Ceramic Joining by Gas Phase Pulsed Laser Processing

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Abstract

The method of Selective Area Laser Deposition (SALD) and Vapor Infiltration (SALDVI) has been successfully used to fabricate small three-dimensional SiC/SiC and SiC/metal powder parts. Ceramic joints made by this technique have been limited by the throwing power of the laser resulting in incomplete joint penetration. Studies were performed to show the effectiveness of a fiber laser, with a wavelength of 1070 nm, for a joining process. The ability of the laser to penetrate a powder bed was utilized in the joint fabrication. The combination of powder fill, and deep laser penetration into the powder bed shows potential in the field of ceramic joining.

Introduction

Ceramic joining of components can be done by several methods. Green pieces can be joined ultrasonically before firing, frictional joining or the use of brazing materials can be used if the components are already fabricated. These methods involve special conditions that must be considered before component design. Another method that has been studied is the use of a focused laser in a reactive gas environment to selectively heat a substrate and decompose the ceramic precursor and form a joint.[3,6-9] This process, known as selective area laser deposition (SALD), can be performed without bulk heating of the components and without the use of materials that will be ineffective at higher temperatures. The SALD has been used with some success to join components with a rotational symmetry.[7] A variant of the SALD process that involves the infiltration of a substrate powder with precursor gas during decomposition is known as the SALDVI process where the VI indicates vapor infiltration. This technique has been used for free form fabrication of arbitrarily shaped components. The powder that is to be infiltrated can be the same material as the decomposed species or a different material making a composite. Several types of ceramics have been fabricated with the SALD and SALDVI process including Si_3N_4, graphite and composite materials utilizing different metallic and ceramic powder reinforcements. This study utilizes Tetramethylsilane ($Si(CH_3)_4$) pyrolytic decomposition in the presence of hydrogen in the overall process reaction:

$Si(CH_3)_4(g) \Rightarrow SiC + 3CH_4(g)$

Alternative precursors to form SiC include methyltrichlorosilane (MTS , $SiCH_3Cl_3$), and combination of silane (SiH_4) and methane (CH_4). [5,9-13]

Experimental Procedure

Previous joining work has concentrated on the use of deposition without the use of a powder to infiltrate. This study combines the use of SALDVI with the previously established joining techniques. A 40-watt average power pulsed fiber laser with 1.5 mJ per pulse (SPI Lasers) was used for the studies. A single focusing lens was used with a focused spot of 50 microns. The beam was delivered off of a series of mirrors on an XY motion stage controlled by a Labview motion program and a National Instruments control card. The deposition chamber is a vacuum chamber flushed with argon and evacuated to 80 mtorr and then filled with desired gas proportions. The starting gas operating pressure for all experiments was 50 torr TMS and 50 torr

H_2,below the vapor pressure of TMS, to prevent condensation within the chamber. Semi Infinite powder bed deposition was used to evaluate deposition conditions using a 7 micron average size SiC powder. Laser powers of 20 and 38 watts average were used with laser scan speeds of 100, 200, and 400 microns/s. The higher power at all speeds ejected considerable powder from the point of focus leaving trenches in the powder with deposits into the trench walls. With the lower power the most consistent deposit was produced at 100 micron/s. To determine the spacing to be used for the joint deposition raster pattern, powder bed deposits were made with decreasing line spacing from 1800 microns to 80 microns. A powder bed raster deposit was made 1.5 mm wide with a scan line spacing of 85 microns and a total length of 6 mm and a scan speed of 100 micron/s at 20 watts average power.

The joint configuration explored was an abutting of two beveled edges with a powder fill of 7 micron SiC powder, packed into the joint cavity by ultrasonic vibration. A joint component thickness was chosen so that the depth of penetration of the laser into a free powder bed was equivalent to the SiC plate. A parallel rectangular raster pattern was chosen with a line spacing of 130 microns in the travel direction. The deposited width of the raster pattern was designed as 130% of the largest part of the joint gap. The sample geometry used in this study is indicated below, Figure 1.

