SALDVI OF SiC INTO METAL AND CERAMIC POWDERS

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Abstract

Selective Area Laser Deposition Vapor Infiltration (SALDVI) is the SFF technique using gas phase precursors to locally infiltrate a powder bed into a desired shape. Experiments were performed with a CO_2 laser and the silicon carbide forming gas precursor $Si(CH_3)_4$. This paper will report on the microstructural aspects of SiC into a variety of metal and ceramic powders including Mo, SiC, ZrO_2, and WC.

Introduction

SALDVI is a layer-by-layer approach to SFF in which porous layers of loose powders are densified by depositing solid material from gas precursors into the pore spaces using laser chemical vapor infiltration (CVI)[1]. Because the CVI process can deposit a variety of desirable materials in pure form, SALDVI is capable of building ceramic and composite net shapes and shapes with functionally graded compositions such as embedded devices[2] in a single machine. The nanocrystalline nature of the solid material deposited from the $Si(CH_3)_4$ gas precursor by laser CVD on solid ceramic substrates has also been well characterized on the micro and atomic scales[3]. SALDVI using ceramic powders and the SiC-forming gas $Si(CH_3)_4$ has been previously investigated for single line bar geometries with single and multiple layers[4]. The laser induced processing temperature distribution, gas precursor pressure, laser scanning speed, and the particle size of the starting powder were found to affect the densification rate and mechanical properties. In this paper we examine some SALDVI processing issues related to the fabrication of multiple layer rectangular geometries.

Experiment

The SALDVI workstation consists of a vacuum chamber, a powder delivery system, a 50 watt continuous wave CO_2 (10.6 μm wavelength) laser beam, an xy table with scanning mirrors, and an optical pyrometer temperature probe. All components are computer controlled. In these experiments the surface temperature measured by the optical pyrometer is used in a feedback loop to adjust the laser output power as necessary to maintain a constant surface temperature, termed the target temperature, throughout the experiment. The target temperature in this work is 1000°C, the $Si(CH_3)_4$ gas pressure is 10 Torr, and layer thickness is 120 μm. The laser beam spot size was nominally 1 mm in diameter and Gaussian in shape. The rectangular sample has dimensions 10 mm long by 3 mm wide and is obtained by rastering the beam with a scan spacing of 0.75 mm. Figure 1 shows two scan patterns used to generate the rectangular shape, one in which the major scan direction is horizontal (a) and one vertical (b). In order to obtain a more uniform structure, the scan direction is alternated from horizontal to vertical on alternating layers.

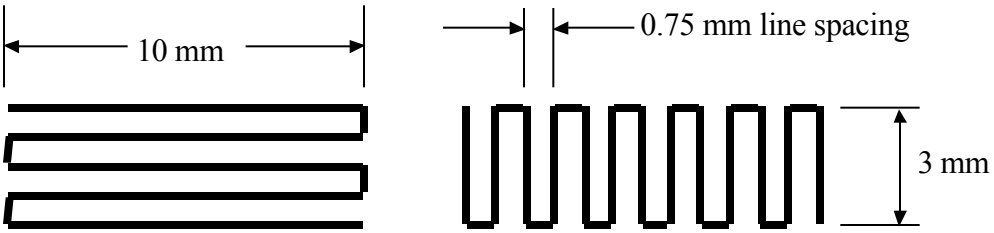


Figure 1. Scan patterns for 10 mm by 3 mm rectangle with 0.75 mm line spacing and (a) horizontal and (b) vertical major scan direction.

Results and Discussion

A. Effect of starting powder

Rectangular samples containing at least four layers were processed by SALDVI using SiC, ZrO_2, Mo, and WC powders and 1000°C target temperature, 10 Torr $Si(CH_3)_4$ gas pressure, and 120 μm layer thickness. Figures 2a-5a show the surface of the samples just after processing, and Figures 2b-5b show a polished cross-section through the thickness direction for SiC, ZrO_2, Mo, and WC powders, respectively. The arrows indicate the location of each cross-section.

