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**Manufacturing of Carbon Fiber Reinforced Silicon Carbide -  
Zirconium Diboride Composite Brake Rotors using Electric Field  
Assisted Sintering**

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**ABSTRACT**

*High performance fiber reinforced ceramic rotors have the potential to greatly improve metrics in heavy vehicles such as braking distance, acceleration time, maximum speed, fuel consumption, improved handling, and increased vehicle maximum loads. Three types of carbon ceramic composite brake rotor materials were created using polymer infiltration pyrolysis (PIP) for carbon fiber reinforced silicon oxycarbide, reactive melt infiltration (RMI) for carbon fiber reinforced silicon carbide, and electric field assisted sintering (EFAS) for carbon fiber reinforced silicon carbide-zirconium diboride to investigate the manufacturing of 396mm diameter heavy vehicle brake rotors. The microstructure of parts created by each manufacturing method were discussed and contrasted. The EFAS manufactured rotor created the highest quality part due to extremely fast processing times, uniform material microstructure, and fusing of adjacent fibers in the carbon fiber network. Thermal conductivity was measured to be greater than that of a traditional steel rotor.*

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**1. BACKGROUND**

Carbon ceramic brake discs, which are made primarily of carbon fiber reinforced silicon carbide, have been around since 2001 when they debuted on the Porsche 911 GT2 as a major performance upgrade. The brakes not only lasted 3-4 times longer than steel brakes, but they also were 2-3 times lighter.

Less rotational mass improved acceleration, deceleration, and overall turning performances of the vehicle, which are highly sought after in performance vehicles. Since then, carbon ceramic brake discs have been a staple in the racing performance industry as a lightweight brake good enough for weekend racing and exceptional street performance.

Other vehicle applications have been explored for various vehicles that could benefit from carbon ceramic brakes but they all had two problems in common. Carbon ceramic brake discs are several times the cost of your average cast iron brake rotor and you must heavily modify your entire brake system and nearly the size of your wheel to accommodate them. This makes installing carbon ceramic brakes even more expensive than just the rotor cost itself, ultimately limiting its application use to exotic sports cars where consumers are willing to pay the premium for added performance.

Industries that would benefit include commercial and defense vehicles. For example, freeing up weight on a commercial vehicle could save up to 600 pounds in freight revenue per day. That's equal to roughly \$200,000 in additional freight revenue annually. In the defense sector, a tactical vehicle can improve the survivability of a soldier stopping before driving over an IED or accelerating faster to get out of enemy territory without losing control.

In this technical paper, Carbon SiC Technologies has developed a unique material for carbon ceramic brake discs to address these issues and have compared to existing market materials.

Silicon carbide (SiC) ceramics have been used for decades in the ceramics industry due to their low density, thermal shock stability, high creep resistance, high temperature strength, high oxidation resistance, and high hardness/modulus [1, 2]. Monolithic SiC is one of many covalently bonded, very hard

ceramics, alongside materials such as boron carbide ( $B_4C$ ), boron nitride (BN), and diamond, with the primary limitation of having low fracture toughness. Unlike ionic and metallic bonds, covalent bonding is directional and as a result the atomic structure of covalent ceramics are symmetrically arranged creating a highly ordered structure. This gives them very high hardness but also creates a low fracture toughness condition as cracks can easily propagate down certain crystallographic planes.

Mitigation of this low fracture tolerance has been investigated by many through the inclusion of a fiber matrix running through the ceramic material. These materials are often referred to as ceramic matrix composites (CMC's) or fiber reinforced composites. The fibers themselves can be any material but carbon fiber is the most common due to its low cost and ability to maintain its mechanical properties at temperatures more than  $2100^{\circ}C$  [3-6]. The inclusion of carbon fiber into a ceramic has the primary purpose of preventing brittle fracture and subcritical crack growth through mechanisms like fiber pull out and crack bridging [7].