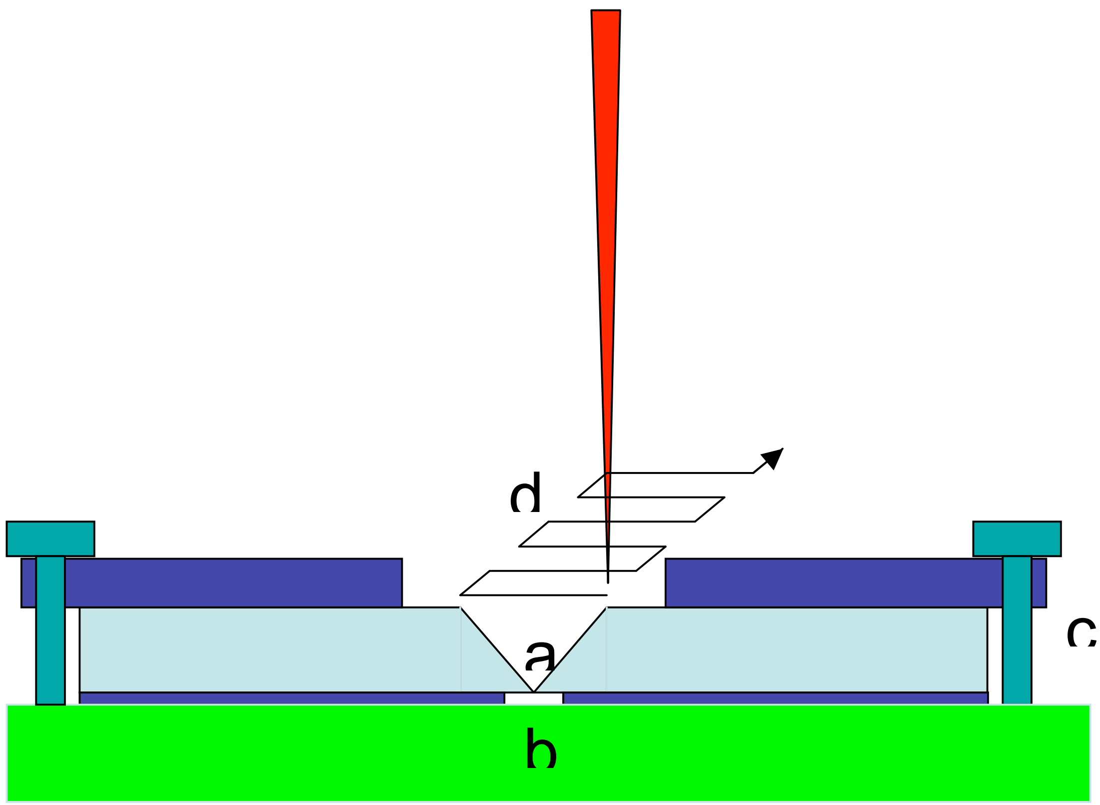
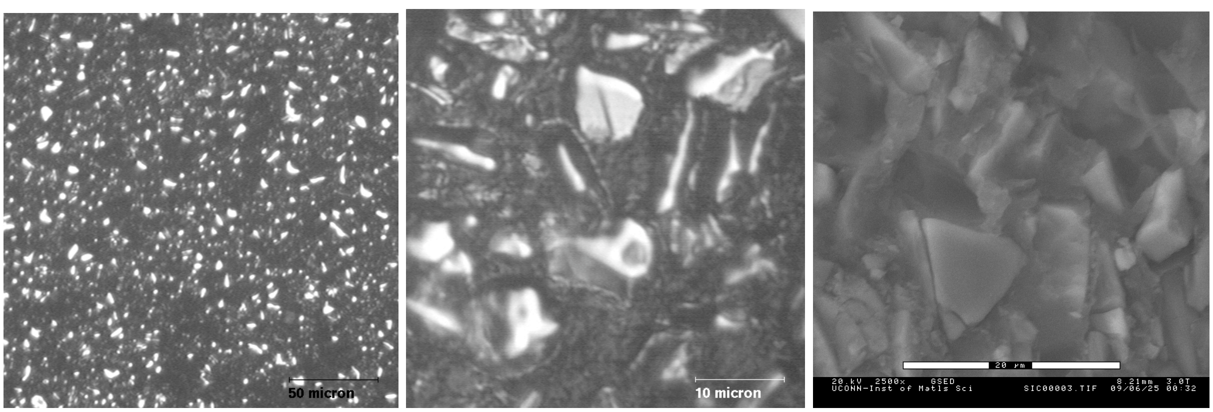


