

# Light Water Reactor Sustainability Program

## Status of Silicon Carbide Joining Technology Development

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**Light Water Reactor Sustainability Program**  
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**Development**

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## **ABSTRACT**

Advanced, accident tolerant nuclear fuel systems are currently being investigated for potential application in currently operating light water reactors (LWR) or in reactors that have attained design certification. Evaluation of potential options for accident tolerant nuclear fuel systems point to the potential benefits of silicon carbide (SiC) relative to Zr-based alloys, including increased corrosion resistance, reduced oxidation and heat of oxidation, and reduced hydrogen generation under steam attack (off-normal conditions). If demonstrated to be applicable in the intended LWR environment, SiC could be used in nuclear fuel cladding or other in-core structural components. Achieving a SiC-SiC joint that resists corrosion with hot, flowing water, is stable under irradiation and retains hermeticity is a significant challenge. This report summarizes the current status of SiC-SiC joint development work supported by the Department of Energy Light Water Reactor Sustainability Program. Significant progress has been made toward SiC-SiC joint development for nuclear service, but additional development and testing work (including irradiation testing) is still required to present a candidate joint for use in nuclear fuel cladding.



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# Status of Silicon Carbide Joining Technology Development

## 1. INTRODUCTION

Advanced, accident tolerant nuclear fuel systems are currently being investigated for potential application in currently operating light water reactors (LWR) or in reactors that have attained design certification. An accident tolerant design would offer equivalent or better performance under normal operating conditions as compared to the current zirconium (Zr) alloy-uranium dioxide (UO<sub>2</sub>) fuel system, while offering increased safety in the event of accidents in the reactor or spent fuel pool. Evaluation of potential options for accident tolerant nuclear fuel systems point to the potential benefits of silicon carbide (SiC) relative to Zr-based alloys, including increased corrosion resistance, reduced oxidation and heat of oxidation, and reduced hydrogen generation under steam attack (off-normal conditions). If demonstrated to be applicable in the intended LWR environment, SiC could be used in nuclear fuel cladding or other in-core structural components. A report summarizing the SiC materials considered for LWR applications, including an estimate of their current technology development status, benefits and outstanding issues, was recently issued (Bragg-Sitton et al. 2013). Recent investigations aimed at further determining the applicability of SiC in LWR applications continue to demonstrate the benefits of SiC materials relative to Zr-based alloys. Preliminary analysis results applying the existing data on SiC properties suggest significant improvement in the reactor coping time through the use of SiC materials versus Zr-alloys under a selected loss of coolant accident (LOCA) scenario (Merrill and Bragg-Sitton 2013). However, many data and technology gaps remain, particularly for SiC composite materials.

The two primary design concepts using SiC composites that are considered for LWR cladding include fully ceramic SiC/SiC composite cladding and ceramic / metal “hybrid” cladding. The former describes a fully ceramic structure comprised of SiC materials, possibly incorporating SiC fiber reinforced ceramic matrix composite (CMC) to achieve fracture toughness (i.e., improved strength over zirconium-alloy cladding) in combination with a layer (or layers) of monolithic SiC ceramic to seal the composite structure, providing hermeticity along the length. The latter hybrid design would be comprised of a composite layer or sleeve over an inner metallic liner tube (possibly zirconium-alloy, but other metals also could be considered). Various technical, operational, economic, materials interaction and fabrication issues must be addressed for each design category. Hermeticity (impervious to gas) is a key functional requirement for any cladding design. A critical need for any technology involving silicon carbide composites is development of a reliable joining methodology that can withstand the radiation environment inherent to nuclear applications. Candidate joining technologies currently under investigation are summarized in this report.

### 1.1 Background on SiC/SiC Joining Technology

Joining SiC to SiC is a key challenge that must be resolved before silicon carbide can be used either for structural materials or nuclear fuel cladding in LWRs. The fully ceramic SiC/SiC clad and hybrid ceramic / metal cladding designs both require development of a hermetic structure and end-cap seals that can withstand the radiation, temperature and chemical environment inherent to an operating LWR. A fully ceramic cladding could incorporate silicon carbide fiber reinforced ceramic matrix composite (SiC<sub>f</sub>-CMC) to achieve fracture toughness in combination with a layer (or layers) of monolithic SiC ceramic to seal the composite structure. The end-cap seal for the fully ceramic system requires sealing of the SiC<sub>f</sub>-CMC to itself. In the hybrid design, the hermetic seal for the fuel pin is provided by the inner metal liner; end caps also are welded on the metal liner, just as they are for the standard all-metal cladding designs. Joints in non-fuel SiC components must be reliable under LWR conditions; however, they do not have the same hermeticity requirements as fuel components.

A reliable, reproducible technique to join and hermetically seal silicon carbide composites has been identified as a critical technology gap for SiC-based cladding systems. There are a number of conventional and advanced techniques to join SiC (or SiC/SiC composite) to itself or other materials (Jones, Henager, and Hollenberg 1992; Snead et al. 1996). Successfully demonstrated techniques include pre-ceramic polymer joining (Yajima et al. 1981; Donato, Colombo, and Abadirashid 1995; Lewinsohn et al. 2001), glass-ceramics (Ferraris et al. 1994), reaction bonding (Lewinsohn et al. 2001; Rabin and Moore 1993), active metal / pre-ceramic polymers (Sherwood et al. 1997), and active metal solid state displacement techniques (Halbig et al. 2006; Radhakrishnan et al. 1996; Henager et al. 2007). While the strength of the joints produced by these methods appears to be adequate for LWR applications, there is currently a lack of standards for testing ceramics (Ferraris et al. 2011, 2008) and a variety of tests have been applied to measure the strength of the bonds created using each technique.

Additionally, there currently is limited irradiation data on the joints and the materials used to fabricate the joints; many of joint fabrication techniques that have been tested under irradiation have demonstrated poor irradiation stability. A reliable SiC/SiC joining technique for reactor structural materials has yet to be developed and demonstrated (Kato et al. 2007; Nozawa et al. 2009; Ferraris et al. 2011). Given the functional requirement of hermeticity for nuclear fuel cladding that is necessary to retain helium and gaseous fission products, the SiC/SiC joining technique must be radiation stable for the relevant conditions of applied stress (specific stress condition has not yet been defined), temperature ( $\approx 400\text{--}500^\circ\text{C}$ ) and neutron damage ( $\approx 6$  dpa). Recent positive results on SiC/SiC joining technologies are included in Kato et al. (2013a) and are summarized here; additional work in the development of a reliable SiC/SiC joint is currently under way at General Atomics and at Rolls Royce High Temperature Composites via support from the DOE LWR Sustainability (LWRS) Program.

