Manufacturing Strategies for NITE-processed SiC/SiC Ceramic Composite Components in the Liquid Sandwich Vacuum Vessel for Fusion Reactors

by

Shreya Agarwal

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ABSTRACT

In nuclear fusion tokamaks, vacuum vessels must be able to withstand large disruption forces resulting from potential plasma quenches. The Liquid Sandwich Vacuum Vessel (LSVV) is a novel alternative to conventional thick steel vacuum vessels. In the LSVV design, liquid lead circulates through channels within thin walls made of silicon carbide ceramic composite (SiC/SiC). By dissipating disruption forces within the liquid lead, the SiC/SiC walls can remain thin, improving heat transfer and the overall efficiency of the fusion reactor. However, SiC/SiC's brittleness and anisotropy present challenges in machining and manufacturing. A literature review explores existing strategies for manufacturing, machining, and joining SiC/SiC components within the nuclear fusion context. A manufacturing plan is proposed for the fabrication and joining of tubular components and walled structures with integrated channels, aiming to further SiC/SiC manufacturing capabilities for the LSVV design. Further research is necessary to develop and validate robust manufacturing and joining methods specific to the LSVV application.

Thesis supervisor: Kevin Woller
Title: Principal Research Scientist

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Chapter 1

Introduction

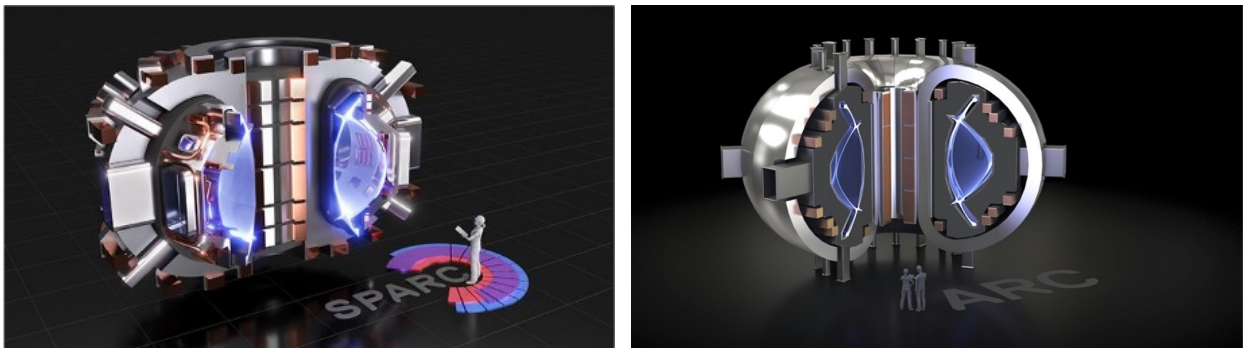
Since the Industrial Revolution, human-driven processes powered by fossil fuels have been emitting large amounts of carbon dioxide and other greenhouse gases into the atmosphere. This has caused warming of the planet along with an intensification of extreme weather patterns, affecting all life on Earth. An immediate transition to cleaner energy sources to replace fossil fuels and meet the growing energy demand is necessary.

Nuclear energy has long been considered one of the potential solutions to the climate crisis. Nuclear energy includes both nuclear fission and nuclear fusion. Nuclear fission occurs when a neutron splits a heavy radioactive atom, releasing energy along with two smaller radioactive atoms and additional neutrons. The fission reaction of uranium-235 with a neutron produces around 200 MeV, resulting in a fuel energy density of around 82 TJ/kg [1]. The neutrons produced in the fission reaction continue reacting with other radioactive elements, leading to the potential for chain reactions. Nuclear fission has existed commercially since 1957 [2].

Nuclear fusion is the combination of two lighter atoms. The most promising nuclear fusion reaction for fusion energy is that of deuterium (^{2}H) and tritium (^{3}H), producing a helium nucleus, a neutron, and 17.6 MeV of energy. This corresponds to a fuel energy density of 340 TJ/kg, which is over 4 times larger than that of fission and around 4,000,000 times larger than the energy output of burning oil or coal [3].

Fusion energy has immense potential due to the energy density and vast availability of fuel. However, there are significant engineering challenges that still exist in the way of commercializing fusion energy. Fusion energy has been studied since the 1930s [3]. There has been a recent surge in interest in fusion energy, both through academic and government organizations as well as the private sector. The MIT Plasma Science and Fusion Center (PSFC) and company Commonwealth Fusion Systems (CFS) are developing SPARC (Fig. 1.1a), a tokamak fusion reactor designed to achieve $Q > 2$ and expected to achieve a Q value as high as 11 [4]. The Q value is an important metric used to measure the viability of fusion as a source of power. The Q value of a plasma is equal to the ratio of power generated by a plasma to the power consumed by the plasma. A Q value of 2 corresponds to the plasma generating over twice as much energy as it consumes, which is an important milestone in reaching commercially viable nuclear fusion. CFS plans for the ARC reactor to come after SPARC, which would be the world's first commercial-scale tokamak reactor (Fig. 1.1b).

The tokamak is the commonly accepted best design for fusion reactors, with others also



(a) (b)

Figure 1.1: Renderings of (a) SPARC [5] and (b) ARC [6] tokamak fusion reactors, with a person for scale.

looking into concepts including inertial confinement and stellarators. In a tokamak, nuclear reactions take place in the toroidal plasma, enclosed by a vacuum vessel. Disruptions, or quenches, can occur in the plasma, where the current in the plasma jumps to the next most conductive object, typically the vacuum vessel, which contains the plasma. This vessel must be able to withstand extremely high temperatures and potential disruption forces from plasma quenches. While experimental reactors employ thick steel walls, scaling up to a commercial reactor would require even thicker walls to withstand larger disruption forces, on the order of 10 MN [7]. The increased thickness of these walls reduces heat transfer from the plasma, decreasing the overall efficiency of the reactor. Moreover, it inhibits the transfer of high-energy neutrons from the plasma to the breeding blanket, an essential process for breeding tritium, a reactant in deuterium-tritium fusion.

To address these challenges, the "liquid sandwich vacuum vessel" (LSVV) design has been proposed by researchers at the MIT Plasma Science and Fusion Center (PSFC) as an alternative to traditional metal vacuum vessels. The LSVV comprises thin walls of silicon carbide ceramic matrix composite (SiC/SiC) with channels through which liquid lead flows. Disruption forces from potential plasma quenches now jump to the liquid lead, a highly conductive material, as opposed to the semiconducting SiC/SiC walls. This approach eliminates the need for walls to withstand substantial disruption forces, enabling them to remain relatively thin. The thin walls of the LSVV promote higher heat transfer and neutron collection in the breeding blanket, increasing the overall efficiency of the reactor [7].

The LSVV team is looking into SiC/SiC composite manufactured using the Nano-Infiltration Transient Eutectic (NITE) process, which ensures low porosity. Currently, the NITE SiC/SiC composite being considered is manufactured by the NITE Corporation in Japan in the form of pre-impregnated sheets (PPS). The sheets are then stacked and sintered at high temperatures and pressures into a ceramic matrix composite (CMC).

Challenges exist in manufacturing, forming complex geometries of, and joining components made of SiC/SiC for the LSVV design, all while maintaining hermeticity and the thermomechanical properties of SiC/SiC. A review of the current status of SiC/SiC manufacturing, machining, and joining is done. A manufacturing plan for representative components to further manufacturing capabilities is developed and proposed.

Chapter 2

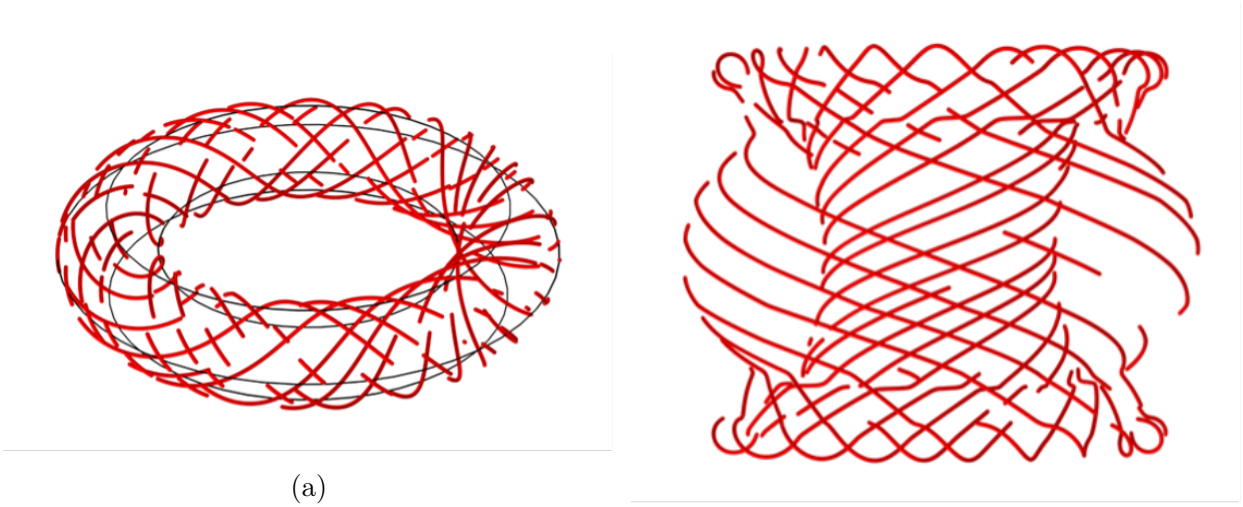
Background and Literature Review

2.1 Liquid Sandwich Vacuum Vessel

Vacuum vessel walls must be able to withstand significant forces from potential disruptions in the plasma. Traditionally, vacuum vessels have been made from steel or nickel-based alloys. When disruptions, or quenches, occur in the plasma, the current jumps to the next most conductive component, the metal vacuum vessel. Commercial-scale tokamaks require thick metal walls to handle potential disruptions. The increase in wall thickness leads to decreased heat transfer and subsequently reduces the overall energy output of the fusion reactor. Mitigating these losses is crucial for improving reactor efficiency, and the overall commercial viability of fusion.