From a material processing perspective, the high energy covalent bonding that provides materials like SiC their desirable properties also can make them difficult to consolidate/sinter in their pure form. Extremely high temperatures and the addition of sintering aids are often required which can sometimes reduce the performance of the original material. When it comes to carbon fiber reinforced silicon carbide composites (Cf/SiC) the process is often lengthy and expensive. Conventionally, production of Cf/SiC composites occurs via a combination of hot isostatic press (HIP), hot press (HP), or pressure assisted microwave sintering and chemical vapor infiltration (CVI), polymer impregnation and pyrolysis (PIP), or melt

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infiltration (MI). There are benefits and drawbacks to each however all their pathways require long processing times and costs too much except for only the highest financial margin of uses [8-12]. In addition, such methods are challenging to process borides which belong to a group of ceramics classified as ultra-high temperature ceramics (UHTC). Popular boride UHTC's include zirconium diboride (ZrB<sub>2</sub>), titanium diboride (TiB<sub>2</sub>), hafnium diboride (HfB<sub>2</sub>) and several others. Borides are very promising due to their material properties having high melting points above 3,000 C, high thermal conductivities, and high electrical conductivities making them excellent candidates for the next generation composite matrices.

Regardless, SiC is still highly sought after as a light weight, extremely hard, thermal management material in applications that span UHTC areas such as rocketry, hypersonic vehicles, and other aerospace uses where maintaining mechanical stability at high temperature and wear rates is mandatory. These traits are also critical in the automotive industry, specifically with brake rotors, which must be able to handle enormous amounts of frictional induced forces along with simultaneously generated heat.

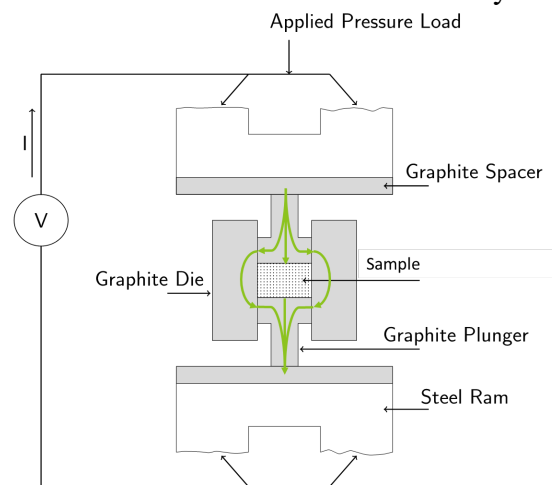
In this work a 16" diameter brake rotor comprised of a proprietary carbon-fiber reinforced SiC-ZrB<sub>2</sub> mixture was created using the advanced manufacturing technique electric field assisted sintering (EFAS), also known as spark plasma sintering (SPS). EFAS is a unique heating technique where material, often powders, are densified through uniaxial pressure and direct heating by passing electrical current through sample molds. Mold and sample electrical resistance causes self-heating through Joule/resistive heating. The change in temperature is a

function of the current density at any point in mold/sample ensemble along with their associated material properties and generally follows Equation (1) where  $I$  = current [A],  $\rho$  = material density [kg/m<sup>3</sup>],  $L$  = length of conductor [m],  $A$  = cross-sectional area [m<sup>2</sup>],  $c_p$  = heat capacity [J/(kg·K)],  $m$  = total mass [kg],  $t$  = time [sec], and  $T_0$  = initial temperature [K].

$$\Delta T = \frac{I^2 \rho L t}{A c_p m} \quad (1)$$

This self-heating behavior makes it extremely efficient at transferring energy into the sample body and when combined with the application of uniaxial pressure provides a highly effective powder consolidation technique. EFAS processing times are regularly a fraction of most other more traditional methods such as free sintering in furnaces or hot pressing. A schematic of the process is shown in Figure 1.

Carbon fiber reinforcements were added to the powder matrix as a toughening mechanism to improve the fracture toughness of the ceramic as well as increase the thermal conductivity of the rotor. Thermal management is a critical component to any braking system as the heat generated can cause reduced performance, warping, and even failure of the entire brake assembly.



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**Figure 1:** Schematic of EFAS configuration with rams, tooling, and sample with current flow shown by green arrows.

*The goal of the C/SiC-ZrB<sub>2</sub> brake rotor developed is to provide an alternative to the current state of the art metal alloy rotors being use on heavy ground transport vehicles. Through improved performance, weight reduction, and durability metrics such as acceleration, deceleration, fuel consumption, part lifetime, and vehicle handling can all be significantly enhanced.*

## METHODS

Commercially available ZrB<sub>2</sub>, SiC powders and short carbon fibers were used as starting materials. The raw materials were weighed based on predetermined values and mixed using a Turbula Mixer for 20-40 minutes.