## **1.2 Overview of LWRS-Supported SiC/SiC Joint Development**

Through funds made available by the DOE LWRS Program, work was conducted in FY13 to develop SiC/SiC joint technology within the laboratory system, specifically at the Oak Ridge National Laboratory (ORNL) and via collaboration with industry. The Battelle Energy Alliance (BEA), which operates the Idaho National Laboratory (INL), sought industry support and involvement in FY12 to develop advanced processing technology to form a robust joint between a SiC CMC nuclear fuel cladding tube and its end plug. A competitive proposal process was initiated via the BEA “Expression of Interest – Light Water Reactor Sustainability Program: Silicon Carbide Ceramic Matrix Composite Nuclear Fuel Cladding,” resulting in contracts being awarded to General Atomics (GA) and Rolls-Royce High Temperature Composites (RR-HTC, formerly Hypertherm High Temperature Composites). The period of performance of the GA award was August 2012 – September 2013, while the RR-HTC award period of performance began in December 2012 and extends through September 2014.

## **1.3 Overview of Candidate Joining Methods**

Several methods of joining SiC ceramic composites are considered promising for general applications; however, not all are expected to hold promise for in-reactor applications. These methods are summarized in Table 1, along with reported strength properties and anticipated performance under irradiation. Primary considerations for nuclear applications (both fission and fusion) include resistance to neutron irradiation; mechanical properties, such as strength and reliability during mechanical loading; compatibility of the processing condition with the design requirement; chemical compatibility with the operating environment for the intended application; and the ability to satisfy the hermeticity requirement. Reliable joining has been identified as the most significant technology gap / challenge for SiC-CMCs as a cladding material.

Ongoing work on SiC/SiC joint development has encouraged strong communications between the various institutions developing joint technology to ensure commonality of test techniques for future comparison of test results. Additionally, the LWR Sustainability Program supports involvement of ORNL

SiC researchers in the ASTM committees tasked with developing ASTM standards for ceramic composites intended for nuclear service.

Table 1. Methods for joining SiC-based materials (Katoh et al. 2013a).

<b>Joining Method</b>	<b>Typical Reported Strength</b>	<b>Irradiation Performance</b>	<b>References</b> (see Katoh et al. 2013a or Bragg-Sitton et al. 2013 for detailed references)
Metal diffusion bonding	≈150 MPa shear	Expectedly good	Halbig et al. 2006; Cockeram 2005
Transient eutectic-phase joining	≈250 MPa tensile	Expectedly good	Ferraris et al. 2011; Hinoki et al. 2005; Katoh, Dong, and Kohyama 2002
Glass-ceramic joining	≈100 MPa shear	Positive result reported (European Union program)	Ferraris et al. 2008, 2011, 2012; Mailliat et al. 2008; Coghlan et al. 1986; Katoh et al. 2000
Brazing	Various	Generally poor; high activation	Raffray et al. 2001; Mailliat et al. 2008; Chaumat and Coing-Boyat 2001; Gasse, Coing-Boyat, and Bourgeois 2001; Gasse 2003; Gasse, Chaumat and Saint-Antonin 1997
SiC reaction bonding	≈200 MPa shear	Expectedly unstable	Herderick, Cooper, and Ames 2012; Corelli et al. 1983; Matthews 1974; Matheny and Corelli 1979
MAX-phase joining <sup>a</sup>	≈100 MPa shear	Unknown	Radhakrishnan et al. 1996; Henager and Kurtz 2011; Henager et al. 2007; Henager, Brimhall, and Brush 1995; Colombo et al. 2000
Pre-ceramic polymer joining	Tens MPa shear	Expectedly unstable	Henager et al. 2007; Colombo et al. 2000; Lewinsohn et al. 2002; Kleebe and Blum 2008; Blum, MacQueen, and Kleebe 2005; Modena et al. 2005
Transient liquid metal joining	No data	Unknown	—
Selective area CVD	No data	Expectedly very good	Lippmann et al. 2004

a. MAX-phase =  $M_{n+1}AX_n$  phase ceramic composition

## 2. Status of SiC Joining Technology

As discussed above, three research efforts have been supported by the LWRS program in FY13 (via funds made available in FY12). These projects are led by ORNL, General Atomics and Rolls-Royce High Temperature Composites. The current status of each of these development programs is summarized below.

### 2.1 ORNL Joint Development

Various mechanical testing methods for assessment of the SiC/SiC joint were evaluated and compared at ORNL. Neutron irradiation experiments of selected joints and irradiation effects on joint microstructure and strength have been conducted, as summarized in Katoh et al. (2013a). Several joint methods showed no strength degradation at intermediate fluence levels ( $\approx 7$  dpa) at temperatures of interest for LWR systems. Detailed joint evaluation results are included in Katoh et al. (2013a). ORNL continues to develop joint strength test methods that are compatible with the utilization of small specimens, which is preferred for neutron irradiation experiments.

Candidate joining technologies recently tested by ORNL were limited to those joints that provide low induced radioactivity and include: titanium diffusion bonding; Ti-Si-C Mn+1AX<sub>n</sub> phase ceramic composition (MAX-phase) joining; calcia-alumina glass-ceramic joining; and transient eutectic phase SiC joining. The methods and the development status of the SiC joining studied at ORNL in support of the LWRS Fuels Program are listed in Table 2.

Table 2. Status summary of SiC joint processing in the present work.

<b>Method</b>	<b>Insert Material</b>	<b>Status</b>
<b>Metallic diffusion bonding</b>	Ti foil	Optimum joining condition established
	Mo foil	Optimum joining condition established
<b>MAX-phase bonding</b>	Ti-Si-C slurry	Successful with commercial joining kit
<b>Transient eutectic-phase joining</b>	NITE* slurry	Optimum joining condition being studied

\*NITE: Nano-Infiltration and Transient Eutectic-phase process for sintering SiC

The processing parameters including hot pressing temperature, pressure, time, and joining material thickness were selected for each joint type based on thorough literature review. Multiple joining conditions were attempted for each joining material in order to determine the optimal processing conditions to be used for fabricating joint specimens for the planned irradiation behavior study. Table 3 lists the selected processing conditions, shear strengths, joint microstructures and fracture behaviors observed in the present study. Images of each type of bonding joint (as-fabricated) are provided in Figures 1-4.

Table 3. Processing conditions, shear strengths, joint microstructures and fracture behaviors of CVD-silicon carbide joined by different joining materials.