The Liquid Sandwich Vacuum Vessel (LSVV) project explores a new design for the vacuum vessel in a tokamak. The LSVV consists of walls constructed out of SiC/SiC, a semiconductive ceramic matrix composite (CMC), with channels of liquid lead flowing through the vacuum vessel, "sandwiched" by the SiC/SiC CMC walls. To minimize vertical disruption events (VDEs), liquid lead is designed to flow in helical channels around the toroidal plasma (Fig. 2.1) [8]. The SiC/SiC walls in the LSVV can be much thinner than their steel counterparts in traditional vacuum vessels, increasing the energy generation potential of the tokamak. Thinner walls also promote neutron collection in the breeding blanket, which is essential for the production of tritium which is put back into the plasma as fuel.

Silicon carbide is selected for its favorable electrical properties and for being a low activation material, necessary in the high irradiation conditions in the fusion environment [9]. It is also suitable for high-temperature environments. However, being a ceramic, silicon carbide suffers from poor mechanical properties and exhibits brittle failure. When used as SiC/SiC in a composite form, the material exhibits increased ductility and is more suitable as an engineering material [9]. In order to be used in the LSVV, silicon carbide must be able to be manufactured and joined into complex geometries, as helical channels in walls surrounding a toroidal plasma are required. SiC/SiC and corresponding joints must be hermetic, or leak tight, to maintain a high-vacuum environment for the plasma and prevent leaking of lead from channels. The manufactured components must retain their strength, thermal conductivity, and hermeticity at the high temperatures and under the high irradiation conditions of the fusion environment.



(a)

(b)

Figure 2.1: Concept for helical channels of liquid lead around toroidal plasma. Liquid lead would be contained in channels in SiC/SiC vacuum vessel walls [8].

2.2 Silicon carbide

Ceramics are a class of materials defined as "nonmetallic inorganic solids" [10]. Ceramics are typically brittle, which can be a problem when implemented in engineering applications where fracture and crack propagation are unacceptable [10]. Above the glass transition temperature, ceramics behave as a viscous liquid. Ceramics tend to be stronger in compression as opposed to tension, which is an important consideration when designing parts out of ceramic materials [10].

Silicon carbide (SiC) is a ceramic material. It is often used in aerospace and nuclear applications due to its desirable properties in extreme conditions (Fig. 2.2, 2.3) [11][12]. In aerospace, SiC/SiC is chosen for hot section components in engines and turbines due to its high performance under extreme conditions and relatively low weight [13]. Specifically in the context of nuclear fusion, silicon carbide is chosen for its favorable electrical properties, resistance to corrosion, low neutron activation, and low tritium permeability [7].

Monolithic SiC is produced through a sintering process where SiC powders are heated and form a solid without melting [10]. Powder particles, often modeled as spheres, are joined together at grain boundaries. Depending on how monolithic SiC is manufactured and how particles are packed, monolithic SiC can be quite dense and satisfy the need for hermeticity in a fusion material. However, its inherent brittleness and low fracture toughness make it difficult to be used as a fusion material [14].

Ceramic matrix composites (CMCs) consist of ceramic fibers held together in a ceramic composite. CMCs combine properties of ceramics with an increased toughness, which can be attributed to a higher amount of energy required for crack propagation [10]. CMCs are often used in engineering applications. Silicon carbide fiber-reinforced silicon carbide matrix (SiC/SiC) is a CMC that has been proposed to be used for the ceramic walls in the LSVV design.

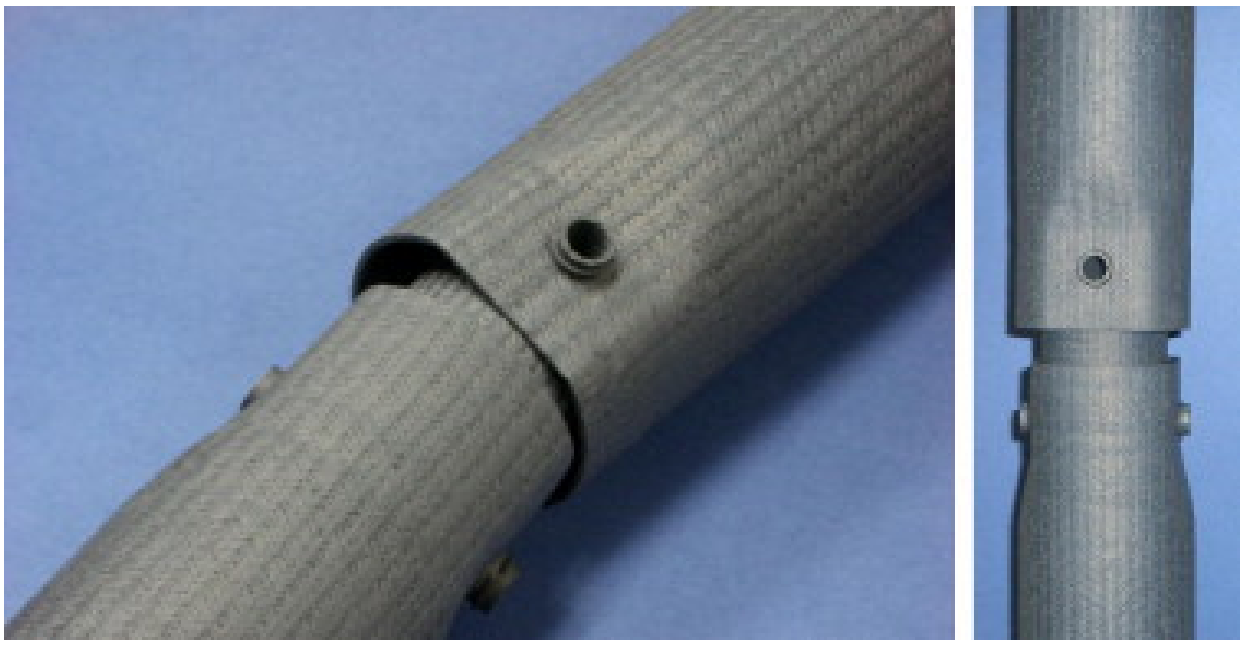


Figure 2.2: Assembled nuclear grade SiC/SiC control rod sheath joint. Designed for a high-temperature gas-cooled fission reactor. [9]

2.2.1 SiC/SiC: Manufacturing

SiC/SiC is manufactured by various processes including CVI, PIP, and NITE. Chemical vapor infiltration (CVI) uses reactive gases at high temperatures to infiltrate the SiC preform with matrix SiC [16]. SiC/SiC composites produced through the CVI method are inherently porous, decreasing the maximum stress that the material can handle and its thermal conductivity. CVI is also expensive as it can take days or weeks to achieve full densification of a composite [7]. In the polymer impregnation and pyrolysis (PIP) process, a polymer-based silicon slurry is impregnated into preform SiC fabric. The infiltrated fabric is then heated, causing the polymer to decompose and turn into a ceramic, with only silicon and carbon remaining from the polymer [17]. This process has been developed industrially and is cost-effective. It requires relatively low temperatures (1100-1200°C) and has a time scale on the order of hours [7]. However, PIP-formed SiC/SiC typically has a poor microstructure compared to composites formed by CVI and NITE processes and is often subject to cracks from thermal stresses [7]. There are also concerns about the radiation instability of PIP-formed SiC/SiC in neutron environments due to non-stoichiometric matrices resulting from the pyrolysis process [18].