Figure 1:
Schematic of sample configuration: a. Beveled SiC plates 1270 micron thick with standardized 20° bevels. b. Diffusion gap created by spacers to allow diffusion of reactant as well as a measure so the joint would not adhere to the sample mount. c. Mounting clamp that secures each beveled section. d. Rectangular raster beam path.

The two beveled SiC plates were abutted and clamped onto a sample stage. The samples were separated from the sample stage by thin sheet steel spacers to prevent any adhesion to the stage and to allow for possible diffusion of the reactant species to the non-beveled side of the joint. Two variations of the joint were evaluated, both were formed with a 50 micron spot size scanned raster with a 20 watt average power with a starting TMS pressure of 50 torr, H_2 pressure of 50 torr, and a scan speed of 100 micron/s. The first joint fabrication type was performed with no joint surface preparation. The second joint was formed by pre ablating the faces of the joint components without the presence of powder or reactive gas in order to produce a micro textured surface and then filling the joint cavity with powder and performing the SALDVI process as had been performed in the joint type 1. Analysis of the deposited material was performed with the use of visible light microscopy, environmental scanning electron microscopy as well as X-ray diffraction.

Results and Discussion

The decomposition of TMS in this study has two general microstructural types. The SiC formed by the infiltration of the powder and the SiC that has formed either by displacing powder or by decomposing on already infiltrated powder. The powder infiltration deposit type is seen below, Figure 2.



a b c

Figure 2: The decomposition product has surrounded the SiC grains with a somewhat dense deposit. a. Infiltrated powder at 25X visible light magnification taken from a progressive line spacing scan in a region of uniform deposition. b. a 125X visible light image taken from an infiltrated region of a joint sample. c. an ESEM image of a powder bed scanned deposit at 2500X magnification

When infiltration paths into the powder bed are closed by the growth of the SiC deposit, a condition arises that allows SALD to occur, deposition without infiltration, Figure 3.

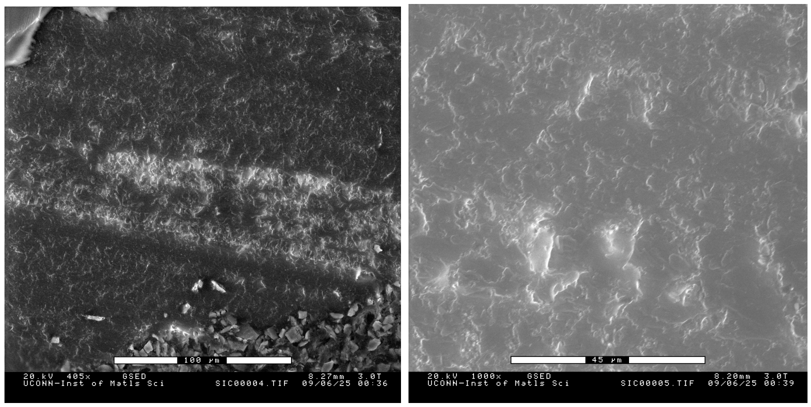


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Figure 3:
These images are representative of the decompositions that occur without powder infiltration. a. an ESEM image at a magnification of 405X of the deposit above the powder bed. Image b is a higher magnification, 1000X, of the same region above the powder. Reasonable densities are observed.