Insert materials	Specimen ID	Temp. (°C)	Press. (MPa)	Time (h)	Shear Strength (MPa)	S.D. (MPa)	Phases	Joint Appearance	Failure Location
Ti foil	Tif-1300-1	1300	20	1	1.7		Ti <sub>5</sub> Si <sub>3</sub> , TiC	Ti <sub>5</sub> Si <sub>3</sub> /SiC debonding	Interface
	Tif-1500-1	1500	15	1	97.6	8.8	85.1% TiC, Ti <sub>3</sub> SiC <sub>2</sub>	Intact	Through joint
	Tif-1500-5	1500	20	5	28.4	13.7	Ti <sub>3</sub> SiC <sub>2</sub>	Intact	Mix
Mo foil	Mof-1300-1	1300	20	1	46.4	30.6	Mo <sub>2</sub> C, Mo <sub>5</sub> Si <sub>3</sub>	Porous interface	Interface
	Mof-1300-5	1300	20	5	15.2	7.2	Mo <sub>2</sub> C, Mo <sub>5</sub> Si <sub>3</sub>	Intact	Interface and base
	Mof-1500-1	1500	15	1	55.2	15.4	Mo <sub>2</sub> C, Mo <sub>5</sub> Si <sub>3</sub>	Intact	Through joint
	Mof-1500-1-Ti <sup>1</sup>	1500	15	1	83.1	41.7	Mo <sub>2</sub> C, Mo <sub>5</sub> Si <sub>3</sub>	Intact	Mostly base
	Mof-1500-1-H <sub>2</sub> <sup>2</sup>	1500	15	1	105.5	4.6	Mo <sub>2</sub> C, Mo <sub>5</sub> Si <sub>3</sub>	Intact	Base
	Mof-1500-5	1500	20	5	18.8	11.2	Mo <sub>2</sub> C, Mo <sub>5</sub> Si <sub>3</sub>	Partially debonding	Primarily at base
	Mof-1700-1	1700	20	1	68.2	18.7	Mo <sub>2</sub> C, Mo <sub>5</sub> Si <sub>3</sub>	Intact	Mostly base
	Mof-1700-1-H <sub>2</sub> <sup>2</sup>	1700	20	1	94	20.6	Mo <sub>2</sub> C, Mo <sub>5</sub> Si <sub>3</sub>	Intact	Mostly base
HHTC MAX	HMX	N/D <sup>3</sup>	N/D	N/D	69.9	6.3	N/D	Intact	Interface
NITE slurry	NITEs-1860-1	1860	20	1	43.9	38.8	SiC	Porous/dense joint, debonding at porous joint	Interface
NITE spray	NITEc-1875-1	1875	20	1	67.3	33.2	SiC	Porous/dense joint, debonding at porous joint	Interface

1, 2: Suffix Ti indicates that titanium powder was used as an oxygen getter in vacuum environment. Suffix H<sub>2</sub> indicates that hydrogen was added to flowing argon atmosphere. Other processing runs were performed in vacuum for metal diffusion bonding. NITE processing runs were performed in an argon flow.

3: not disclosed

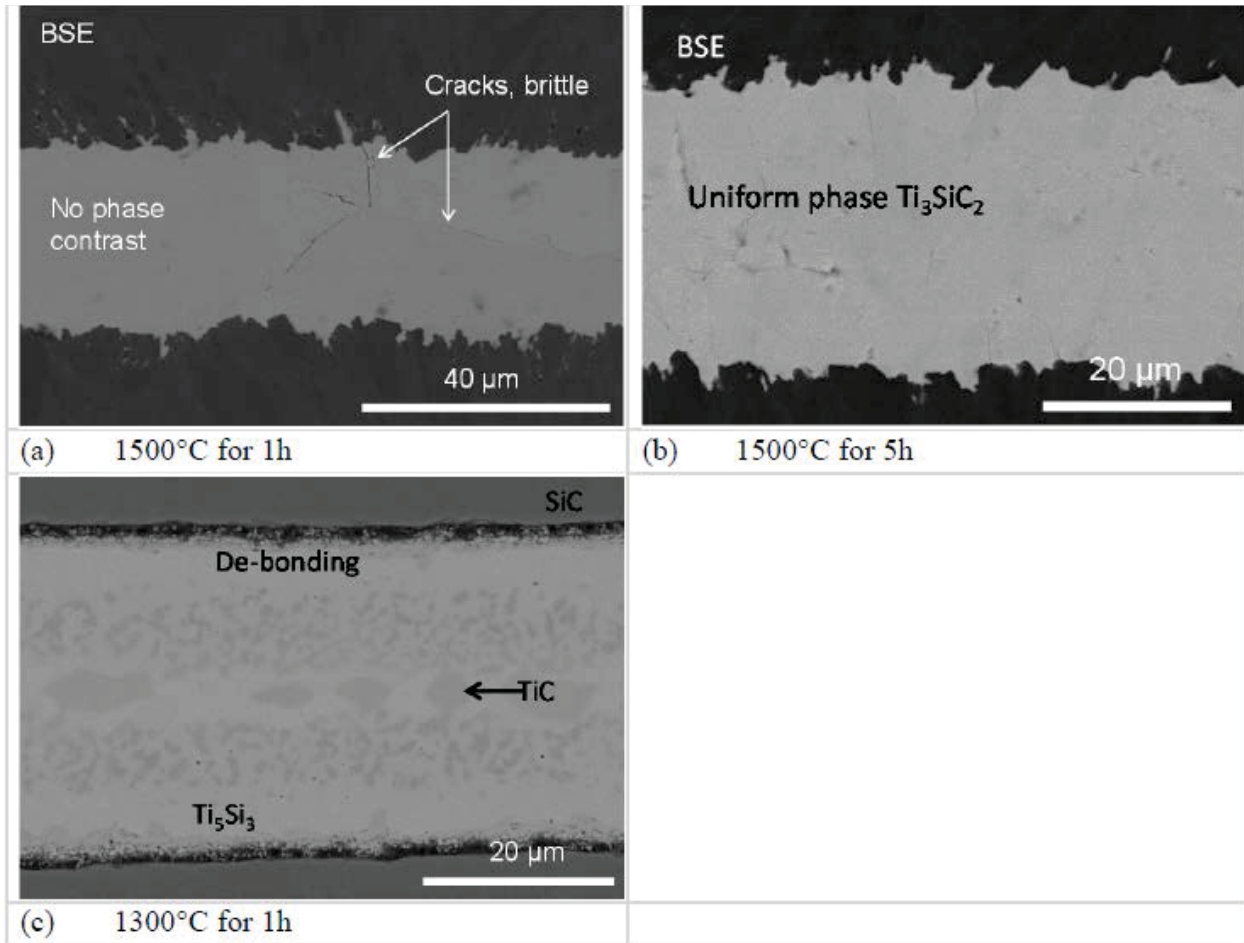


Figure 1. Back Scattering Electron (BSE) images of Ti diffusion-bonded joints.