NITE process

The nano-infiltration and transient eutectic (NITE) process has been developed to minimize porosity, thus increasing the maximum applicable stress and thermal conductivity of SiC/SiC and maintaining hermeticity (Fig. 2.4) [18]. In the NITE process, fiber surfaces are first coated in pyrolytic carbon (PyC). These coated fibers are then impregnated in a nano-slurry during infiltration. The nano-slurry consists of nano-sized SiC powder and sintering additives

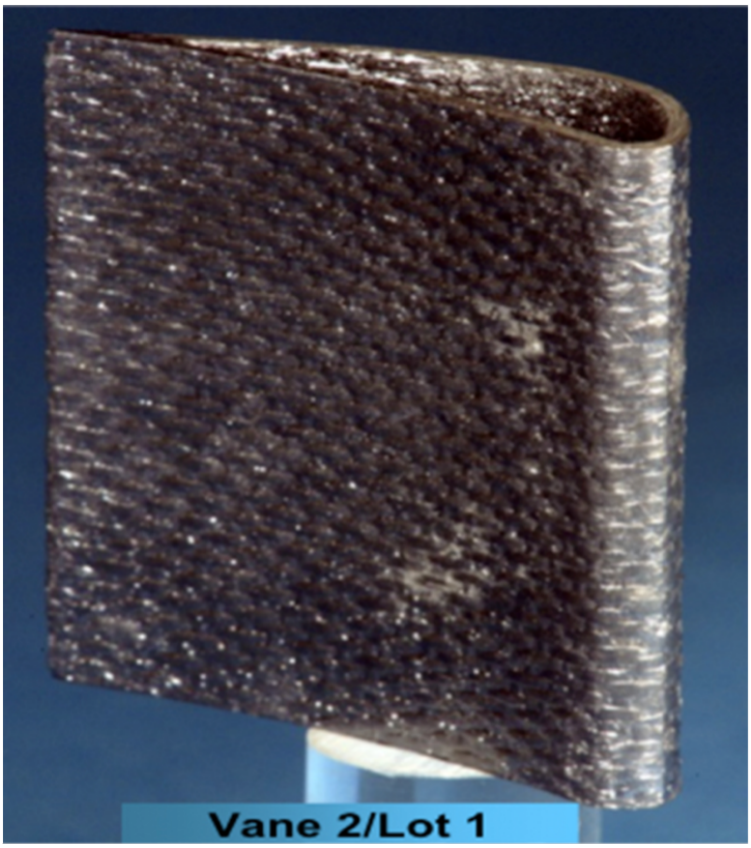


Figure 2.3: SiC/SiC turbine engine guide vane developed at NASA Glenn. [15]

such as Al_2O_3 and Y_2O_3. The infiltrated fibers are then formed into pre-impregnated, or "prepreg", composite sheets and dried. Prepreg sheets are stacked and hot pressed at high temperatures (1800-1900°C) under high pressures (∼20 MPa) to form the solid SiC/SiC [19].

During hot pressing at temperatures higher than the transient eutectic phase melting point, liquid-phase sintering occurs. The SiO_2 particles from the nano-sized SiC and sintering additives including Al_2O_3 and Y_2O_3 enter a transient eutectic liquid phase since the combination of substances has a lower melting point than individual substances alone. This allows for improved infiltration and the formation of a denser composite [18].

NITE-processed SiC/SiC has demonstrated high strength, high thermal conductivity, hermeticity, and stability at high temperatures. These properties are due to the better infiltration of SiC powders and additives into the SiC preform and the resulting dense SiC matrix [18]. The increased strength and pseudo-ductile behavior of NITE SiC/SiC is also attributed to the PyC coating, which allows for a weak interface between the matrix and fabric. A weak interface allows loads to cause movement between the matrix and fabric, resulting in a toughening of the composite. In a stronger interface, loads would instead lead to cracking of the interface, and thus fracture in the composite [21].

Various silicon carbide fibers have been developed and used in SiC/SiC. The manufacturing process and resulting microstructure of fibers affect the final properties of the SiC/SiC CMC. The Tyranno SA3 is a third-generation stoichiometric fiber, with a near stoichiometric ratio of carbon to silicon atoms. It is designed for performance at high temperatures and

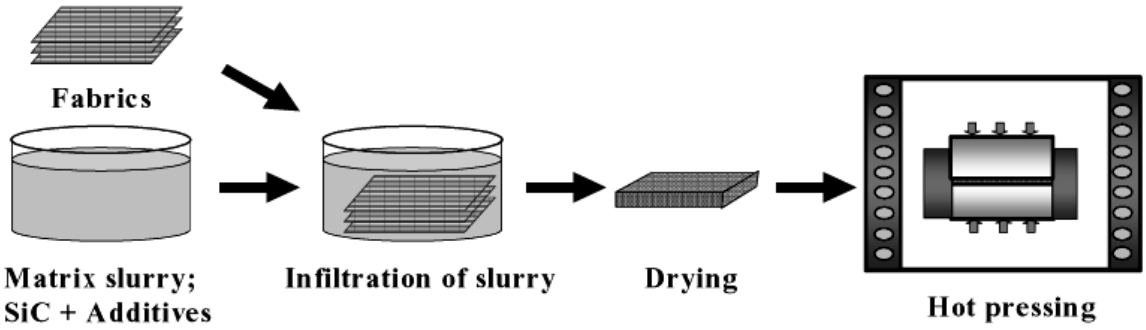


Figure 2.4: NITE SiC/SiC manufacturing process. [20]

extreme environments, including nuclear contexts [22][23]. Tyranno SA3 has been used in the NITE manufacturing process for SiC/SiC.

Electrophoretic deposition

Electrophoretic deposition (EPD) is the infiltration of ceramic fabric preforms by suspending the fiber preform in a ceramic slurry and applying an electric field to it, with the fiber preform serving as the positive electrode (Fig. 2.5). This process attracts particles to the preform, facilitating their deposition within it and allowing for high infiltration and consequent densification of the composite. Subsequently, the fabric undergoes hot pressing and additives enter the transient eutectic liquid phase, similar to the NITE process [24]. EPD has been used extensively in ceramic applications. Novak et al. observed that EPD increased the infiltration of SiC fabric, leading to a higher density and reduced pore formation in the final composite [25]. Lee et al. found that SiC/SiC composites produced via EPD exhibited superior density and flexural strength compared to those made using conventional methods [26]. Achieving densification and minimizing pore formation in SiC/SiC composites is crucial for their fabrication and utilization as hermetic materials. Idris et al. found that swelling resulting from neutron irradiation was smallest in SiC/SiC composites formed through EPD compared to those formed through the CVI and NITE processes [27].

SiC tape

SiC tape has been shown to increase density in composites. SiC tape is cast from a slurry of SiC powder, sintering additives, and polyester/polyamine co-polymeric dispersant [21]. In a study done by Yonathan et al., NITE SiC/SiC composites, both with and without layers of tape, and monolithic SiC were compared. The slurry used to form the tape consisted of

- polyvinyl butyral (PVB): a resin used as the binder phase,
- dioctyl phthalate (DOP): plasticizer,
- SiC powder (purity $>$ 97.5%, $<$0.75% carbon, $<$1.25% oxygen) with additives Al_2O_3, Y_2O_3, and MgO at weight ratio 6.4:2.6:1.0, and
- a polyester/polyamine co-polymeric dispersant.

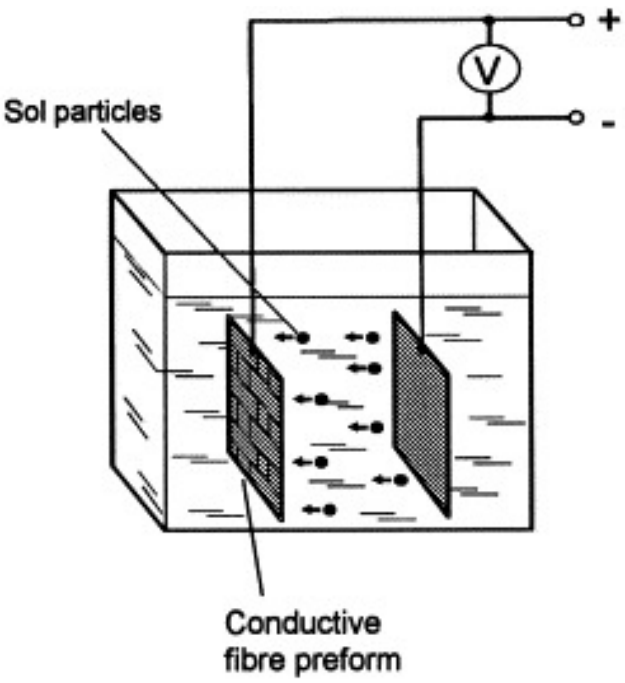


Figure 2.5: EPD process for ceramic fiber preforms. A voltage is applied to the ceramic slurry, causing particles to travel to the preform and increasing infiltration. [24]

The binder phase binds SiC tape together into a tape-like structure. It is burned out of the composite at a lower temperature before hot pressing.

Monolithic SiC had a maximum density of 3.19 g/cm^3 and NITE composites without tape inserts had a maximum density of 3.02 g/cm^3. NITE composites formed with tape inserts between every layer of SiC fabric had a maximum density of 3.13 g/cm^3, less than monolithic SiC but greater than tape-less NITE SiC/SiC [21]. The SiC/SiC surface of the composite without inserts has pores, as seen in SEM images in Figures 2.6a and 2.6b. In comparison, the SiC/SiC surface of the composite with tape inserts (Fig. 2.6c, 2.6d) has no visible pores in the section shown. The layering of tape between SiC fabric helped densify the matrix by providing extra SiC and sintering additives at every layer, which then enter the transient eutectic liquid phase during hot pressing at high temperature and fill gaps. NITE SiC/SiC without tape only has the SiC powder and sintering additives already present in the matrix during the infiltration step. The composite with tape inserts also demonstrated a higher flexural strength of 562 $\pm$ 36 MPa, compared to a strength of 312 $\pm$ 28 MPa, in the traditional composite. Thus, the layering of this tape increases the overall composite density and strength, since fewer pores mean fewer locations for crack propagation [21].