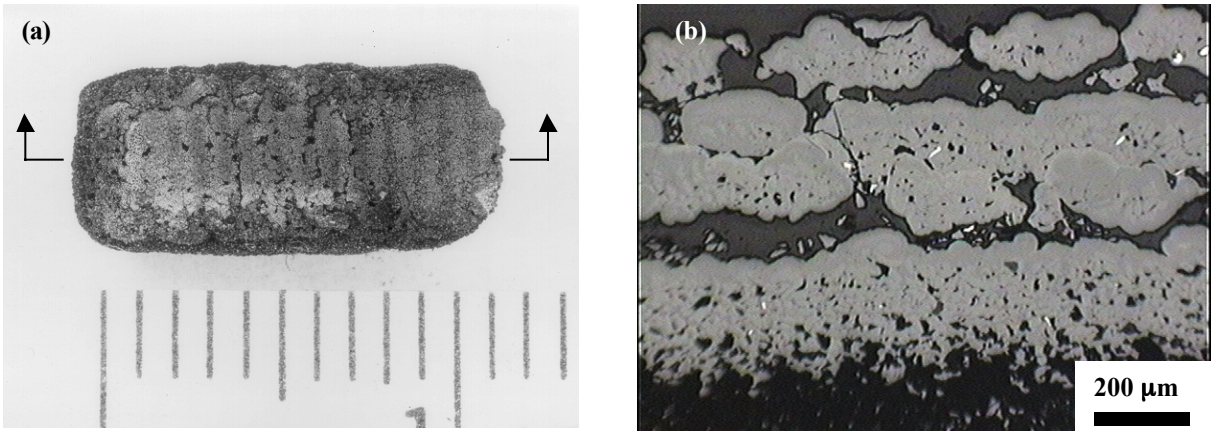


Figure 2. SiC powder/SiC matrix rectangle with 4 layers (a) as-fabricated and (b) cross-section across layers.

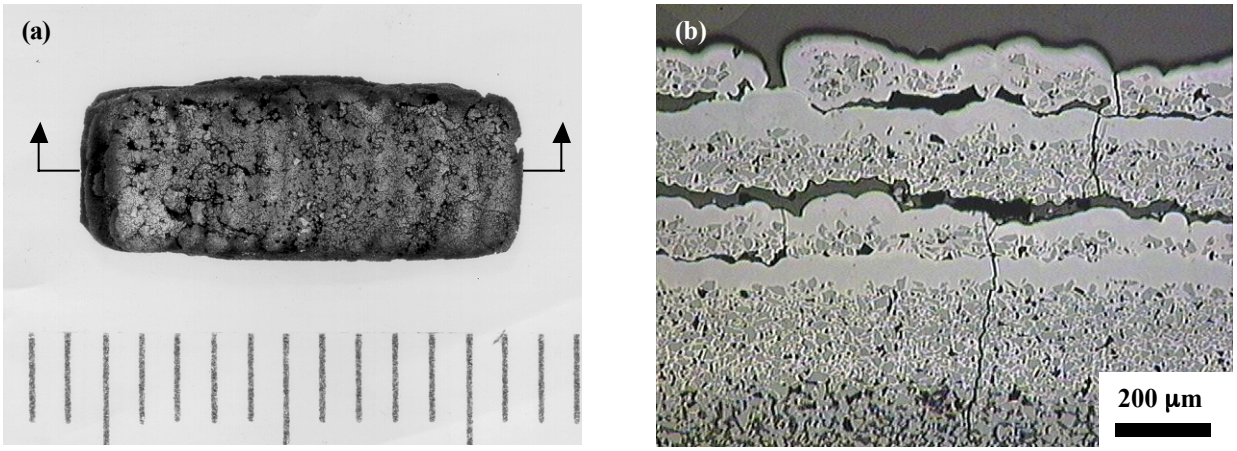


Figure 3. ZrO_2-Y_2O_3 powder/SiC matrix rectangle with 4 layers (a) as-fabricated and (b) cross-section across layers.