To determine the optimal spacing for the parallel lines of the raster pattern a deposition was performed with variable line spacing. The factors considered for optimal spacing were: uniformity of the deposit in the vicinity of a scanned line, the extent to which an adjacent line was resolvable and the presence of beam damage. Light microscopy was used to evaluate the beam spacing, Figure 4.

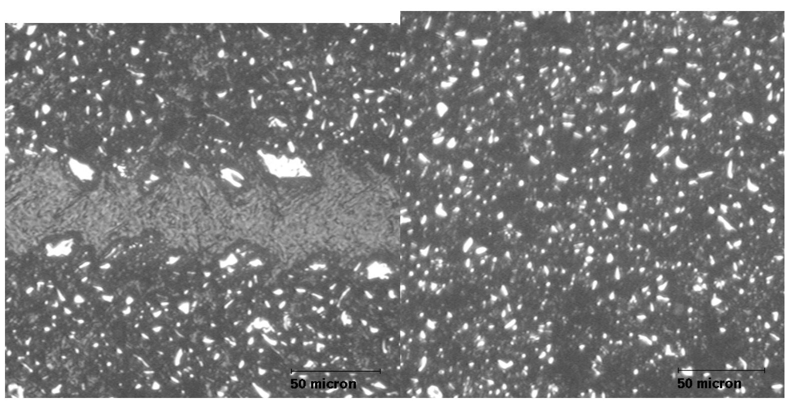

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Figure 4: The proximity of one line to the next in the powder bed deposition, affects the quality of the deposit. a. the central line is epoxy material which has filled the space left by the laser powder displacement and this micrograph represents the most widely spaced scan width (1.8mm). b. an intermediary scan spacing (300 microns).

The spacings of the raster lines that produced the most uniform deposits were found to be in the range of 300-80 micron. Deposition into a powder bed with a raster pattern with line spacing of 85 microns was performed on a semi-infinite powder bed. Resulting in a cohesive unit of good uniformity and reasonable density

Deposition for the purpose of joint formation was performed. The goal of the joining studies is the formation of a strong and minimally porous joint with a full penetration to the base of the joint, with good cohesion to the joint wall. Two joining techniques were attempted. Joint type 1 is characterized by flat joint wall surface prepared by diamond grinding (to 220 grit). Joint type 2 was laser scribed by the fiber laser in order to provide a surface roughness. Previous joining attempts were met with greater success when the joining surfaces showed some porosity.[8]

The porosity of each joint was qualitatively evaluated by the ESEM. The initial particle size for the substrate powder is less than 7 microns. The structure of the deposition indicates the growth of SiC surrounding the particles, Figure 5.