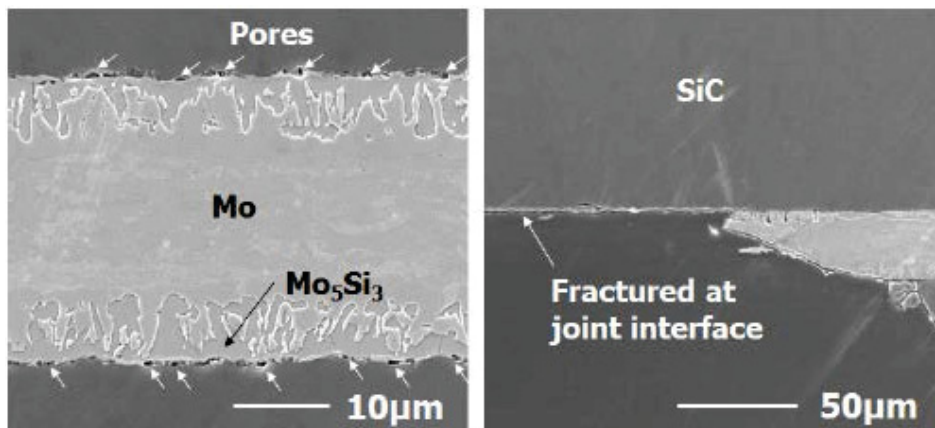


Figure 2. Secondary Electron (SE) images of Mo foil joint hot pressed at 1300 °C for one hour: (left) joint interface before double-notch shear (DNS) test and (b) joint interface after DNS test.

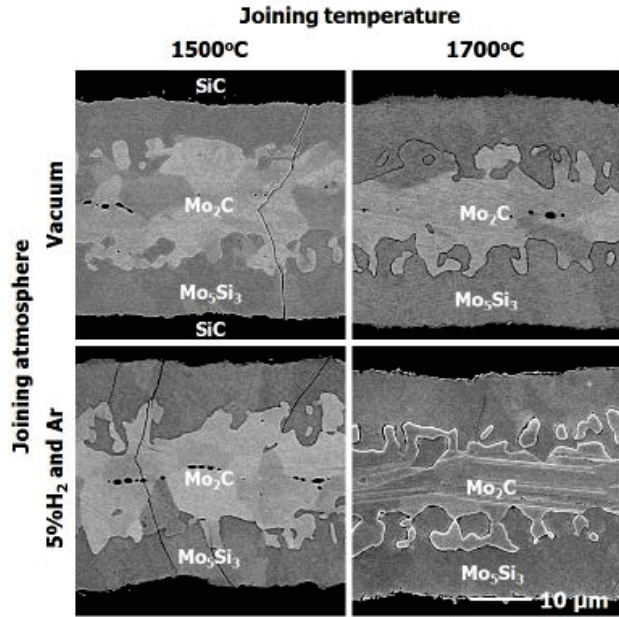


Figure 3. BSE images of Mo joints formed at 1500 and 1700°C, with and without oxygen eliminator.

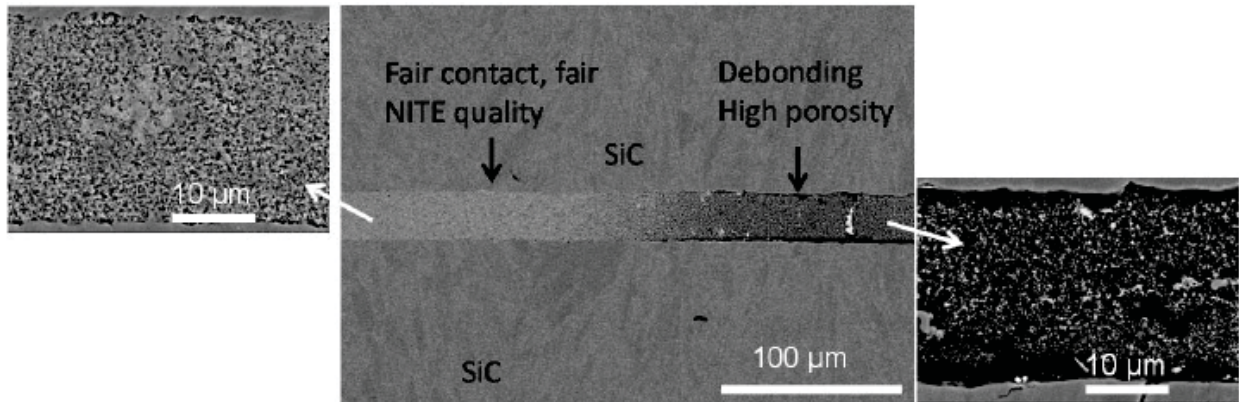


Figure 4. BSE images of NITE slurry joint hot pressed at 1860°C for one hour.

Samples of joints have been irradiated in HFIR up to  $\approx 5$  dpa at 500 or 800°C. Analysis of the results indicates that their microstructure and mechanical properties compared to pre-irradiation conditions. This irradiation was supported by the DOE Office of Science Fusion Materials Program; therefore, the irradiation temperatures are somewhat higher than what is of interest to normal LWR operation and may not provide a conservative estimate of performance under LWR conditions (some defects may anneal out at elevated temperature). Within the limitations of statistics, all joining methodologies presented retained their joint mechanical strength. However, the higher irradiation temperature may not provide a conservative estimate of performance under LWR conditions, because radiation damage in ceramics is often more severe at lower temperatures. This work provides the first positive results for irradiation-stable SiC joints. For the higher temperature irradiation conditions (800°C,  $\approx 5$  dpa) some joint materials exhibited significant irradiation-induced microstructural evolution; however, the effect of irradiation on joint strength appeared rather limited. Joints currently being developed at GA and RR-HTC should be subjected to similar testing to allow down-selection and optimization of the joint technology.

In summary, ORNL researchers applied various methods to prepare joints of SiC ceramics and composites for evaluation of torsional shear strength, microstructures, and the effects of neutron

irradiation at elevated temperatures. All methods produced strong joints, having unirradiated torsional shear strength  $\approx 100$  MPa or higher. The primary conclusions drawn from the ORNL work are summarized below.