The sample was consolidated at Idaho National laboratory in Idaho Falls, ID with a DCS-800 furnace system (Thermal Technology, LLC, Minden, NV). The DCS-800 is a direct current sintering system capable of applying 800 tons of uniaxial pressure and 150,000 amps of electrical current across a 24 inch diameter ram. An image of the DCS-800 is shown in Figure 2. The part was processed at approximately 15 to 55 MPa of pressure and between to 1700 °C and 2100 °C.



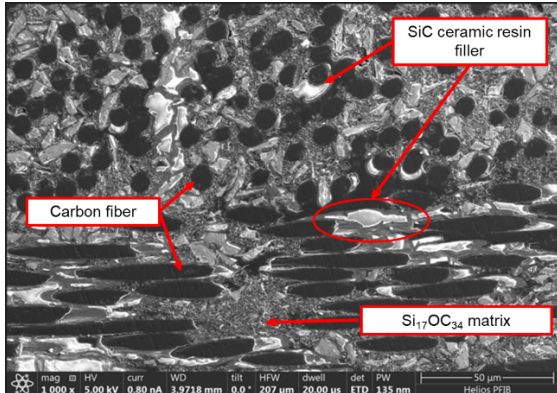
**Figure 2:** Overview image of DCS-800 at Idaho National Laboratory.

The tooling used for processing the part was made from purified, high density, isostatic graphite and custom designed by Carbon SiC Technologies to achieve the desired thickness and net dimensions required of the rotor. Graphite foil was used throughout the mold assembly to seal the various parts and provide good electrical contact between mold pieces.

Surface grinding and polishing of each face as well as machining of the inner and outer diameters was performed after extraction from the tooling. Density was measured using Archimedes measurements in water following ASTM C20-00. Characterization of the Cf/SiC-ZrB<sub>2</sub> material was performed by optical microscopy using a Keyence VHX-5000 Series Digital Microscope and scanning electron microscopy (SEM) coupled with energy dispersive x-ray spectroscopy (EDX) using Nova NanoSEM 450 Field Emission Scanning Electron Microscope. Thermal conductivity measurements were taken using a ThermTest Transient Plane Source (ISO/DIS 22007-2.2).

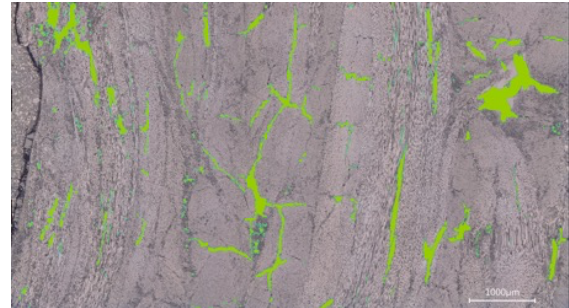
## RESULTS AND DISCUSSION

SiC composite brake rotor materials were produced by three different methods. Initially, the PIP method was attempted with a commercially available infiltration polymer. PIP is a process that begins by infiltrating a ceramic fiber preform with a preceramic polymer (polycarbosilane). That polymer matrix composite is pyrolytically decomposed at elevated temperatures in argon. The leaves a porous structure in the newly created ceramic matrix so then the part is infiltrated and pyrolyzed again. This iterative process can occur up to 10 times before the target part density is high enough.



**Figure 3:** SEM micrograph of a Cf/SiOC brake rotor manufactured via the PIP method. The image shows carbon fiber, carbon, silicon oxycarbide, residual filler/resin, and porosity

A representative cross section of the rotor microstructure is shown in Figure 3. The structure of the carbon fiber silicon oxycarbide (Cf/SiOC) material showed good distribution of matrix and fibers however there were areas of residual unpyrolyzed resin. This is not desirable as it weakens the strength of the ceramic with unreacted polymers that have not converted to the hardened ceramic matrix. A fair amount of porosity remained in the part as the percent relative density was 92.5% with a measured density of 2.05 g/cm<sup>3</sup>. Porosity is a problem not only because it weakens the overall part but it provides pathways for oxygen to migrate into the part and during operation when heat is generated the carbon fibers can readily oxidize, significantly weakening their tensile strength. High surface porosity also accelerated faster wear on brake pads in an SAE J2928 thermal crack test performed on the rotor using a brake dynamometer. As the brake pads heated up, the porosity on the surface had a “cheese grater” effect on the pads wearing them down faster than a competing OEM C/SiC brake rotor.