2.2.2 SiC/SiC: Machining

Machining is still a major challenge when working with SiC/SiC CMCs in engineering applications. SiC/SiC is brittle, hard, heterogeneous, and anisotropic, making it difficult to machine. Silicon carbide's high brittleness negatively impacts the quality of the machined surface. High microhardness causes high tool wear. The heterogeneity and anisotropy of

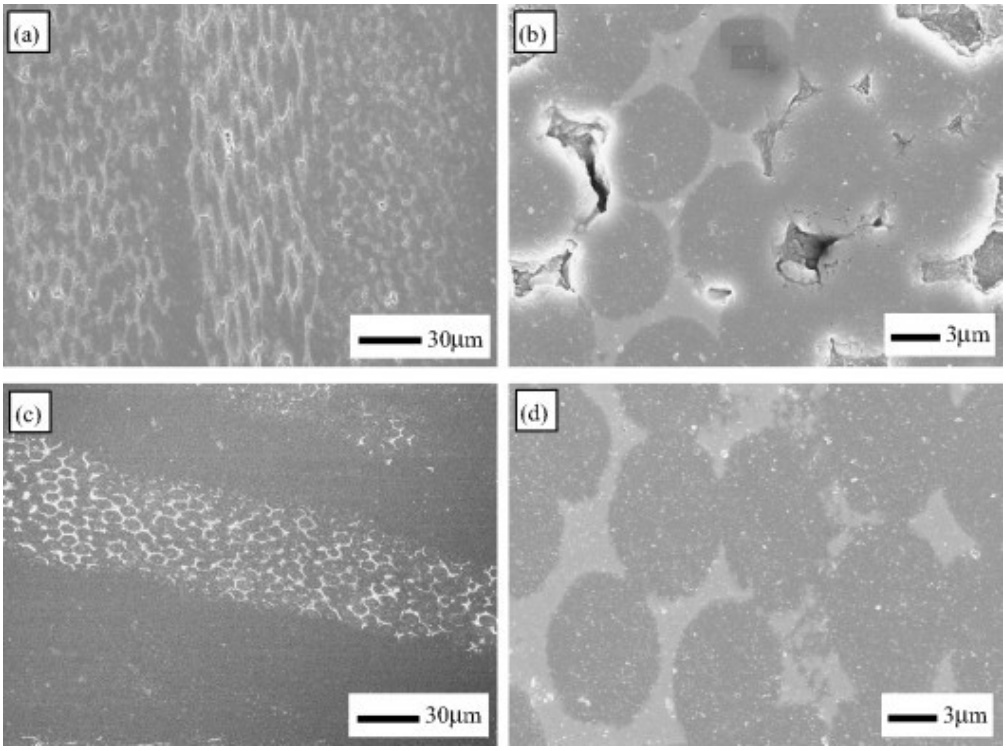


Figure 2.6: SEM images of SiC/SiC surface after polishing (a,b) no tape, typical NITE process and (c,d) NITE process with tape inserts. Note the difference in the presence of pores. [21]

SiC/SiC mean machining quality is highly dependent on fiber orientation and machining direction [13]. Both conventional machining (CM) and ultrasound-assisted machining (UAM) have been found to be effective for machining SiC/SiC.

Conventional machining

Conventional machining includes grinding, milling, and drilling. These processes are well-known and typically the easiest to access. Optimizing parameters is key in implementing conventional machining of SiC/SiC. Due to silicon carbide's high hardness, diamond abrasive tools are typically required. Carbide tools were found to have significant tool wear [13]. Key material failure modes are matrix breakage, fiber fracture, and delamination [13]. When considering grinding, it was generally found that decreasing the depth of grinding and decreasing the feed rate result in better surface finish and a smaller grinding force. Polishing is conventionally done using diamond abrasives. Drilling was most effective with diamond drills, high speed, and low feed rate [13]. Polycrystalline diamond (PCD)—synthetically sintered diamond particles—tools have been effective in the milling and drilling of silicon carbide composites. Minimal quality lubrication (MQL) is the use of a relatively small quantity of lubrication alongside air pressure and was found to decrease grinding force and improve the surface quality of the machined part. Decreasing grinding forces allows for decreased grinding wheel wear and, thus, improved grinding efficiency [13]. However, liquid lubricant can be difficult to remove from pores in SiC/SiC, so it is important to consider the compatibility of

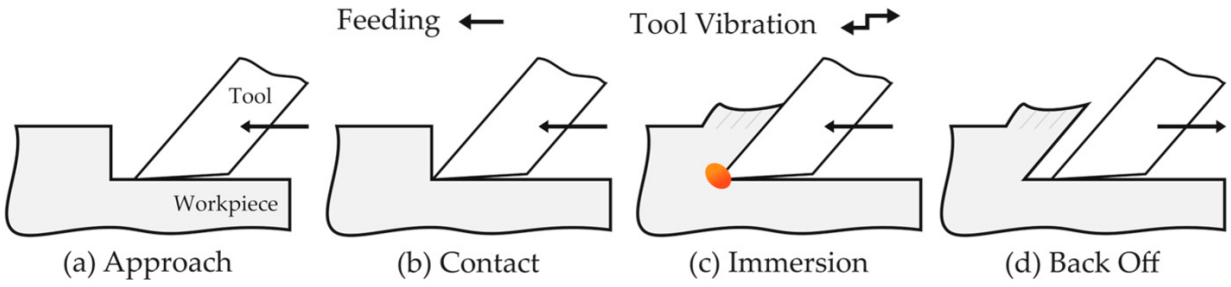


Figure 2.7: Movement of tool in vibration cycle for UAM process. [28]

the lubricant, and coolants in general, with SiC/SiC and its applications [13]. MQL systems must be added onto traditional machines. Overall, conventional machining with diamond abrasive tools or PCD tools can be effective, but extremely high forces are still required, and parts often result in poor surface finishes. Machines and tools also suffer severe wear. The overall efficiency of the conventional machining process is quite low due to low feed rates, increasing overall machining costs.

Ultrasound-assisted machining

Ultrasound-assisted machining (UAM) is known to be effective for hard and brittle materials, including silicon carbide composites. In the UAM process, high-frequency, low-amplitude vibration is applied to a typical cutting tool (Fig. 2.7). This makes the tool-material interaction discontinuous, allowing for greater efficiency. Through vibrations, the workpiece material is broken down and the overall cutting resistance is decreased. Vibrations can occur in either the tools or the workpiece. This reduces the required cutting force and heat generated, improves surface finish, and reduces general wear on the machine and tool. UAM can be used in turning and drilling processes [28]. Ultrasonic-assisted grinding of C/SiC composites was found to reduce cutting force by 20% and surface roughness by 30% [13].

Other machining methods being explored for SiC/SiC include abrasive water jet (AWJ), laser-assisted machining (LAM), and electrical discharge machining (EDM).

2.2.3 SiC/SiC: Joining

It is challenging to machine and form SiC/SiC into the complex geometries required for the LSVV. It is also impractical to manufacture the LSVV SiC/SiC wall in one piece due to its sheer size and complex geometries. Thus, learning to join SiC/SiC CMCs is crucial to manufacturing the liquid sandwich vacuum vessel in tokamaks out of SiC/SiC. Joints must be hermetic and maintain strength under neutron irradiation and in high temperatures.

Reliable joining methods for CMCs have been developed and are used regularly in non-fusion applications. Mechanical joints are a popular method of joining SiC/SiC in existing engineering applications (Fig. 2.2, 2.8, 2.9). However, mechanical joints will not work for fusion applications because they are typically not hermetic [29].

Various methods of joining have been proposed, attempted, and have the potential for use in nuclear fusion applications. These include solid-state diffusion bonding, metal diffusion

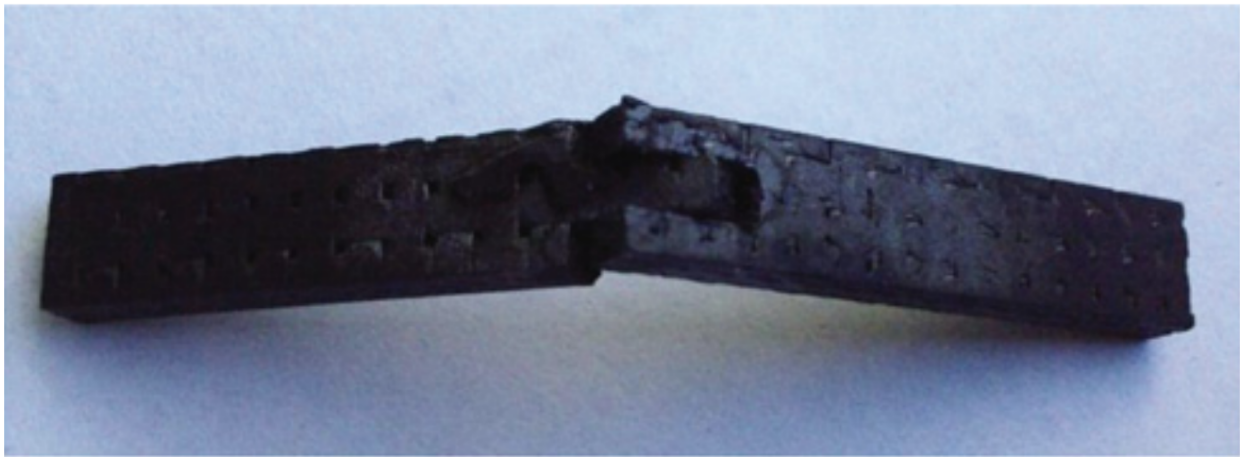


Figure 2.8: A laser-machined SiC/SiC joint. [30]

bonding, MAX-phase joining, transient eutectic-phase joining, glass-ceramic joining, metallic braze-based joining, Si-C reaction bonding, and polymer-derived SiC joining [29].