1. Diffusion bonding with a Ti active insert resulted in a CVD SiC joint with reasonable shear strength when  $\text{Ti}_3\text{SiC}_2/\text{Ti}_5\text{Si}_3$  layered microstructure is obtained. The effect of irradiation at  $500^\circ\text{C}$  to  $\approx 3$  dpa appeared insignificant.
2. Transient eutectic-phase (TEP) joining using SiC- $\text{Al}_2\text{O}_3$ - $\text{Y}_2\text{O}_3$ -based slurry produced very robust joints of CVD SiC, NITE-like sintered SiC, and NITE SiC/SiC composite. Joining process-induced damage to the composite integrity was identified a potential concern. The effect of irradiation at  $500^\circ\text{C}$  to  $\approx 3$  dpa appeared insignificant.
3. TEP joining using a commercial SiC- $\text{Al}_2\text{O}_3$ - $\text{Y}_2\text{O}_3$ -based green tape produced robust joints of CVD SiC and NITE-like sintered SiC. The joint strength is likely limited by a porous microstructure within the joint plane. The effect of irradiation at  $500^\circ\text{C}$  to  $\approx 3$  dpa or at  $800^\circ\text{C}$  to  $\approx 5$  dpa appeared insignificant or minor.
4. CaO-  $\text{Al}_2\text{O}_3$  glass-ceramic-joined CVD SiC exhibited reasonable shear strength. The effect of neutron irradiation on shear strength appeared minor or insignificant. Microstructural features remain unaltered after irradiation at  $500^\circ\text{C}$  to  $\approx 3$  dpa, whereas the  $800^\circ\text{C}$  irradiation induced observable microstructural evolution.

A complete summary of the ORNL joint development status is included in Katoh, et al. 2013b. In addition to fabricating SiC joint specimens, a rabbit capsule has been designed and fabricated for joint irradiation in the ORNL High Flux Isotope Reactor (HFIR). Materials being considered for the planned irradiation study are summarized in Table 4. It is expected that a portion of the irradiation test will be covered via carry over funds in the LWRs Fuels program, which will then be supplemented by other SiC development efforts.

Table 4. Materials considered for HFIR SiC-SiC joint irradiation study.

<b>Material description</b>	<b>Supplier</b>
General Atomics Proprietary, Polymer/CVD Hybrid	General Atomics
Rolls-Royce HTC Proprietary, Pressureless MAX-phase	Rolls-Royce HTC
Ti Diffusion Bonding	ORNL
Mo Diffusion Bonding	ORNL
NITE, Slurry*	Kyoto University
NITE, Green Tape*	Kyoto University
Calcia-Alumina Glass-Ceramics*	Politecnico di Torino
MAX-phase, Green Tape*	PNNL <sup>1</sup>
Metallic Braze*	Ceramatec
NITE, spray coating*	ORNL
NITE, screen printing*	ORNL
NITE, Pressureless Sintering*	ORNL

\*Final test matrix will be determined based on availability of these samples.

1: Pacific Northwest National Laboratory



## 2.2 General Atomics Joint Development

General Atomics performed SiC composite joining work under a one-year contract with the Battelle Energy Alliance (BEA, which operates the Idaho National Laboratory). The objective of the GA work was to advance the technological readiness of SiC-SiC composite materials for nuclear reactor applications. The ultimate goal set out in this contract was to join an endplug to a SiC-SiC composite tube. Note that only a summary of the GA-led work is included here. Complete details are provided in the GA Final Report, *Technology for Joining Silicon Carbide Ceramic Matrix Composites for Nuclear Fuel Cladding* (this report was not yet available at the time this status summary was written, but is expected to be released in late September / early October 2013). *Many of the details that follow were extracted directly from the GA Final Report.*

The GA work progressed in three overlapping, increasingly complex phases: planar joints, cylindrical joints, and cylindrical joints between a monolithic endplug and a SiC-SiC composite tube. These studies use joint material developed previously by General Atomics (without DOE funding). Activities occurred in all three task areas in accordance with the schedule given in the proposal. The GA work was divided into three task areas, as summarized below. The key accomplishments in each of these areas are summarized.

### 2.2.1 Task I

Task I of this project was a scoping study on planar joints to make a comparative study of joint designs. Although the ultimate application entails joining tubular SiC-SiC composite components, the variation on planar testing provides unambiguous information on the effect of varying joint length and joint geometry.

*Key Task I accomplishments:*

1. Planar joints of four geometries were prepared and fabricated (Figure 5). Twenty-three joints were mechanically tested. Based on the test results, the most promising joint geometry was identified and targeted for the continued cylindrical geometry testing.
2. Helium permeability test rig were constructed and validated for planar samples.

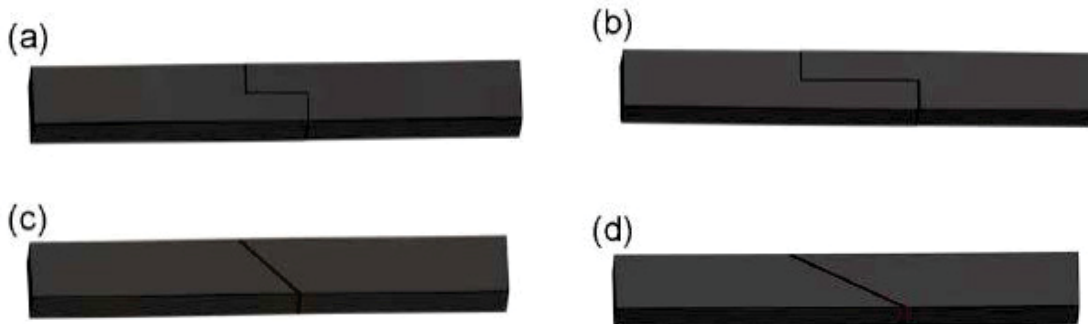


Figure 5. The different double-butt lap (DBL) and scarf joint geometries evaluated with 4-point bend flexural testing: (a) L1-5 mm DBL, (b) L2-10 mm DBL, (c) S1-5 mm scarf (45°), and (d) S2-10 mm scarf (26.5°).