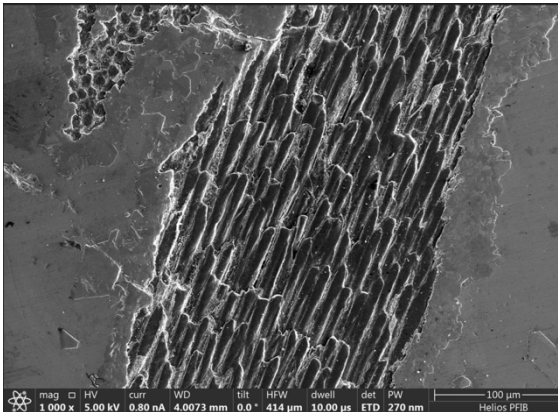
**Figure 4:** SEM micrograph of Cf/SiOC brake rotor with porosity colored in green.

The number of infiltration and pyrolyzation cycles required for this rotor was seven which made it expensive and time consuming to produce. For those reasons, suboptimal microstructure and elevated processing time and cost reactive melt infiltration (RMI) was investigated for SiC rotor manufacturing.

RMI is very different from PIP in that a carbon fiber reinforced plastic (phenol formaldehyde resin) containing preform is carbonized at 1,100 °C for up to 30 hours typically in a retort furnace creating a porous carbon body. The goal is burn off and carbonize the plastic. It is then infiltrated with molten silicon and heated in a vacuum furnace at around 1,700 °C in argon gas. In RMI, as silicon melts at 1,400 °C the vacuum in the chamber assists in helping liquid silicon penetrate the porous carbon preform like a sponge and reacts with carbon in the preform to create SiC. The matrix then is made of silicon carbide and, very often, unreacted silicon due to the inconsistencies in randomization of the carbonization process.

Figure 5 shows a bundle of carbon fiber surrounded by reacted SiC and residual silicon in the PIP manufactured C/SiC rotor. Residual silicon is sometimes not an issue however for brake rotor applications the extreme heat generated is a problem for the areas of residual silicon since silicon has a tendency to oxidize around 700 °C. This can significantly impact the performance of the

brake rotor. Temperatures experienced in heavy vehicles can exceed 900 to a 1,000 °C in extreme conditions often seen on the battlefield or as simple as a class 8 semi-truck using heavy braking down a steep hill. This would cause premature oxidation of regions of the rotor leading to catastrophic failure of the part. Porosity was also measured to be at approximately 4%.



**Figure 5:** SEM micrograph of a Cf/SiC brake rotor manufactured using RMI.

A final process was investigated, creation of a C/SiC-ZrB<sub>2</sub> brake rotor heated in a unique and novel heating method called EFAS or SPS. The goal was to have no residual silicon, lower cost in manufacturing, low porosity, and higher thermal conductivity (for easier replacement and brake pad compatibility of swapping steel rotors out for C/SiC-ZrB<sub>2</sub> rotors) in the final part. To accomplish this a mixture of turbula mixed chopped short carbon fiber (30-45%), SiC powder (35-45%), ZrB<sub>2</sub> powder (10-20%), liquid polymer (5%), and 1-5% proprietary additive was created. The microstructure of the matrix without the inclusion of carbon fiber can be seen in Figure 8. This new composition when consolidated via EFAS at 1700 °C - 2100 °C exhibited substantially lower porosity, only 0.3-1.2% depending on mixture composition and processing condition, and a well

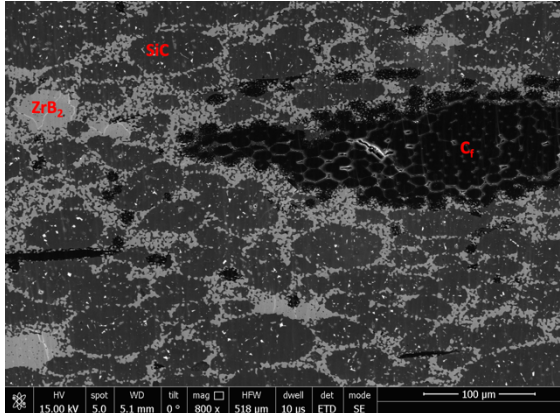
distributed microstructure when compared to similar rotors manufactured via PIP and RMI methods. Furthermore, no residual silicon was found anywhere in the part.