Solid-state diffusion bonding

"Pure" solid-state diffusion bonding refers to the joining of ceramics at high temperatures and pressures. Bonds are formed at an atomic level, allowing for the joint to be chemically pure and structurally continuous [29]. While joints formed through solid-state diffusion are desirable, the high temperatures and pressures required often make them infeasible. Typically, temperatures must reach at least $0.75T_{melting}$ to be effective in forming atomic bonds [29]. The melting temperature of silicon carbide is 2830°C [32], meaning effective solid-state diffusion bonding requires temperatures of around 2100°C.

Metal diffusion bonding

Metal diffusion bonding is the same process as solid-state diffusion but also includes a metal insert in between joining surfaces. Metal diffusion bonding occurs at temperatures greater than 1200°C. Typical metals used are titanium, molybdenum, tantalum, niobium, and tungsten. Metals are either inserted as a thin metal foil or powder slurry. At these high temperatures, the metals form carbides and thus are converted into ceramic phases [29].

$$(xy)\mathrm{SiC} + (x+y)\mathrm{M} \rightarrow (y)\mathrm{MC}_x + (x)\mathrm{MSi}_y. \tag{2.1}$$

In silicon carbide, titanium is often used in metal diffusion bonding. Silicon carbide joints using titanium have been shown to have shear strengths of over 100 MPa [29]. Silicon carbide and titanium have different coefficients of thermal expansion (CTEs) which can limit the overall strength of joints. Tuning process parameters allows for some control over the distribution of products (TiC,), and thus over the CTE. In Koyanagi et al., SiC/SiC was joined through molybdenum diffusion bonding. Significant vertical cracks were introduced in the component after neutron irradiation (Fig. 2.10). Cracks came together and formed "networks". Cracks propagated similarly in the titanium diffusion bond. The cracking of

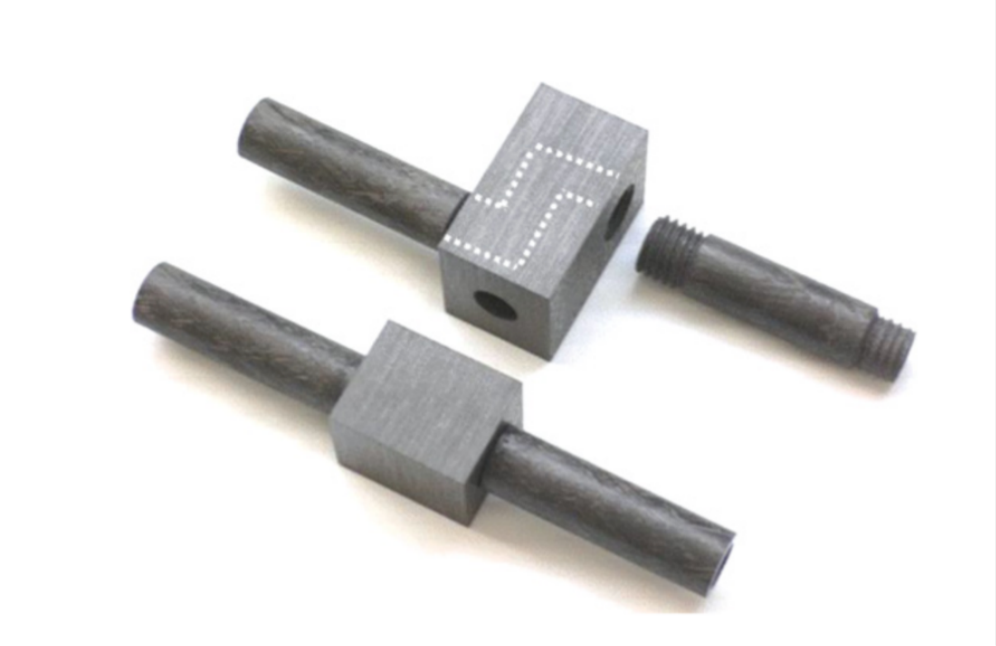


Figure 2.9: A threaded SiC/SiC joint. [31]

molybdenum and titanium joints is attributed to a difference in the CTEs of SiC and the metal reactant. The difference in CTEs causes residual tensile stress during bonding and different swelling rates, resulting in crack propagation [33].

Tungsten has been considered to form favorable joints with SiC and is said to be preferred in the long term due to minimal neutron activation [29]. Tungsten and SiC have similar CTEs, which would decrease swelling differences due to neutron irradiation [34]. However, limited testing has been done on the effects of neutron irradiation on tungsten diffusion joints.

MAX-phase bonding

MAX-phase joining is a variation of solid-state diffusion joining, where MAX-phase ceramics are used as inserts. MAX-phase materials are a group of carbides and nitrides that exhibit both favorable metallic and ceramic properties and are often used in ceramic applications [35]. MAX-phase ceramics often have properties including strength at high temperatures and pseudo-ductility, which can be beneficial in SiC/SiC joints [29]. Ti_3SiC_2 joints have been successfully demonstrated in SiC/SiC [29]. However, there is concern about the integrity of MAX-phase joints under high neutron irradiation. The CTEs of silicon carbide and titanium do not match, limiting the overall strength of the joints [29]. In Katoh et al, microcracks appeared in the Ti_3SiC_2 layer after neutron irradiation at 800°C to around 5 dpa [29]. Koyanagi et al found that MAX-phase joints did not suffer degradation after high-dose neutron irradiation at 530°C to 20 dpa over 350 days. However, irradiation-induced cracks appeared in all MAX-phase joints, likely from differential swelling in the SiC and MAX-phase ceramic joints [36].

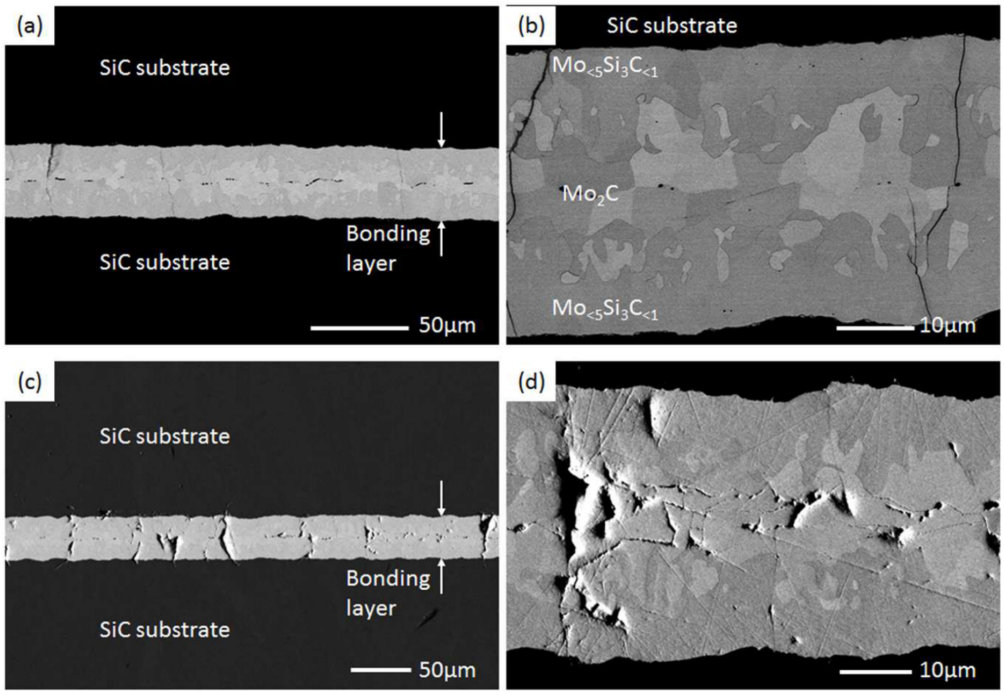


Figure 2.10: (a,b) Unirradiated SiC/SiC composite with molybdenum diffusion joint. (c,d) Irradiated SiC/SiC composite with molybdenum diffusion joint. "Vertical" cracks can be seen in the bonding layer after irradiation. [33]

Transient eutectic-phase joining

Transient eutectic-phase (TEP) joining, also known as liquid-phase sintering (LPS) has been shown to be successful in creating high-strength joints between SiC/SiC CMCs for fusion conditions. TEP is a broader group of processes under which the NITE process falls. TEP joining is sometimes referred to as NITE-like joining. The TEP, or LPS, joining process refers to the hot pressing of two substrates with a sinterable powder at the joint [37]. This process can form hermetic joints, which are essential for fusion applications, specifically the LSVV. "Pressureless", or low-pressure TEP joining has also been demonstrated to be a successful joining technique, where lower pressures (from 0.1 to 5 MPa) are used to hold components in place without the objective of subjecting them to high pressures [36][38].

