INL/EXT-05-00652

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Idaho National
Laboratory

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ABSTRACT

A research program has been established to investigate fiber reinforced ceramic composites to be used as control rod components within a Very High Temperature Reactor. Two candidate systems have been identified, carbon fiber reinforced carbon (C_f/C) and silicon carbide fiber reinforced silicon carbide (SiC_f/SiC) composites. Initial irradiation stability studies to determine the maximum dose for each composite type have been initiated within the High Flux Isotope Reactor at Oak Ridge National Laboratory. Test samples exposed to 10 dpa irradiation dose have been completed with future samples to dose levels of 20 and 30 dpa scheduled for completion in following years. Mechanical and environmental testing is being conducted concurrently at the Idaho National Laboratory and at Pacific Northwest National Laboratory. High temperature test equipment, testing methodologies, and test samples for high temperature (up to 1600º C) tensile strength and long duration creep studies have been established. Specific attention was paid to the architectural fiber preform design as well as the materials used in construction of the composites. Actual testing of both tubular and flat, “dog-bone”-shaped tensile composite specimens will begin next year. Since there is no precedence for using ceramic composites within a nuclear reactor, ASTM standard test procedures will be established from these mechanical and environmental tests. Close collaborations between the U.S. national laboratories and international collaborators (i.e. France and Japan) are being forged to establish both national and international test standards to be used to qualify ceramic composites for nuclear reactor applications.

SUMMARY

Fiber reinforced ceramic composites are being considered for possible use as control rod cladding and guide tubes within a Very High Temperature Reactor (VHTR) design. A research program has been established to investigate these materials within the parameters of a VHTR core during service. Two candidate systems have been identified, carbon fiber reinforced carbon (C_f/C) and silicon carbide fiber reinforced silicon carbide (SiC_f/SiC) composites. Irradiation stability experiments for both candidate composites have been initiated within the High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory (ORNL) to determine the limiting dose level for each type. Irradiation test capsules are currently being exposed to 10, 20, and 30 dpa dose levels. Samples exposed to 10 dpa irradiation doses have been completed; samples to dose levels of 20 and 30 dpa are scheduled for completion in following years. Preliminary indications show that SiC composites may be stable for the full lifetime of the reactor (up to 30 dpa) while C_f/C composites become compromised at 8 dpa.

Concurrently, mechanical and environmental test capabilities for both candidate material systems are being established for temperatures up to 1600º C. Test equipment, high temperature testing methodologies, VHTR reactor core conditions, and composite sample design for high temperature tensile strength and long duration creep studies have been established. Since the control rods will be composed of tubular segments containing the high neutron cross-section material (i.e. B_4C), tubular test specimens were designed and fabricated for both composite types. In addition, small, flat tensile specimens were fabricated from composite plate materials in anticipation of the need for small, flat irradiation specimens that will actually fit within the reduced volume of a material test reactor core. Therefore, both tubular and flat plate tensile specimens were designed and fabricated for future mechanical testing. Actual testing of both tubular and flat, "dog-bone"-shaped tensile composite specimens will begin next year.

Specific attention was paid to the architectural fiber preform design as well as the materials used in construction of the composites. For maximum irradiation stability and moderate composite strength, both composite types used a simple ±45° bi-axial braiding architecture. Much stronger three-dimensional weaving was considered unnecessary for these moderately low stressed components. A much more important consideration was material selection, which resulted in significantly higher irradiation stability for both composite types. SiC composites used Hi-Nicalon Type-S fiber preforms with chemical vapor infiltration while the C_f/C composites used pitch fiber-pitch matrix composites with liquid infiltration techniques. These preform materials have been demonstrated to have superior irradiation stability to other SiC and carbon-based material systems.

Currently, there is no precedent for using ceramic composites within a nuclear reactor. Consequently, no ASTM standards or ASME code cases exist for using ceramic composite components in a nuclear core. This research program will use the test procedures and methodology established during these studies to create standardized mechanical and environmental test procedures for use in validating a structural ceramic composite for use in a nuclear reactor system. ASTM (or equivalent) standards are being created for the composite architectures

used, the high temperature test methods developed (both for tensile strength and creep tests), and environmental testing of SiC composites. In addition, ASTM round-robin test methods will be used to validate that these test standards are truly international standards that can be applicable to all reactor designs.

The development of international test standards will require close collaborations between the U.S. national laboratories and international collaborators. To this effect, International Nuclear Energy Research Initiatives (I-NERI) have been forged with Japan and France to establish both national and international test standards to be used to qualify ceramic composites for nuclear reactor applications. These international agreements will allow the researchers to share data, materials, and test samples, as well as provide a basis for working groups to create the standards.