**Table 1:** Temperature dependent thermal conductivity measurements of Carbon SiC Technologies C/SiC rotor.

Temperature (°C)	Average Thermal Conductivity (W/m*K)
20	96.63 ± 0.11
100	94.96 ± 0.48
300	93.79 ± 0.84

ZrB<sub>2</sub> was also specifically added to the SiC matrix, along with the chopped carbon fiber, to raise the part’s thermal conductivity to compete with that of a steel brake rotor. With carbon fiber-carbon (Cf/C) measuring around ≈20 W/m\*K, carbon fiber-SiC (Cf/SiC) measuring at ≈40 W/m\*K, and steel measuring at ≈54 W/m\*K, C/SiC-ZrB<sub>2</sub> demonstrates a very promising thermal conductivity of greater than 90 W/m\*K, see Table 1. This is extremely promising for managing the heat generated from the immense friction loads seen on heavy vehicles. The lower thermal conductivity of traditional Cf/SiC brake rotors requires them to be much larger diameters to dissipate heat as well as requiring specialized brake pads that can handle the elevated heat generated. Leveraging high thermal conductivity materials in braking systems protects the brake pads due to the enhanced heat dissipation that would otherwise warp the pad when becoming soft at high temperatures. Brake pad warping decreases the ability for the braking system to function and creates a positive feedback loop of decreased contact area between the rotor and pad thereby creating areas of increased heat load further warping the pad. This process continues to grow until the brake system fails completely. Therefore, a Cf/SiC-ZrB<sub>2</sub> with nearly double the thermal conductivity of

steel means they can be swapped into current braking systems without modifying other vehicle brake components. This would normally be required with a traditional C/SiC rotor.



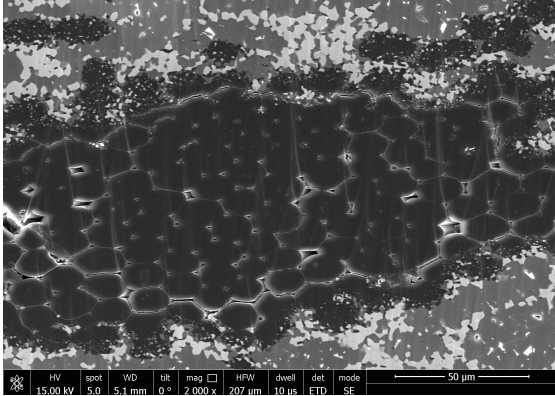
**Figure 6:** SEM micrograph of a Cf/SiC-ZrB<sub>2</sub> brake rotor manufactured via EFAS. The image shows well dispersed areas of Carbon Fiber, SiC, and ZrB<sub>2</sub> that were created through side reactions.

Through utilization of the DCS-800 EFAS system at Idaho National Laboratories a full scale 396mm prototype brake rotor was made and can be seen in Figure 7. It was created using the same recipe described above but with longer 1 inch carbon fibers added for strength. It is designed to be used on the Joint Light Tactical Vehicle (JLTV) with a reduction in processing time of more than 90%. This is an unprecedented reduction in processing time to create Cf/SiC-ZrB<sub>2</sub> composite materials and resulted in what could be the world's largest carbon fiber silicon carbide composite part that has been produced by EFAS.



**Figure 7:** SEM micrograph of a prototype 396mm C/SiC brake rotor processed by EFAS.

A unique characteristic to EFAS technology of larger parts is the potential for extremely elevated localized heating at particle contact points. This is due to the driving force for heating being electrical current density. If an electrically conductive material has areas of extremely small cross-sectional areas, which is the case when two round objects touch, massive heating can occur. This was observed in the Cf/SiC-ZrB<sub>2</sub> part where areas of carbon fiber that were in contact with one another look to be fused with its nearest neighbors, see Figure 8. Because of the high localized heating the carbon fibers, which are mesophase, started to graphitize. The benefits of fiber graphitization within the part can include increase tensile strength, higher thermal conductivities, and higher electrical conductivities [13, 14].



**Figure 8:** SEM micrograph of the latest Cf/SiC formula for production of the scaled-up prototype brake rotor processed by EFAS.

## CONCLUSIONS

Cf/SiC-ZrB<sub>2</sub> brake rotors made via EFAS demonstrated high thermal conductivity, extremely low porosity, and maintained a high purity of materials in the matrix. Processing via EFAS methods allowed for it also to be created in dramatically reduced time making it far less expensive to manufacture. Bonding between fibers in the carbon fiber matrix was observed which is hypothesized to increase overall performance of the part.

These results make Cf/SiC-ZrB<sub>2</sub> a very strong candidate for brake rotors on heavy wheeled vehicles at a much more affordable cost to manufacture, solve the sizing requirements with high thermal conductivity, and would significantly improve supply chain to the end user.



