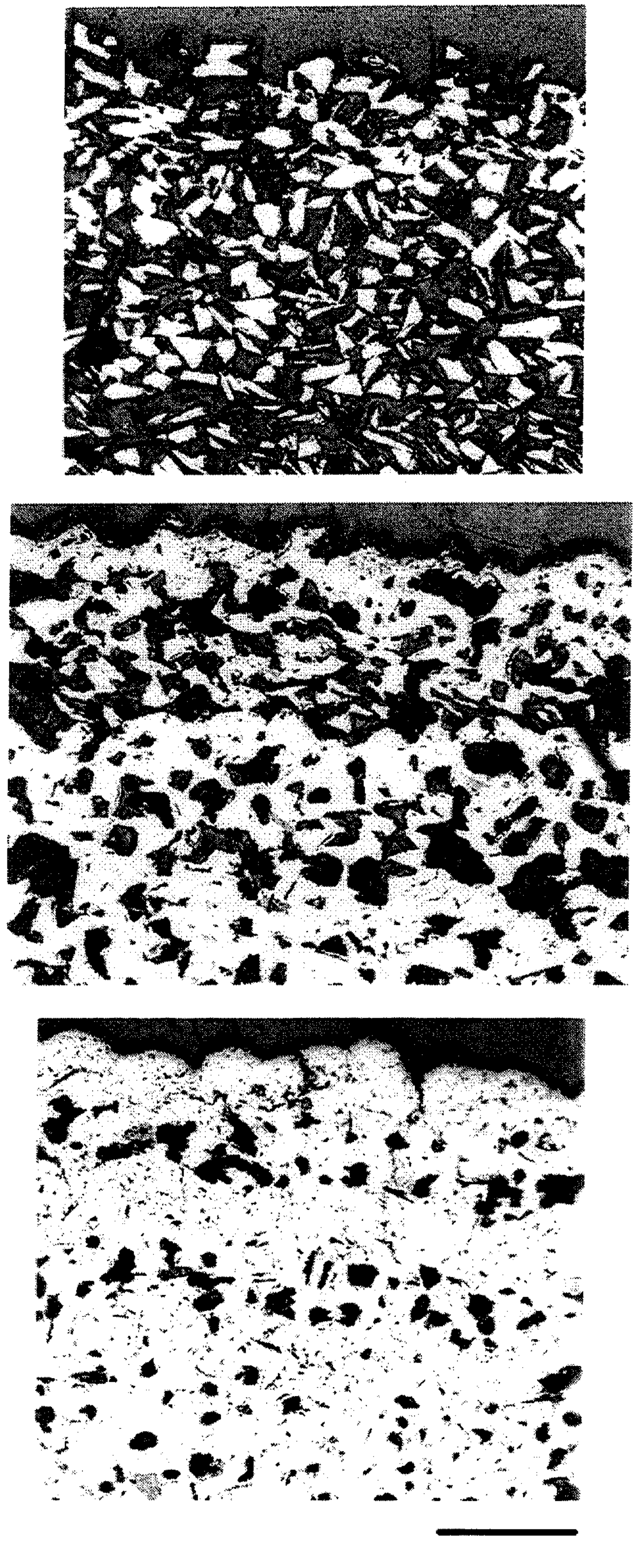

Figure 3. Effect of Tetramethylsilane Gas Pressure
(a) 5 torr (b) 10 torr (c) 20 torr
60-80 μm SiC powder, 1000 °C target temperature parameter, 5 μm/s scan speed, two 250 μm layers on a thick base layer

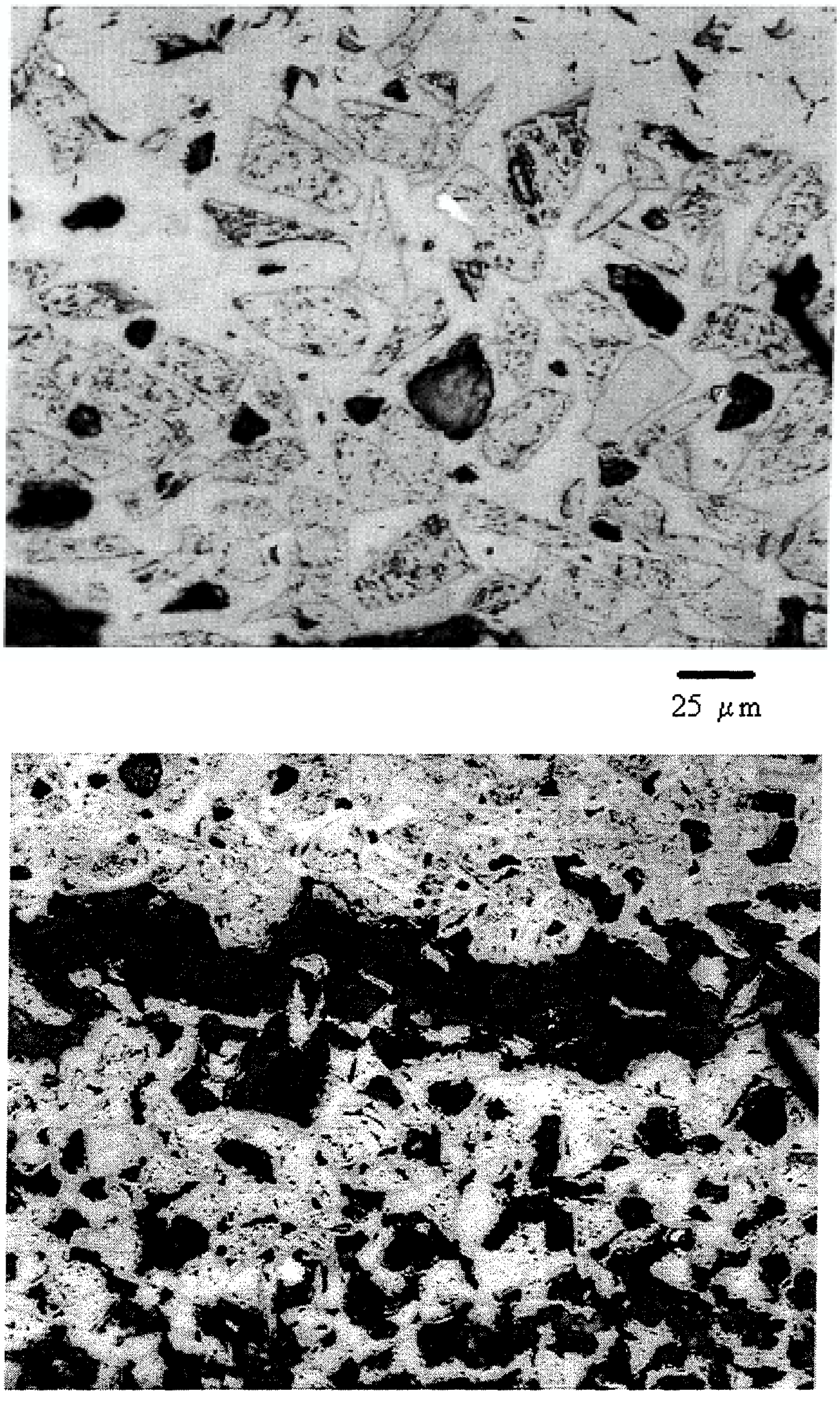

Figure 4. Multiple layer rectangular shape
(a) Infiltration within a layer (b) Gap between adjacent layers
10-20 μm SiC powder, 1000 °C target temperature parameter,
25 torr TMS + 25 torr H_2, 5 μm/s scan speed, 3 mm scan spacing