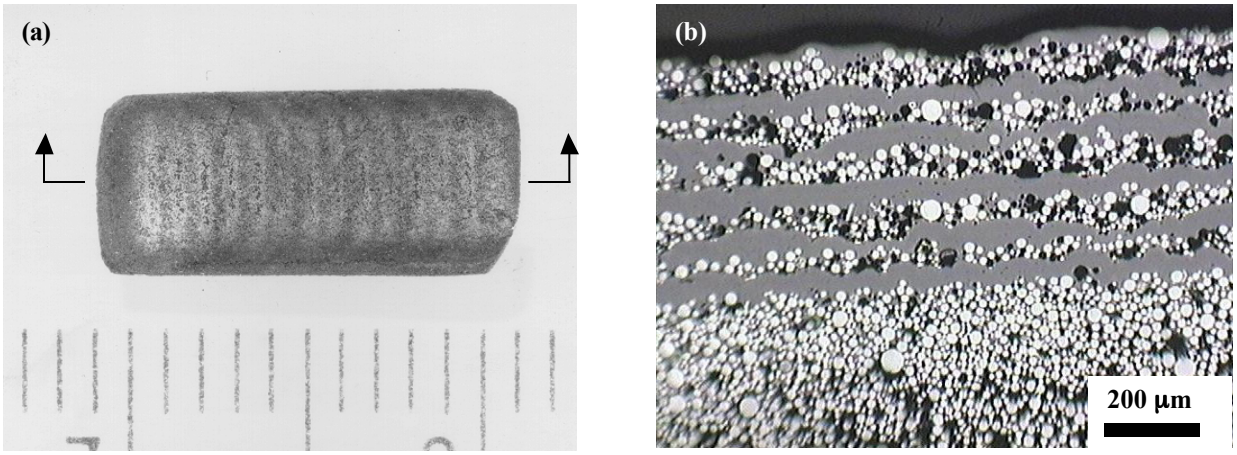


Figure 4. Mo powder/SiC matrix rectangle with 6 layers (a) as-fabricated and (b) cross-section across layers.

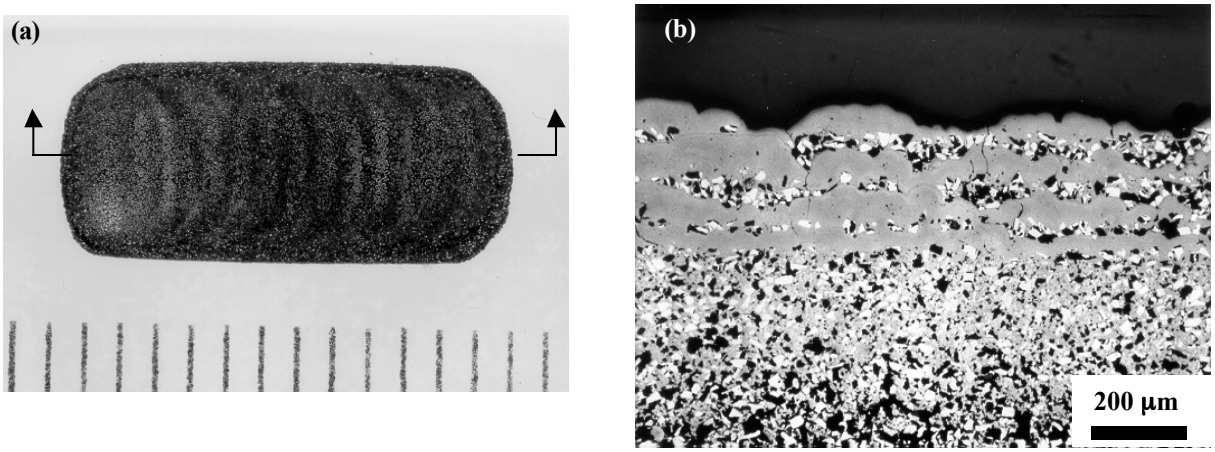


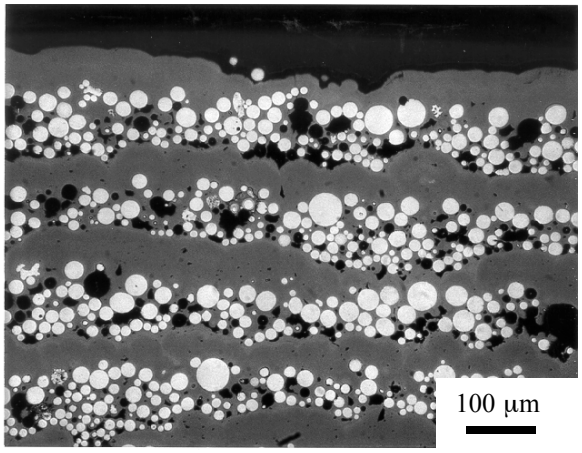
Figure 5. WC powder/SiC matrix rectangle with 4 layers (a) as-fabricated and (b) cross-section across layers.