Hinoki et al. demonstrated successful TEP joining for both monolithic SiC and SiC/SiC CMCs. Plates of both monolithic SiC and SiC/SiC were individually joined with a SiC nano-powder and tungsten as a sintering additive. They were hot-pressed together for one hour at a pressure of 20 MPa and at a range of temperatures from 1700 to 1900°C. SiC/SiC joints were found to have a worse surface roughness compared to monolithic SiC joints, with pores appearing in the joint. Monolithic SiC joints demonstrated the highest shear strength at temperatures of 1780°C and 1850°C. At higher temperatures, the compound W5Si3 formed and resulted in the pores developing at the joint, decreasing the overall shear strength of the joint. This is attributed to the larger size of pore formation when the compound W_5Si3 was present, compared to pores when only WSi_2, WC, and W_2C were present. At temperatures lower than 1780°C, both the tensile and shear strength of the joints dropped drastically. The

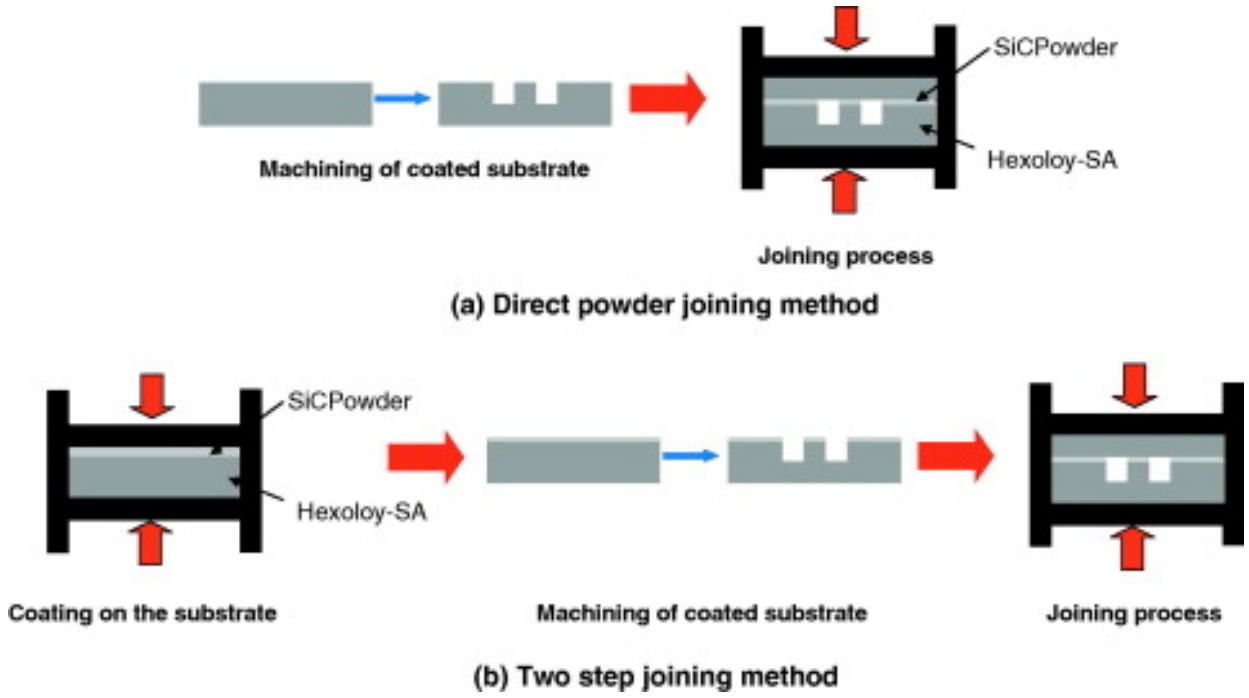


Figure 2.11: (a) Direct powder joining method for SiC/SiC composites with channels, (b) Two step joining method for SiC/SiC composites with channels. [37]

monolithic SiC joint formed at a sintering temperature of 1780°C was found to have the highest shear strength of 90 MPa. The maximum observed tensile strength was 250 MPa. Hinoki et al. found that higher shear strength was correlated to a smaller reaction layer [39].

Jung et al. demonstrated successful TEP joining of monolithic SiC with channels using a combination of Al_2O_3, Y_2O_3, and SiO_2 as joining adhesives and nano-powder SiC. TEP joints were made at a range of temperatures (1500°C to 1900°C) and pressures (5 MPa to 20 MPa). Two different methods were used: direct powder joining and two-step joining (Fig. 2.11). In the direct powder joining method, channels were first machined into the substrate. Then SiC powder and the other sintering additives were coated on the substrate and joined at high pressure and temperature for one hour. In the two-step joining method, the substrate was first coated with SiC powder and additives and hot pressed briefly to adhere the SiC powder and additives to the substrate (first joining step). The channels were machined into the coated substrate and components were then joined at high pressure and temperature for one hour (second joining step). An overview of the machining process and corresponding machining parameters were not given. Two-step joining was only done at 1800°C and 20 MPa [37].

Direct powder joining resulted in the deformation of the silicon carbide and, thus, an incomplete joining layer. The two-step joining method resulted in a uniform joint layer and had a higher tensile strength compared to direct powder joining. Two-step joining as opposed to direct powder joining at 1800°C and 20 MPa increased the tensile strength of the joint from 249 MPa to 300 MPa, greater than the 250 MPa achieved by Hinoki et al. [37].

Koyanagi et al found that high-dose neutron irradiation at 530°C to 20 dpa (displacement per atom) over 350 days on LPS SiC joints did not degrade joints, indicating that joints can

withstand high long-term neutron damage. Resistance to high-dose neutron irradiation was also demonstrated for CA glass ceramics joints and hot-pressed MAX-phase joints [36].

While diffusion bonding and TEP joining are the most promising, other potential joining methods for SiC/SiC composites include glass-ceramic joining, metallic braze-based joining, Si-C reaction bonding, and polymer-derived SiC joining [29].

2.2.4 SiC/SiC: Forming complex geometries

Manufacturing components including the liquid sandwich vacuum vessel out of SiC/SiC will require the fabrication of complex geometries. Although machining methods exist, they are inefficient and expensive and often result in a poor surface finish.

Yu et al. demonstrated the fabrication of dense, tubular SiC/SiC composite through infiltration by electrophoretic deposition (EPD) and hot pressing [40]. Plain-woven Tyranno-SA3 SiC fabric was wrapped 5 or 15 times around a graphite rod and placed in a ceramic slurry made up of SiC and the sintering additive Al_2O_3-Y_2O_3. SiC tape made from SiC and the sintering additive Al_2O_3-Y_2O_3 was inserted between every layer of the SiC fabric. In the EPD process, 10 V was applied for one hour, with the positive and negative ends connected to the cylindrical stainless steel wall containing the slurry and the graphite rod, respectively (Fig. 2.12). During the first 50 minutes, 10 W ultrasonic pulses were applied on a 1-second cycle. Ultrasonic pulses increase the infiltration of matrix particles into the SiC fabric by repeatedly detaching surface-level matrix particles, allowing particles to infiltrate deeper into the fabric. The infiltrated preform fabric was then hot pressed at 1750°C or 1950°C and 20 MPa for 2 hours in a mold designed to apply even pressure to the tubular composite through the use of graphite powder (Fig. 2.13). Uniform pressure distribution allowed for the formation of a uniform, dense tubular SiC/SiC composite. The resulting composite was 96% dense with respect to the density of silicon carbide and had a density of 3.05 g/cm^3. In comparison, a tubular composite manufactured through the same process, but in a conventional mold, without graphite powder for uniform pressure distribution, had a density of 55% [40].

Sharma et al. demonstrated the fabrication of dense and tough SiC/SiC composites, both with channels and in a tubular shape, through EPD and hot-pressing (Fig. 2.14). EPD with ultrasonic pulses was used for infiltration. Fabric and 35 μm-thick SiC tape were layered and hot-pressed. To form the tubular component, pressure was applied in a mold through graphite powder, similar to in Yu et al [40]. The resulting composites had densities of 94–98% of the theoretical SiC density. Pre-ceramic polymers, β-SiC nanopowder, and sintering additives including MAX-based phases (Ti_3AlC_2 and Ti_3SiC_2) were used as fillers in the form of powders and tape for joining. "Pressureless" joining was used, with 3.5 MPa applied to hold components together. The temperature was then raised to 1750 °C and components were held in place for 2 hours. Joints had a strength of 161 ± 12 MPa [38].