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Structural Ceramic Composites for Nuclear Applications

1. BACKGROUND

Fiber reinforced ceramic composites have been identified as possible material candidates for high temperature nuclear reactor components. Specific components of interest are control rod cladding and guide tubes within a Very High Temperature Reactor (VHTR) design. These ceramic components require high thermal stability, good fracture toughness, and high irradiation stability during service. The control rods will be composed of segments of ceramic composite tubes containing high neutron cross-section material (i.e. B_4C). Each segment (approximately 1-m in length) will be joined to the next segment by an articulating joint to allow maximum flexibility of the rod during emergency use. The control rods will be used for both emergency shut-down of the reactor and controlling the active core.

Two ceramic composite systems have been identified as possible candidates for this specific application: carbon fiber reinforced carbon (C_f/C) and silicon carbide fiber reinforced silicon carbide (SiC_f/SiC) composites. C_f/C composites have been fabricated and used in a wide variety of different applications for decades, mainly in the aerospace industry. SiC_f/SiC have many similarities to the C_f/C composites but have only been readily available for a relatively short period of time. Both candidate composite systems were chosen due to their availability and past experience in irradiation environments.

The large market for carbon-based composites along with a wide variety of fabrication techniques to accommodate complex geometry components makes this material system a "mature technology."[1] There is little doubt that the control rod components consisting primarily of tubes and end-cap pieces can be fabricated using these materials. However, based upon fairly extensive studies on carbon-based materials these composites have demonstrated irradiation instability over time and exposure levels in an irradiation environment, Figure 1. As seen, even at relatively low dose levels (~ 7-8 dpa) the bundles of fibers within a composite can shrink or swell significantly creating large cracks and general degradation within the larger composite structure.

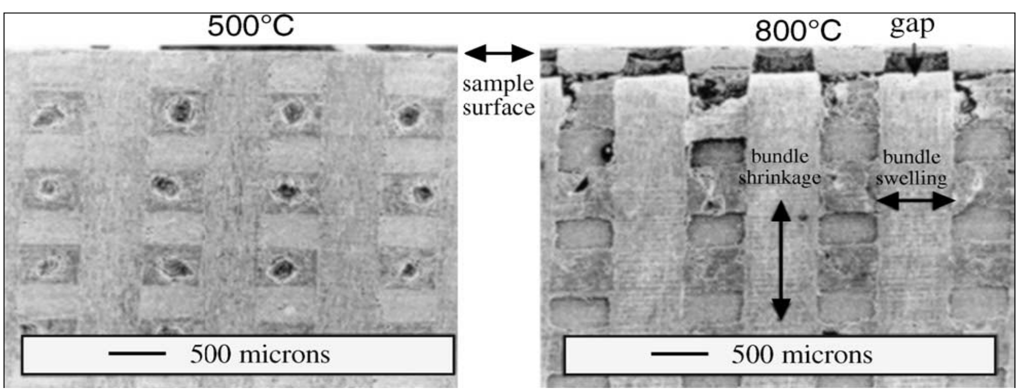

Figure 1. Irradiation damage in C_f/C composites due to dimensional changes in the carbon-based microstructure. (From *L. Snead et al, J. Nuc. Mater., 321 (2003) 165–169*)

Therefore, while there is no doubt that C_f/C composites will perform sufficiently well at beginning of life they will eventually need to be replaced as the material properties become compromised over time and dose.[2] It has been estimated that C_f/C composites will need to be replaced at least three times over the lifetime of the VHTR (nearly 60 years and up to 30 dpa).

SiC_f/SiC composites, however, have shown to be structurally stable to dose levels where C_f/C composites become significantly compromised (~ 8 dpa). It is thought that this material system may be stable enough to withstand a dose of 30 dpa, or the equivalent of the lifetime of the VHTR, Figure 2. Composites fabricated using the latest SiC fibers (Hi-Nicalon Type-S) show considerable stability up to 8 dpa as shown by the Hi-Nicalon Type-S curve (◆). While slightly less stable than monolithic SiC the composites show a threshold behavior where the mechanical properties do not change significantly after about 1 dpa. While the current data only extends to 7-8 dpa rather than the required 30 dpa the irradiation stability trends shown for SiC composites are encouraging.