The SiC powder/SiC matrix and ZrO_2 powder/SiC matrix samples show a rougher as-fabricated surface appearance than the Mo powder/SiC matrix and WC powder/SiC matrix samples. Also there are delaminations between adjacent layers for the SiC and ZrO_2 powder samples while the Mo and WC powder samples have continuous solid material with no delaminations across the layers. The rough surface appearance shown by the SiC and ZrO_2 powder samples appears to be due to a local absence of powder, giving the surface a porous appearance. This absence of solid material is also observed in the cross-sections in the form of gaps between adjacent laser scan lines in the surface layer. These gaps are more pronounced for the SiC powder and less so for the ZrO_2 powder sample. The Mo and WC powder samples show no such gaps at the surface in the cross-sections. We know from in-situ observations of the powder surface during processing that each new powder layer appears smooth and completely covers the previous layer prior to laser heating. It is only after laser heating that the surface gaps appear in the SiC and ZrO_2 powder samples. The reason for the absence of powder in the SiC and ZrO_2 powder samples is likely due to their lower density (3.1 gm/cc for SiC and 5.7 gm/cc for ZrO_2) compared to Mo and WC (10.2 gm/cc for Mo and 15.8 gm/cc for WC). The force to displace the powder likely comes from the gas. Upon the decomposition of the $Si(CH_3)_4$ gas molecule in forming SiC at equilibrium conditions, three methane molecules are released and there is a corresponding increase in local gas pressure. The local gas pressure can also increase due to the local rise in temperature. Assuming the pressure is trapped below the powder, it provides sufficient force to overcome the weight of the loose powder particles and even the weight of the infiltrated layer. This could produce the surface gaps and layer delaminations observed in the SiC and ZrO_2 powder samples. The weight of the Mo and WC powder seems to be sufficient to resist the levitating force of the gas.

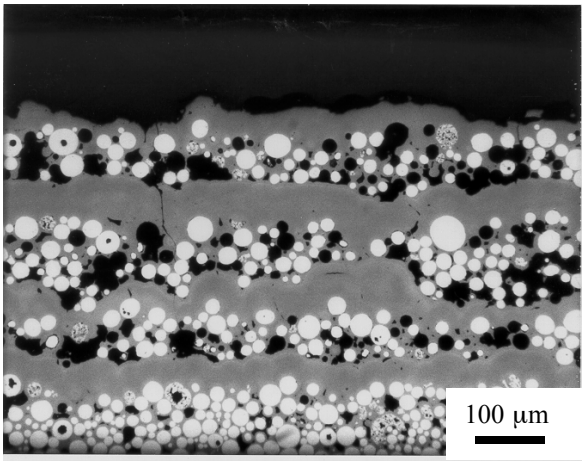
B. Effect of laser scan speed

In SALDVI the amount of local vapor deposition and infiltration depends on the reactant gas concentration and the temperature history, where the temperature history depends on the target temperature and the laser scan speed. It is desirable to achieve parts with both a high