### 2.2.2 Task II

Task II was focused on development of a fabrication method for monolithic  $\beta$ -SiC endplugs, initiation of the transition of joint processing to tube-endplug assemblies, and development of test rigs for evaluating mechanical and permeability performance of joined tube-endplug assemblies. The completion of Task II established the capability to reliably join cylindrical components in the form of monolithic SiC endplugs and monolithic SiC tubes. Specialized test rigs were constructed for the purpose of explicitly

evaluating mechanical and leak rate performance of these joined components. To safely and successfully deploy any joint in a LWR, the joint must be able to retain fission product gases and sustain peak internal pressurization of 2250 psi. The leak rate requirement established for a viable joint is  $3 \times 10^{-8}$  atm-cc/sec at 300°C. The load requirement established for a viable joint is 1700N, which corresponds to 5625 psi, or a 2.5 safety factor.

The data collected in Task II indicate that two candidate joint geometries are capable of meeting these requirements: the scarf and butted scarf geometries. The scarf geometry is comparably easier to fabricate, but requires additional SiC overcoating to meet the leak rate requirement. The butted scarf geometry is more complex to fabricate, but achieves suitably low leak rates with comparably less SiC overcoating. Both endplug geometries present viable solutions to the problem of nuclear grade joining; therefore, both were applied in the final phase of this work, fabricating and testing joined cylindrical components with SiC-SiC composite cladding.

The first joined tubular components revealed some critical differences between the initial endplug geometry types being pursued. Namely, geometries featuring tapers, or angles, promote better interfacial joint contact compared to lap joints, or joint geometries with orthogonal joining surfaces. Cross sectional micrographs of a scarf joint and a butted lap joint in cylindrical specimens are presented in Figure 6. The improved performance arises from the fact that angled features are amenable to uniaxial loading of the joined components during polymer processing, thus preventing volumetric shrinkage induced damage such as interfacial separation. Conversely, the butted lap joint shows significant interfacial separation in the lap region of the joint where load application is not feasible because it would require radial load applied on the cladding tube. Due to the inability to apply radial load in the butted lap geometry, it was dropped as a candidate endplug geometry in this study.

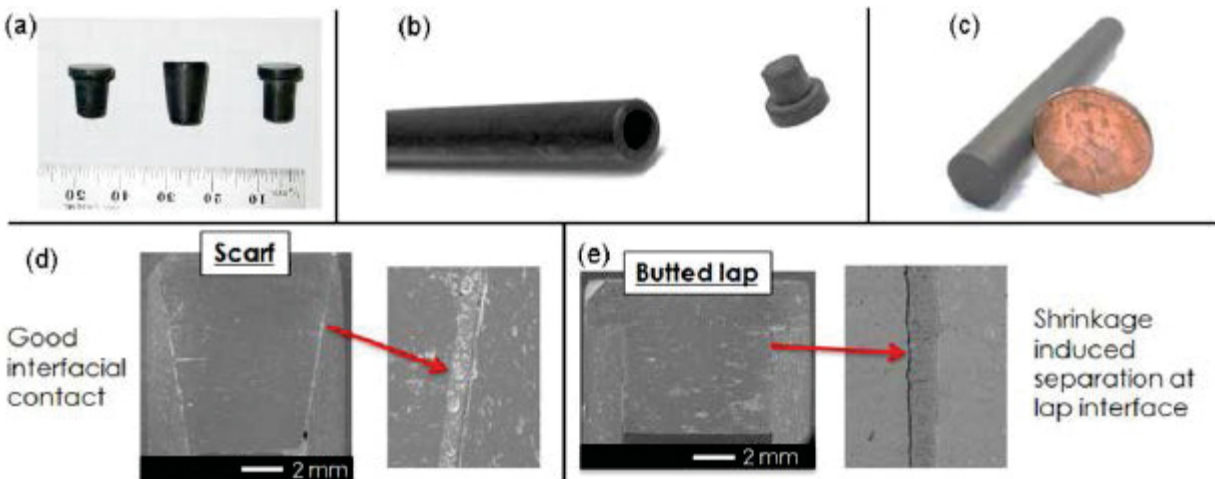


Figure 6. Comparison of endplug joint geometries for cylindrical joint assemblies: (a) as fabricated monolithic SiC butted scarf (left), scarf (middle), and butted lap endplugs, (b) butted lap endplug and mating monolithic SiC tube, prior to joining, (c) joined cylindrical assembly, (d) cross-sectional micrograph of scarf-type endplug joint with high magnification inset of joint layer showing good interfacial contact, and (e) cross-sectional micrograph of butted lap-type endplug joint with high magnification inset of joint layer showing poor interfacial contact in lap (vertical) region of joint due to volumetric shrinkage-induced separation.

Final selection of a joint design must take into account leak rate performance, fabrication time, fabrication cost, and mechanical performance (e.g. strength). The scarf and butted scarf geometries were found to be the two most viable candidates. While the scarf endplug and associated joint preparation is

more straightforward, this approach may require additional processing to meet the leak rate requirement. Conversely, the butted scarf endplug and joint preparation can deliver suitably low leak rates with less SiC overcoating, but entails a considerably more complex and geometrically constrained preparation. Either are suitably robust joining geometries that meet the permeability requirement.

*Key Task II accomplishments:*

1. Endplugs were fabricated in four geometries. Butt, butted lap, scarf and butted scarf geometries were evaluated.
2. Joined monolithic SiC tubular specimens have been fabricated with the aforementioned endplug geometries and subjected to mechanical and permeability testing. Based on these results both scarf and butted scarf geometries were identified as viable solutions for achieving strong, low permeability joints.
3. Planar and tubular joint specimens have achieved He leak rates surpassing the design specification of  $4 \times 10^{-9}$  atm\*cc/sec.
4. An endplug pushout test fixture was constructed to simplify mechanical testing of joined cylindrical components. This test is also adaptable to elevated temperature testing.
5. Joined monolithic SiC tubular specimens with scarf and butted scarf endplug geometries demonstrated endplug pushout failure loads in excess of 1.7 kN, thus meeting the requirement for a 2250 psi internal pressurization at end of life with a safety margin of 2.5.
6. Hydrostatic burst test fixtures have been assembled and validated using stainless steel and SiC monolithic tubes. This involved the identification of appropriate bond epoxy for the tests.

### **2.2.3 Task III**

The final task of this contract entailed fabrication of joints between monolithic SiC endplugs and SiC-SiC composite tubes. All of the information gleaned from Tasks I and II directly apply to the objectives of this final phase. The joint material and geometries, the characterization methods used to test and validate these specimens, and the performance requirements these specimens must meet are nominally the same. The singular difference between this task and prior work is the transition to SiC-SiC composite cladding. The fiber architecture residual porosity in SiC-SiC composites has the potential to impact joint integrity and performance.