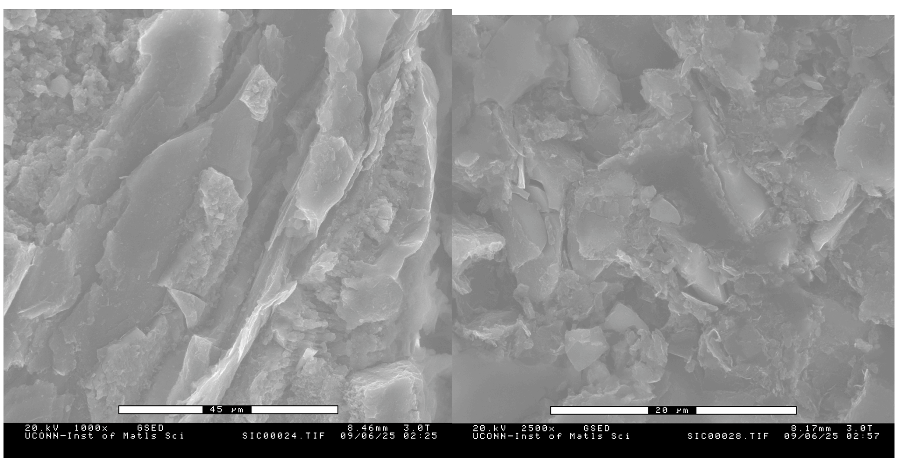

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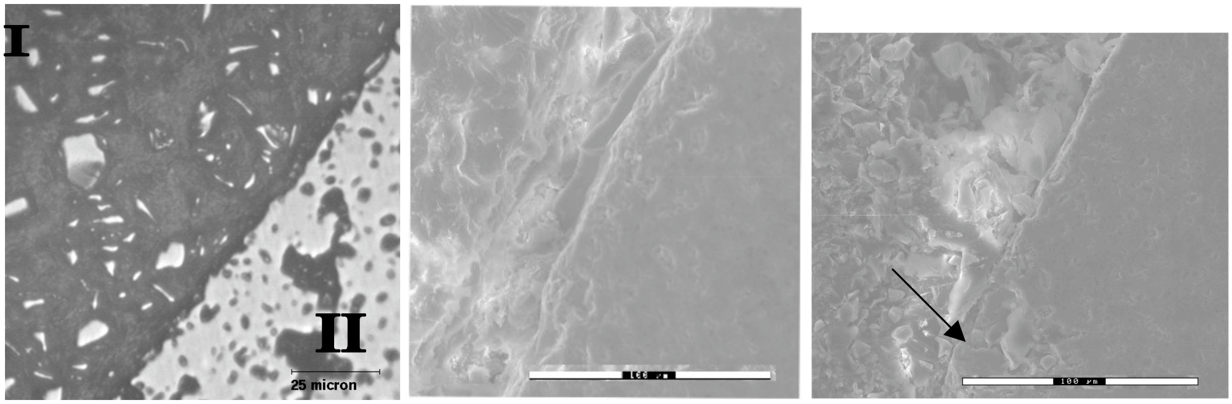
Figure 5
a. Deposited joint material with a 1000 X magnification that displays a region of partially reacted powder in the upper left next to a larger region of deposited material. It can be seen that deposition was occurring with some irregularly in this sample, joint type 1. Image b taken from a sample of joint type 2 at a magnification of 2500 X displays a more uniform deposition with no regions of uncoated powder. Both images display reasonable density.

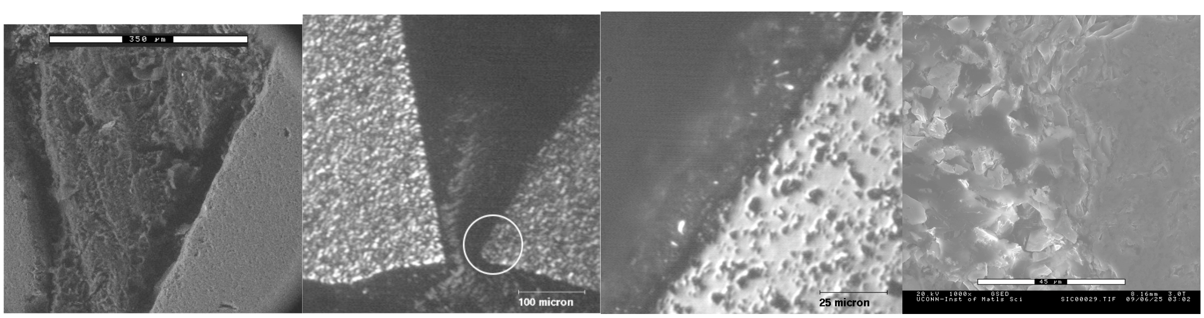
The use of a high quality beam source, in the form of a fiber laser, and a focusing optic with a 50-micron spot size, has allowed a deep penetration into the joint cavity. Although both joints experienced a de-cohesion as a result of moments applied during the sample removal, the joint type 2 shows evidence of deep joint penetration. Although fracture occurred along one deposition edge, the un-fractured edge shows little porosity at the boundary, Figure 6.



a b c

Figure 6
a. A polished sample taken from a joint type 1 on an optical microscope with a magnification of 50X that shows a distinct and non porous boundary between the deposit material, I, and the joint wall/substrate, II. ESEM images of Joints type 1 (b.), and Joint type 2 (c.) where both show the same type of adhesion at a magnification of 500X where the left side of each interface is the deposit. c. shows a region, identified by an arrow, where the deposition material penetrated a micro ablation present from the laser pretreatment.