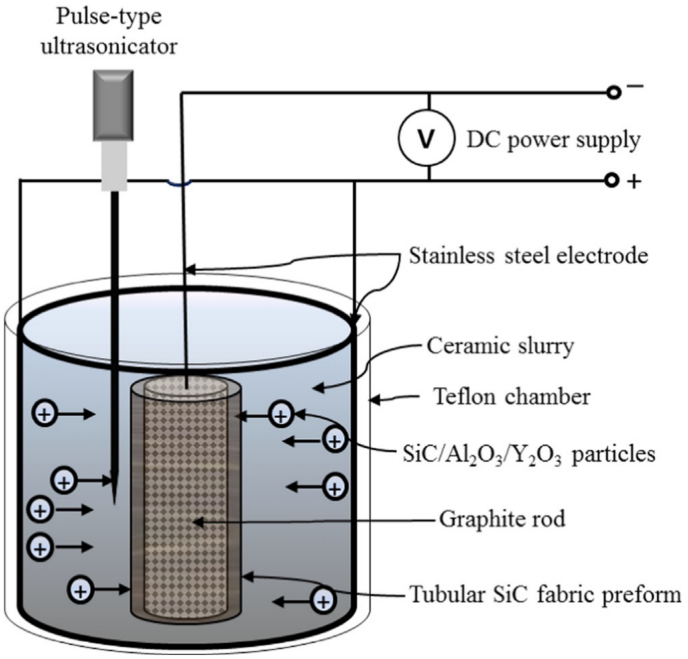


Figure 2.12: EPD diagram. SiC fabric preform wrapped around a graphite rod is placed in a ceramic slurry. A voltage with ultrasonic pulses is applied to the stainless steel container and graphite rod to enhance the infiltration of slurry particles into the preform. [40]

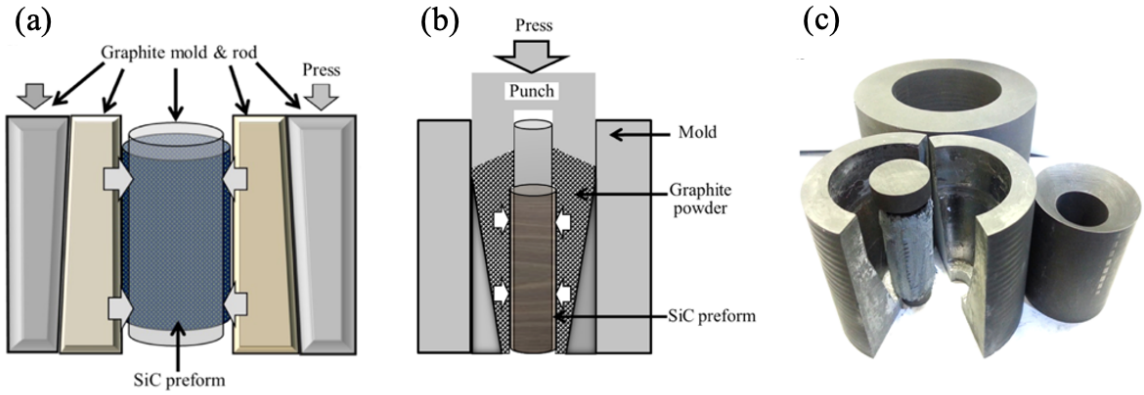


Figure 2.13: (a) Conventional mold design, (b) out-in mold design, and (c) out-in mold. [40]

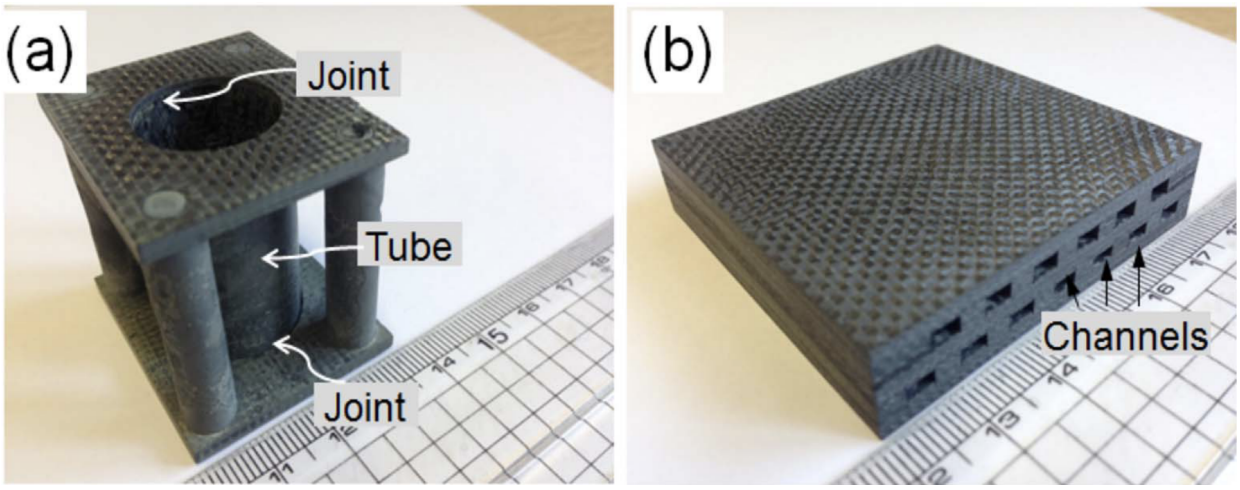


Figure 2.14: Channels were machined, and then components were joined. [38]

Chapter 3

Manufacturing plan

In order to design and manufacture the SiC/SiC LSVV for an ARC-scale tokamak, composite components with curvature and channels for liquid lead will need to be formed and joined to form the vacuum vessel. The following manufacturing plans explore key capabilities that must be developed in order to manufacture the SiC/SiC LSVV.

The first manufacturing plan is for the forming and joining of two tubular components. From the literature review, no work was found joining two tubular components of SiC/SiC. This joint will be important if the LSVV is manufactured in pieces where components with channels for liquid lead will need to come together at leak-tight joints. In this joint, there is also limited area for bonding, which may affect the strength of the joint. The manufacturing plan is as follows.

1. Begin with plain-woven preforms of Tyranno SA3 SiC fabric.
2. Coat the preforms with a 200 nm-thick layer of PyC [21].
3. Infiltrate the SiC fabric preforms using the EPD process. Place a sheet of preform fabric around a graphite rod and place in a stainless-steel vessel containing ceramic slurry made up of SiC and the sintering additive Al_2O3-Y_2O_3 (Fig. 2.12). Apply 10 V for one hour, with the positive terminal connected to the stainless-steel wall and the negative terminal connected to the graphite rod. Apply 10 W ultrasonic pulses on a 1-second cycle for the first 50 minutes. Repeat this process for all required preforms.
4. Manufacture SiC tape. Create a ceramic slurry containing PVB, DOP, SiC powder, sintering additives (Al_2O_3, Y_2O_3, and MgO), and a polyester/polyamine co-polymeric dispersant. See Yonathan et al for ratios [21]. Cast tape at a thickness of 40-60 μm using a tabletop tape caster.
5. Wrap 10 layers of infiltrated preforms around a graphite rod. Alternate layers with SiC tape, for a total of 19 layers of SiC preforms and SiC tape.
6. Heat the wrapped graphite rod at 350°C for 2 hours in air to burn out the polymeric binder PVB before hot pressing.

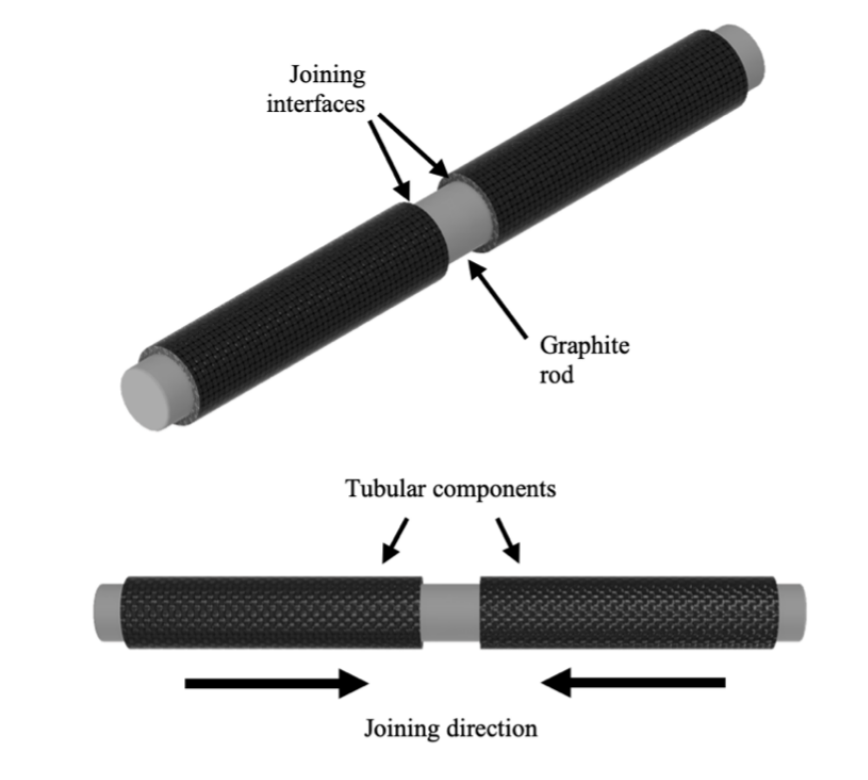


Figure 3.1: Rendering of tubular SiC/SiC components in joining orientation with graphite rod. Note: this is a rendering, manufactured SiC/SiC will likely look different.

7. Hot press component at 1750°C for 2 hours at a pressure of 20 MPa to achieve liquid phase sintering. Use a special mold (Fig. 2.13) as designed in [40] to apply even pressure to component using graphite powder.