*Key Task III accomplishments:*

1. SiC-SiC composite tubes were fabricated via chemical vapor infiltration for the final joined SiC-SiC composite components. This includes acquiring fiber, making associated fixtures, producing preforms of the nominal Light Water Reactor dimensions, and infiltrating the preforms in GA's composite synthesis facility.
2. Joined SiC-SiC composite tubular specimens have been fabricated with endplugs of the butted scarf and scarf geometries (Figure 6).
3. Joined composite assemblies achieved steady state helium leak rates surpassing the design specification of  $4 \times 10^{-9}$  atm\*cc/sec with either scarf or butted scarf joint geometries.
4. Joined composite SiC tubular specimens with scarf and butted scarf endplug geometries demonstrated endplug pushout failure loads in excess of 1.7 kN, thus meeting the requirement for a 2250 psi internal pressurization at end of life with a safety margin of 2.5.
5. A joined composite assembly exhibited no degradation in leak rate or mechanical performance after being thermally cycled to 1000oC and subsequently pre-loaded to 2250 psi equivalent.

The culmination of endplug pushout testing has generated compelling evidence that the joint material and joint geometries evaluated in this program are robust, resilient, and capable of enduring the loads and conditions expected during LWR operation. Furthermore, the testing has established that this new joining technology is applicable to both monolithic and composite components. In fact, the deployment of this joining technology is more robust when applied to composite specimens due to the impregnation of residual composite void space by the joint material. The results compiled in this program represent a positive step for pre-ceramic polymer derived joining.

It should be noted that improvements can still be made in joint processing and specimen preparation. A limited number of specimens were tested in this program. With each new sample set fabricated during the program, significant improvements in mechanical integrity were observed.

The objectives of the contracted GA research program have been achieved. A robust, impermeable joint between SiC components has been developed, and this joint has demonstrated leak rates and failure loads exceeding the requirements imposed on existing LWR components.

#### **2.2.4 GA Conclusions**

The primary outcome of this one year contract was the successful fabrication of joined cylindrical SiC-SiC composite tubes exhibiting ample strength and permeability performance to meet LWR design specifications. Deliverables prepared for the contract include 10 monolithic SiC tube-endplug joint assemblies for independent testing and five assemblies of SiC-SiC composite joined to a monolithic SiC endplug for ultimate irradiation testing. During this work period, studies identified critical joint lengths, and joint geometries to produce a robust joint. To ensure ample strength, a joint length exceeding 9 mm and having a scarf geometry must be used. Furthermore, specialized test rigs were designed and built to characterize the critical parameters used to evaluate strength and permeability.

These studies show that the GA polymer derived joining technique and the joint geometries identified in this study are capable of supplying a suitably robust joint. The next steps to develop the technology and improve on joint performance include expanding the data set, developing a more instrumented and expanded endplug pushout test, and conducting different test types to independently characterize the measured properties. While the measurements here are compelling, statistically relevant data sets are needed to draw more global comparisons to alternative technologies. Additional irradiation testing must also be conducted to verify joint robustness in the intended LWR operating environment.

Once the full technology has been developed, final deployment of this technology necessitates the joining of SiC components after fuel has been inserted into the rod. Furthermore, a complete LWR core will require thousands of such joints to be fabricated in an economical and scalable process. While the methods involved in the joining process developed in this program are scalable, it has yet to be done in practice.

## 2.3 Rolls-Royce High Temperature Composites Joint Development

The objective of the RR-HTC effort is to evaluate joining technology for bonding end caps onto SiC composite fuel cladding. Program milestones include demonstration of an impermeable joint using materials that are either known to be stable under the irradiation environment (SiC) or are currently planned for investigation due to their promise (MAX phase). Joining will be conducted at temperatures that are compatible with Nuclear Grade SiC/SiC composites, whether reinforced with Tyranno SA or Hi-Nicalon Type S fibers. Preliminary testing will be performed to demonstrate applicability under accident scenarios. This effort is divided into six tasks related to the design, fabrication, and testing of Ceramic Matrix Composite components.

### 2.3.1 Planned Work Scope

The overarching objective of the RR-HTC effort is to apply a demonstrated robust joining technology to bonding of end caps onto SiC composite fuel cladding. The critical factors that would be examined in a feasibility study include demonstration of an impermeable joint using materials that are either known to be stable under the irradiation environment (SiC) or are currently planned for investigation due to their promise (MAX phase). Joining will be conducted at temperatures that are compatible with Nuclear Grade SiC/SiC composites whether reinforced with Tyranno SA or Hi-Nicalon Type S fibers. Preliminary tests will be performed to demonstrate the stability under accident scenarios.

Key tasks to be addressed by RR-HTC are listed below. Complete details of these tasks are included in INL SOW-10595, *Robust Joining of SiC for Nuclear Service*. Evaluations will include mechanical, microstructural and hermeticity measurements for tasks 2 and 3. These tasks will nominally run sequentially in that joint design will need to be conducted prior to bonding studies with the candidate materials and evaluations. After these tasks are complete, a down-selection will be made with regard to bonding and sections of SiC fuel cladding will have end close-outs applied and evaluated. This down-select will provide an opportunity for a redesign of the joint geometry if needed based on assumed strengths and performance during the preliminary evaluations.

**Task 1.** Perform analysis of optimal joint design for end cap joining on SiC/SiC composite fuel cladding tubing and its end sealing plug.

**Task 2:** Preliminary screening of adhesive joining using both SiC and MAX bonding.

**Task 3:** Downselection of the preferred bonding method(s).

**Task 4:** Hydraulic pressure burst testing of bonded end caps and tubes.

**Task 5:** Fabrication of SiC/SiC fuel cladding with bonded end caps for testing at BEA facilities.

**Task 6:** [Deleted in final work scope]

**Task 7:** Demonstration of bonded joint NDE.

**Task 8:** [Deleted in final work scope]

With regard to joint design, specific objectives include suitable design of the joint to maximize strength and minimize peel stresses such that a mechanically robust joint with the highest integrity can be produced from the bond strength of the joining material. Additionally, the targeted joining approach will examine a belt and suspenders approach much as has been established for the SiC composite fuel cladding tubes. That is, a monolithic pressure boundary, in this case adhesive bonded joint, that is backed by a SiC matrix composite that can withstand the entire pressure requirements of the application structurally, but

may not have the desired impermeability. The incorporation of a SiC matrix composite into the joint provides a degree of damage tolerance to the joined region. This is a classic leak before break approach that is good safe design practice for pressure containing structures.