## REFERENCES

- [1] G. Savage and G. Savage, *The properties of carbon-carbon composites*. Springer, 1993.
- [2] H. O. Pierson, *Handbook of refractory carbides & nitrides: properties, characteristics, processing and applications*. William Andrew, 1996.
- [3] S. J. Frueh, T. P. Coons, J. W. Reutenauer, R. Gottlieb, M. A. Kmetz, and S. L. Suib, "Carbon fiber reinforced ceramic matrix composites with an oxidation resistant boron nitride interface coating," *Ceramics International*, vol. 44, no. 13, pp. 15310-15316, 2018/09/01/ 2018, doi: <https://doi.org/10.1016/j.ceramint.2018.05.177>.
- [4] G. R. Devi and K. R. Rao, "Carbon Carbon Composites: An Overview," *Defence Science Journal*, vol. 43, no. 4, p. 369, 1993.
- [5] J. D. Buckley, "Carbon-carbon-an overview," *American Ceramic Society Bulletin*, vol. 67, 1988.
- [6] C. Sauder, J. Lamon, and R. Pailler, "The tensile behavior of carbon fibers at high temperatures up to 2400 °C," *Carbon*, vol. 42, no. 4, pp. 715-725, 2004/01/01/ 2004, doi: <https://doi.org/10.1016/j.carbon.2003.11.020>.
- [7] W. Krenkel and F. Reichert, "5.1 Design Objectives and Design Philosophies, Interphases and Interfaces in Fiber-Reinforced CMCs," in *Comprehensive Composite Materials II*, P. W. R. Beaumont and C. H. Zweben Eds. Oxford: Elsevier, 2018, pp. 1-18.
- [8] R. R. Naslain, "SiC-Matrix Composites: Nonbrittle Ceramics for Thermo-Structural Application," *International Journal of Applied Ceramic Technology*, vol. 2, no. 2, pp. 75-84, 2005, doi: <https://doi.org/10.1111/j.1744-7402.2005.02009.x>.
- [9] W. Yang, H. Araki, A. Kohyama, S. Thaveethavorn, H. Suzuki, and T. Noda, "Fabrication in-situ SiC nanowires/SiC matrix composite by chemical vapour infiltration process," *Materials Letters*, vol. 58, no. 25, pp. 3145-3148, 2004/10/01/ 2004, doi: <https://doi.org/10.1016/j.matlet.2004.05.059>.
- [10] C. A. Nannetti, A. Ortona, D. A. de Pinto, and B. Riccardi, "Manufacturing SiC-Fiber-Reinforced SiC Matrix Composites by Improved CVI/Slurry Infiltration/Polymer Impregnation and Pyrolysis," *Journal of the American Ceramic Society*, vol. 87, no. 7, pp. 1205-1209, 2004, doi: <https://doi.org/10.1111/j.1551-2916.2004.tb20093.x>.
- [11] J. J. Brennan, "Interfacial characterization of a slurry-cast melt-infiltrated SiC/SiC ceramic-matrix composite," *Acta Materialia*, vol. 48, no. 18, pp. 4619-4628, 2000/12/01/ 2000, doi: [https://doi.org/10.1016/S1359-6454\(00\)00248-2](https://doi.org/10.1016/S1359-6454(00)00248-2).
- [12] W. B. Hillig, "Melt Infiltration Approach to Ceramic Matrix Composites," *Journal of the American Ceramic Society*, vol. 71, no. 2, pp. C-96-C-99, 1988, doi: <https://doi.org/10.1111/j.1151-2916.1988.tb05840.x>.
- [13] A. Centeno, V. G. Rocha, A. Borrell, C. Blanco, and A. Fernández, "Fabrication of C/SiC composites by combining liquid infiltration process and spark plasma sintering technique," *Ceramics International*, vol. 38, no. 3, pp. 2171-2175, 2012/04/01/ 2012, doi: <https://doi.org/10.1016/j.ceramint.2011.10.060>.
- [14] D. Huang *et al.*, "Ablation behavior and thermal conduction mechanism of 3D ZrC-SiC-modified carbon/carbon composite having high thermal conductivity using mesophase-pitch-based carbon fibers and pyrocarbon as heat transfer channels," *Composites Part B: Engineering*, vol. 224, p. 109201, 2021/11/01/ 2021, doi: <https://doi.org/10.1016/j.compositesb.2021.109201>.