8. Repeat steps 5-7 to form a second tubular component.

9. Slide both tubular components onto the same graphite rod. Coat both joining surfaces with ceramic slurry containing SiC and the sintering additive Al_2O_3–Y_2O_3, the same composition as that used during EPD infiltration. Push tubular components together such that the joining surfaces are in contact. Apply 3.5 MPa of even pressure from both outside ends of the tubular components to hold components in place (Fig. 3.1).

10. Join tubular components through TEP joining at 20 MPa and 1750°C for 2 hours. Components need to be constrained during this process and pressure should be applied uniformly normal to the joining interface of the two components. Uneven application of pressure will lead to stresses and potential fracture. Another option is "pressureless" joining at 3.5 MPa and 1750°C for 2 hours, which decreases the risk of fracture.

The second manufacturing plan is for the forming and joining of two walls with cylindrical channels. In the LSVV design, liquid lead must flow through helical channels around the toroidal-shaped plasma. Thus, SiC/SiC walls will need to be manufactured with these complex channels and joined such that channels are leak-tight. Due to the high cost and

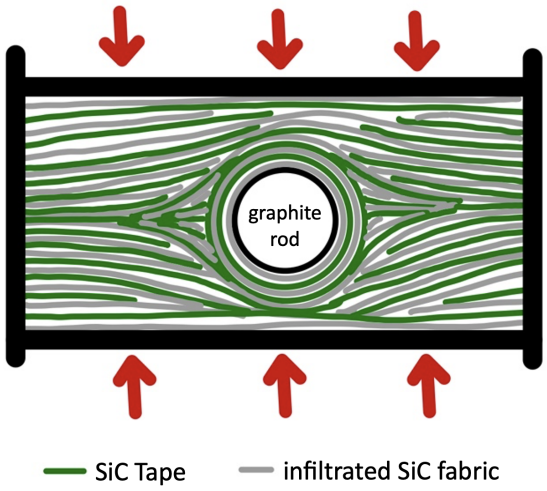


Figure 3.2: Cross-section view of layering of infiltrated SiC fabric and SiC tape in hot press to form SiC/SiC wall with channel. Pressure is applied from top and bottom.

poor surface finish of machining SiC/SiC and the complex geometries required, it would be impractical to machine channels into the walls of the LSVV. Forming necessary geometries during the SiC/SiC composite manufacturing process is also cost-effective as it prevents waste of expensive fibers and minimizes diamond machining required by subtractive machining methods [9].

This test explores manufacturing SiC/SiC walls with channels through them.

1. Repeat steps 1-4 from the previous manufacturing plan.
2. Wrap infiltrated SiC preform fabric alternating with SiC tape around the graphite rod: 5 layers of fabric with 4 layers of SiC tape. The graphite rod is a placeholder for the cylindrical channel.
3. In the rectangular, wall-shaped mold of the hot press, alternate between varied sizes of layers of infiltrated SiC preform fabric and SiC tape to get an even thickness of SiC as demonstrated in Figure 3.2. An even thickness of SiC will allow for the formation of a composite with uniform density, which is important for creating a dense and strong SiC/SiC composite.
4. Hot press the component at 1750°C and 20 MPa for 2 hours.
5. Repeat steps 1-4 to form a second wall component with a cylindrical channel.
6. Place graphite rod through channels in both components. Coat both joining surfaces with ceramic slurry containing SiC and the sintering additive Al_2O_3–Y_2O_3. Push components together such that joining surfaces are in contact (Fig. 3.3).
7. Join components through TEP joining at 20 MPa and 1750°C for 2 hours.

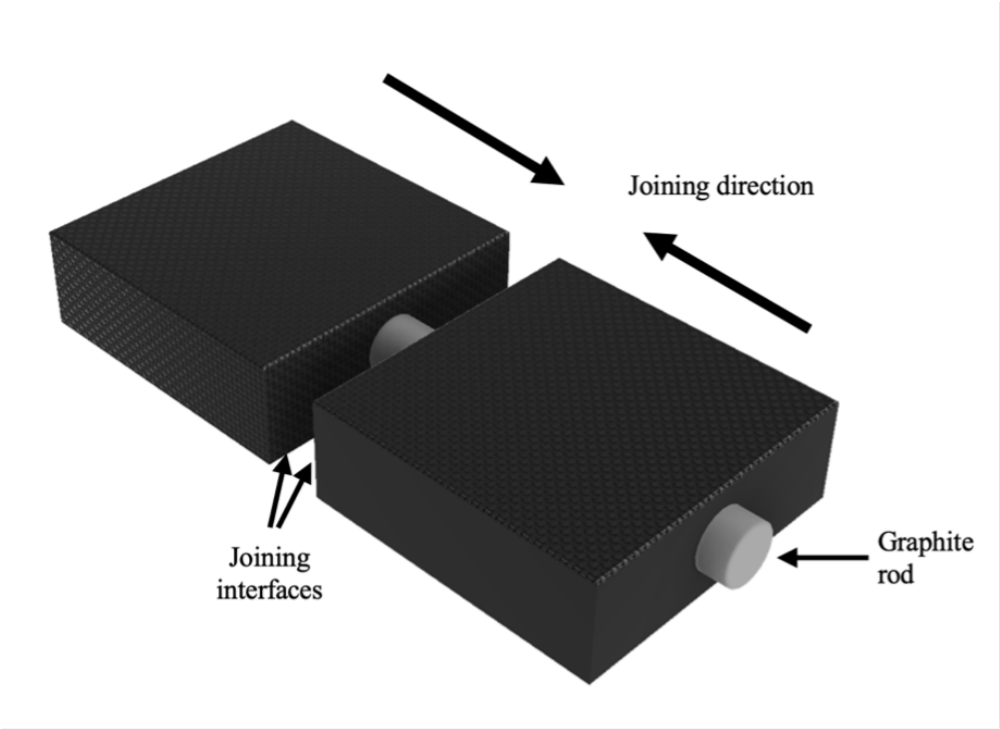


Figure 3.3: Rendering of SiC/SiC wall components with cylindrical channels in joining orientation with graphite rod. This is a rendering, manufactured SiC/SiC will likely look different.

Components and joints should be evaluated for microstructure, density, hermeticity, strength, and effects of neutron irradiation.

- Microstructure: Use SEM imaging to observe microstructures of joint interfaces. Take note of features including pores and cracks.
- Density: Use Archimedes' method to measure the volume of a component and thus evaluate density. Compare component density to theoretical density of SiC 3.22 g/cm^3 [21].
- Hermeticity: Conduct hermeticity testing by flowing pressurized helium through channels and measuring the leak rate.
- Strength: Conduct tensile and shear strengths on joints.
- Neutron irradiation: Conduct neutron irradiation testing representative of long-term fusion environments on joined components. Test again for microstructure, hermeticity, and strength to compare unirradiated and irradiated joints.

Chapter 4

Discussion and Conclusions

A review of existing literature on SiC/SiC machining, forming, and joining showed there is still much to learn about manufacturing components out of the composite material for the fusion context. The NITE and EPD processes are promising in manufacturing dense, hermetic SiC/SiC components. The layering of SiC tape, both in the NITE/EPD processes and in joining has been shown to increase the overall density and flexural strength of SiC/SiC composites.

A manufacturing plan is proposed to further SiC/SiC manufacturing capabilities for the LSVV design. Manufacturing and joining tubular components will inform designs for the LSVV SiC/SiC walls. Manufacturing a wall-like component with a tubular channel will explore designing SiC/SiC components with varying thicknesses. Testing will be required to check for hermeticity, composite and joint strength, and effects of neutron irradiation on strength and hermeticity.

The proposed manufacturing plan only considers flat components with curvature in only one direction, referring to the tubular component. The LSVV would require various degrees of curvature in multiple directions due to the toroidal shape of the tokamak and the helical liquid lead channels needed to minimize vertical disruption events [8]. SiC/SiC components will need to be manufactured and joined with varying amounts of curvature. Further work is required to develop manufacturing and joining techniques for curved components. Applying uniform pressures to components has been shown to increase the overall density and hermeticity of SiC/SiC components [40]. Methods to apply uniform pressures to curved surfaces will need to be developed. Further work is also required on the long-term effects of neutron irradiation on joints and the fatigue resistance of joints.

Questions regarding the assembly and maintenance of the SiC/SiC vacuum vessel remain. Mechanical joints are not viable due to a lack of hermeticity [29]. Joining methods including solid-state diffusion and TEP joining cannot be easily taken apart since bonding is occurring at the chemical level. Strategies for the assembly and maintenance of the LSVV which work around these constraints must be developed for the LSVV to be viable.

Looking ahead, designing a SiC/SiC vacuum vessel with helical channels on the scale of a commercial fusion reactor has significant challenges. NITE SiC/SiC is currently expensive to manufacture and is created at a small scale. Supply chains for components made from prepreg, or pre-infiltrated fabrics, are not well-established [41]. Facilities must be scaled up in order to make SiC/SiC feasible. Kyoto Fusion is constructing in "UNique Integrated Test-

ing facilitY" (UNITY), a facility in Japan with various capabilities including cost-effective manufacturing methods to scale the production of SiC/SiC components specifically for their Self-Cooled Yuryo Lithium-Lead Advanced (SCYLLA) blanket for commercial fusion reactors [42].

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