### 2.3.2 Current Status

Task 1, which consists of evaluating fuel cladding joint designs, defining test coupons to evaluate material design parameters, has been completed, and a test matrix for Task 2 has been outlined. Mechanical design of the fuel cladding joint has been completed; including pin sizing/location and further analysis incorporating mechanical stresses resulting from thermal expansion. Pin sizing and location for a pinned and bonded joint was determined using closed form solutions for pin shear, shear-out, net-tension, and bearing stresses. While the center-to-edge distance was limited due to geometric constraints of the plug, the pin diameter was optimized for both a shear pin and a shear-out failure. A catastrophic failure may be a result of either failure mode, and thus the margin of safety is determined by the minimum of either. Mechanical stress due to thermal expansion was also analyzed using finite element analysis, where it was determined that the expansion would place compression on the bond, thus increasing its strength in the shear direction.

The mechanical test fixture design to be used in Task 2 has been machined and received, a procedure was written for the test evaluation and equipment and measurement instrument calibration and traceability has been verified. Initial mechanical testing has been conducted for a MAX phase bonded sample. MAX phase bonds will initially be of the composition previously used at Hyper-Therm HTC.

Initial trials for establishing a low temperature LPS SiC bond have been performed. The trials indicate that a minimum temperature of 1600°C is necessary to achieve densification by LPS. A composition was identified that produced densified SiC at 89% of theoretical density with 6% open porosity. Bonds produced with this composition were measured to have a 6.4 ksi average shear strength in double notch shear. Such a strength is adequate for fuel cladding end cap bonding if the scatter in strength is low. Overall, the SiC bond has a much better stress distribution, with lower maximum stress in both radial and hoop stress configurations. The assumed modulus and densities of the SiC bond more closely match the adherends, and are theorized to be an overall benefit to the maximum stress levels. To achieve this lower stress state, a high density/lower porosity SiC bond is most desirable to achieve the preferred modulus matching. Initial trials for establishing a low temperature LPS SiC bond have been performed.

The two triaxially braided fiber architectures that had been identified have been produced as two layer braids over the monolithic SiC impermeable pressure boundary. Fabrication of the SiC/SiC fuel cladding segments by overbraiding a monolithic SiC tube with a nuclear grade SiC fiber, Hi-Nicalon Type S, was selected. A nuclear grade SiC/SiC composite containing the nuclear grade SiC fiber is then formed. This design is believed to combine the best of a monolithic tube (impermeability), with the toughness and non-catastrophic failure of a composite. CVI SiC densification of sixteen (16), 300-mm fuel cladding segments with two fiber architectures is complete. These segments will be used for bonding under Task 3 and Task 4. Physical properties of the fuel cladding segments are shown in Table 5.

Table 5. Basic Properties of Compound Cladding Tubes

Braid Type	$\rho$ Monolithic (g/cc)	$\rho$ Composite (g/cc)	Fiber Volume (%)	Porosity (%)
Lower end count	3.05	3.06	19.9	3.1
Higher end count	3.05	2.96	19.6	6.1

Figure 7 shows the sixteen 300-mm long segments. Plugs (not yet removed in the picture) were placed in the fuel cladding during CVI densification to prevent deposition of the fiber coating and CVI

SiC onto the bonding surface of the monolithic inner liner. These segments will be machined into shorter lengths for the bonding trials, testing, and initial INL deliverables. Figure 8 shows details of the two different braid architectures produced.

Design of the fiber architecture for the pinned joint configuration has been completed. Small diameter nuclear grade SiC/SiC pins are being produced from a triaxially braided architecture that has been shown to have failure strengths in excess of 700 MPa. A means for inserting these through the fuel cladding and the end cap must still be verified with regard to manufacturability. The current planned approach is individual single point drilling of the subcomponents.



Figure 7. Nuclear grade SiC/SiC fuel clad segments produced for bonding studies, pressure testing and initial INL deliverables.



(a)

(b)

Figure 8. SiC braid architectures produced by RR-HTC: (a) lower end count braid architecture with greater bias angle on braid; (b) higher end count braid architecture with lower bias angle on braid. Architecture (b) has greater total axial reinforcement as well due to the higher end count.

RR-HTC currently plans to accelerate MAX phase and LPS SiC bonding of end caps. This work will entail additional mechanical testing for Task 2, including refinement of the test protocol. Verification of the manufacturability and required tolerances for the pinned joint approach is also planned.

### 2.3.3 RR-HTC Contractual Issues

The contract for this work, which extends through September 18, 2014, was originally established between BEA Hyper-Therm High Temperature Composites, considered a small business by the US government. Hence, the contractual provisions for small businesses applied. Rolls-Royce North America Inc. (RRNA) recently purchased all shares of Hyper-Therm High-Temperature Composites Inc., effective May 1, 2013. The company was subsequently officially renamed Rolls-Royce High Temperature Composites Inc. (RRHTC) and is a wholly owned subsidiary of RRNA. RRNA is a subsidiary of Rolls-Royce North American Holdings Inc., which operates under a Special Security Agreement (SSA) as a fully owned company of the UK company Rolls-Royce plc.

Upon acquisition by RRNA and renaming to RRHTC, the provisions specific to small businesses no longer apply. RRHTC has expressed some concern about the change in intellectual property (IP) rights from the existing set for a small businesses and the new set for large businesses. Rolls-Royce has an interest in maintaining patent rights to CMC bonding techniques because they apply to a range of markets beyond nuclear. The BEA legal office has been put into contact with RRHTC legal office to discuss the details of the contract provisions. Exception to the provisions must be requested directly from the Department of Energy, as the clauses within the contract agreement flow down from DOE to BEA.

Rolls-Royce is in the process of making significant investments (via company funding) at RRHTC that will provide benefits to the current work for BEA/INL and other customers. In 2013 a range of capital equipment will be installed to expand RRHTC capabilities. The company intends to apply an advanced thermography system for non-destructive evaluation (NDE) of ceramic matrix composites (CMCs) and bonded joints. The company will also apply a blue light non-contact dimensional characterization system and a laser microscope for CMC and bond evaluation. Rolls-Royce will also be expanding the existing mechanical testing capability that may be beneficial for future work.

### 3. Conclusions and Path Forward

Achieving a SiC-SiC joint that resists corrosion with hot, flowing water, is stable under irradiation and retains hermeticity is a significant challenge. Significant progress has been made toward SiC-SiC joint development for nuclear service, but additional development and testing work is required to present a candidate joint for use in nuclear fuel cladding. Preliminary irradiation testing of SiC-SiC joint samples is scheduled to begin in FY14 at the ORNL HFIR facility.

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