The beam penetration is evident from the deposited material in the presence of the crack tip. The joint type 1 joint did not have full penetration to the joint tip. There is evidence of deposited material at the tip of the second joint type, Figure 7.



a b c d

Figure 7
a. Micrograph of the type 1 joint indicating partial penetration before joint fracture, image at a magnification of 125 X(backscattered ESEM). b. Optical micrograph (12.5 X magnification) of the type 2 joint penetration, the deposited material (black) is adhered to the joint tip. The grey material to the left of the circled region is infiltrated epoxy into the fracture zone. Image c is an optical magnification of the same region at 50X magnification. Image d is an ESEM image with a magnification of 1000X. Image d also shows evidence of the laser pre ablation in the substrate tip, right.

Deposits were confirmed to be silicon carbide through the use of powder x-ray diffraction. The diffraction pattern standard for synthetic hexagonal silicon carbide corresponds closely to the experimental pattern as seen below, Figure 8.

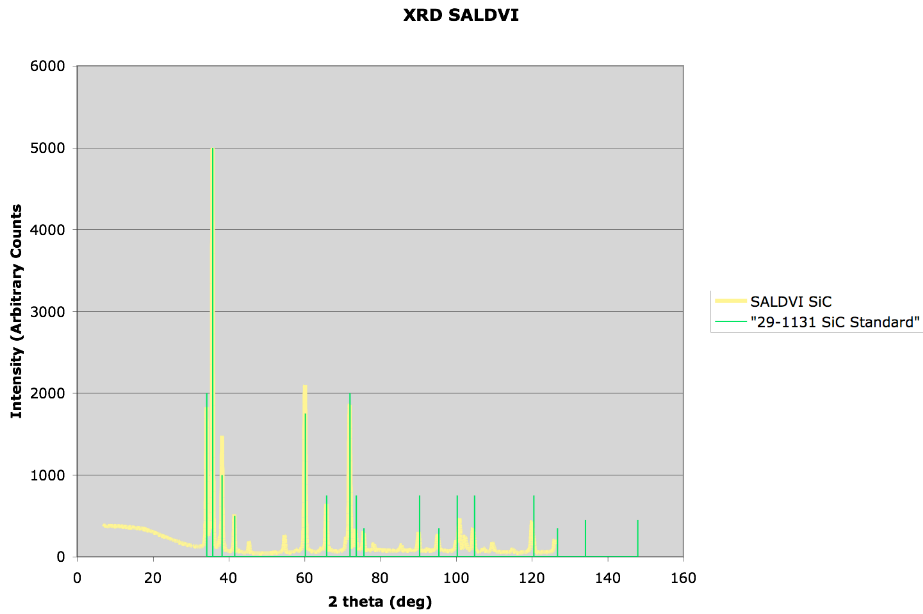


Figure 8
X-Ray Diffraction pattern for the composite joint material compared with a standard for hexagonal SiC.

Conclusion

The novel technologies employed in this study include the use of a high quality fiber laser generated beam focused to 50 micron, the use of powder material filler phase within the joint, and the use of the laser as a means of ablating the surface of the joint before the infiltration process. The powder bed infiltration tests indicate that the beam is capable of penetrating the powder and decomposing the precursor to a uniform depth. If the sample joint is kept to a thickness below that of the penetration depth a SALDVI type joint is possible. Thicker samples would necessitate a multi layer approach. The fiber beam shows promise for future joining studies but a balance must be reached between the desired penetration depth of the beam and the tendency for the beam to displace the powder from the hot zone. The surface pre ablation technique employed here was detectable under microscopic examination. There was deposited material within the ablation tracks. The ablation pattern was made under potentially oxidizing conditions, which could have been a factor in joint de-cohesion. Ongoing work involves the use of an ablation pattern applied in a vacuum before powder infiltration. Additionally a multi layer powder infiltration joining technique is being explored.

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