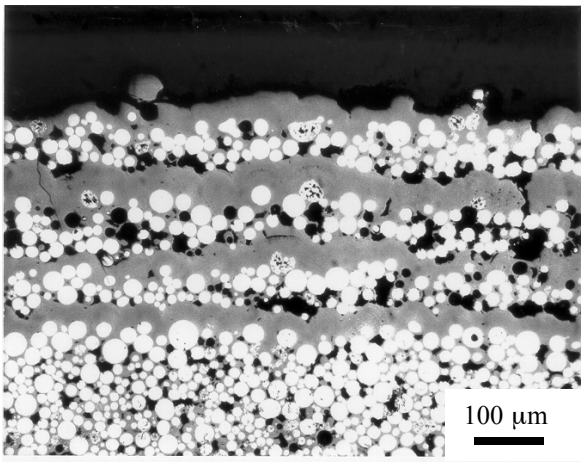
infiltration density and a uniform structure. The Mo powder/SiC matrix sample in Figure 4b shows a non-uniform structure. In addition to the vapor infiltrated powder, each layer also contains a layer of pure vapor deposited SiC at the surface of each powder layer. Here the scan speed is increased to reduce the time at temperature and examine the effect on reducing the thickness of the purely vapor deposited layer at the surface of each powder layer. Figures 6a-e show cross-sections of Mo powder/SiC matrix rectangle samples with four layers processed at 1000°C target temperature, 10 Torr $Si(CH_3)_4$ gas pressure, 120 μm layer thickness, and with scan speeds of 2.5, 3.8, 5.0, 7.5, and 10 μm/s, respectively. As the scan speed increases, the thickness of this vapor deposited layer decreases, from about 75 μm thick for a scan speed of 2.5 μm/s to about 10 μm thick for a scan speed of 10 μm/s. However, at some point decreasing the heating time will also cause the density in the infiltrated powder to start to decrease. The infiltration density begins to decrease when the scan speed increases to 10 μm/s. So there is a compromise between obtaining parts with high infiltration density and uniform distribution of vapor infiltrated powder.



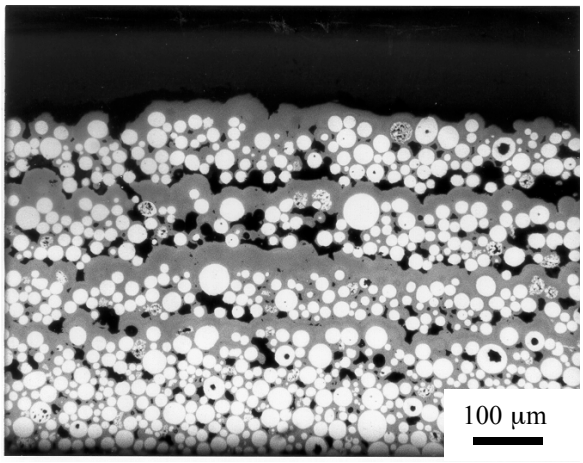
(a) 2.5 μm/s scan speed



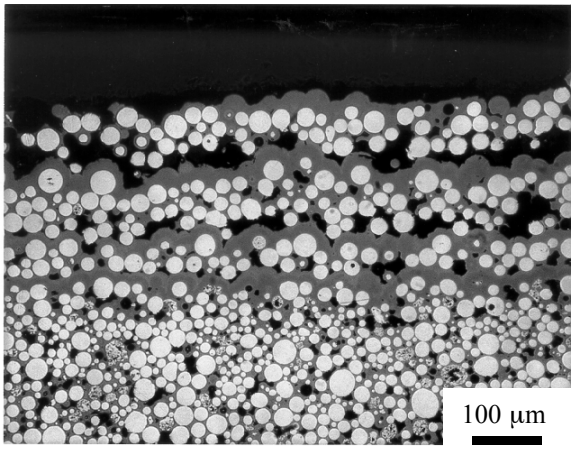
(b) 3.8 μm/s scan speed



(c) 5.0 μm/s scan speed



(d) 7.5 μm/s scan speed



(e) 10 μm/s scan speed

Figure 6. Mo powder/SiC matrix rectangles with 4 layers.

Conclusions

The type of powder was found to affect the surface appearance and internal structure of SiC matrix multiple layer SALDVI rectangles. Samples with SiC and ZrO_2 powder show a porous surface appearance due to displacement of the powder during processing. Mo and WC powder samples have a dense surface and continuous solid material across adjacent layers. The lower density of the SiC and ZrO_2 powders and convective effects in the gas are the likely cause of their poor structure.

The laser scan speed affects the distribution of vapor deposited SiC in Mo powder/SiC matrix multiple layer SALDVI rectangles. Higher scan speeds increase the uniformity of the microstructure of each layer by reducing the amount of pure vapor deposition that occurs at the surface of the powder layer. However, at higher scan speeds the infiltration density in the interior of the powder layer begins to decrease.

Acknowledgement

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References

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