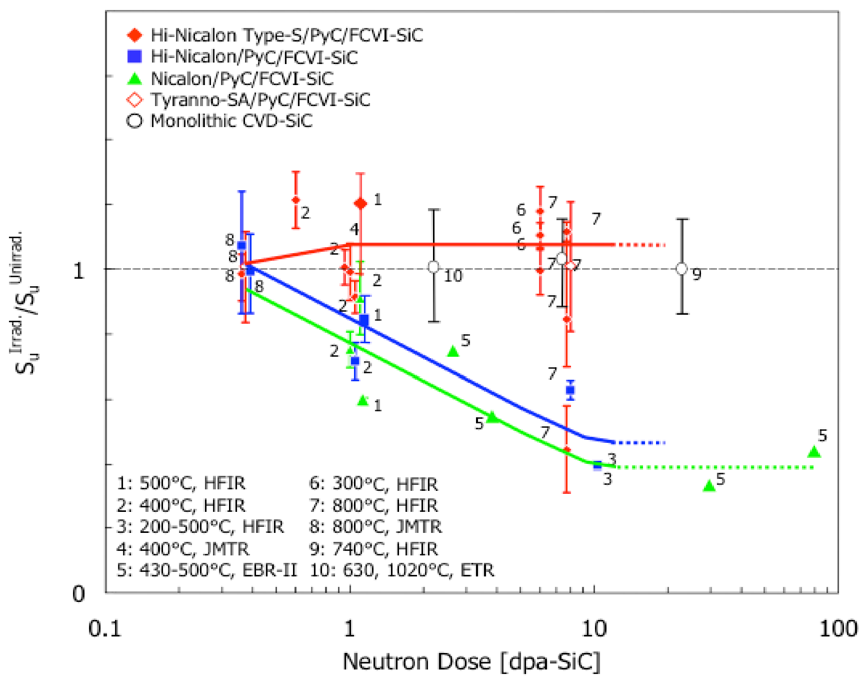

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Figure 2. Irradiation stability of different SiC_f/SiC composite types. The irradiated-to-non-irradiated ultimate strength ratio ($S_U^{Irrad}/S_U^{Unirrad.}$) plateaus after 1 dpa illustrating no change in mechanical properties for composites using Hi-Nicalon Type-S fibers. This stability is seen up to 8 dpa.

Nearly as thermally stable as C_f/C composites and potentially stronger, these composites are considered a viable alternative material system for control rod applications. The primary issue for SiC_f/SiC composites is the small amount of manufacturing experience and relatively few suppliers available to meet the demands of building this complex component. Therefore, the challenges for SiC_f/SiC composites lie in their fabricability, material supply, and the cost of manufacture.

2. INTRODUCTION

A research program within the U.S. is underway which should address these key challenges identified for each composite system. Furthermore, ceramic composites have never been used in such a capacity (i.e. a critical nuclear safety system component) and will require extensive testing and data to verify the viability of these systems for nuclear use. As such, standardized tests and testing procedures are being established for ceramic composites which are acceptable for both national and international nuclear code development.

Currently, a number of studies are in progress to determine material properties and establish testing procedures for each structural composite system. These tasks outline the general path forward that the research is taking in the U.S.:

1. Maximum dose levels for both C_f/C and SiC_f/SiC composites,

2. The cost and key fabrication issues for both C_f/C and SiC_f/SiC composites,

3. Determination of the mechanical properties and primary degradation mechanisms expected in service. Thermal, environmental, and irradiation induced degradation mechanisms will be investigated, and

4. Establishment of standardized tests derived from the mechanical, irradiated, and environmental testing as discussed in #3.

The U.S. research objectives are dual purpose; to down select the preferred material system (i.e. either SiC_f/SiC or C_f/C) and to develop testing methods (and initial results) for eventual code development of composites. Down selection will be achieved by determining if SiC_f/SiC is truly capable of withstanding 30 dpa dose levels without significant structural degradation (*Section 3 - Irradiation stability studies*). If SiC_f/SiC irradiation stability is not significantly increased from the C_f/C composites they will be eliminated from the testing program and only C_f/C will be considered, Figure 3. If the irradiation stability of SiC_f/SiC is shown to be significantly better, then both composite candidates will be tested but more emphasis will be placed on obtaining SiC_f/SiC composite results.

Test programs for both composite types will be developed simultaneously due to the long irradiation time necessary to determine irradiation stability. Mechanical studies (*Section 4 – Mechanical testing*) and environmental studies (*Section 6 – Environmental effects*) will be conducted over a range of temperatures up to 1600º C, the expected maximum off-normal temperature for a VHTR. Environmental tests will include further investigating the slow crack creep growth mechanisms for SiC_f/SiC composites developed in the past by PNNL. These studies will expose SiC_f/SiC material to a He environment simulating what is expected in the VHTR core.

Mechanical tests will focus upon simple tensile strength tests and long duration creep tests for both irradiated and non-irradiated samples. The mechanical testing program addresses two objectives:

1. *Testing composite samples*: Initial tensile strength and high temperature creep strength data will be generated on composite architectures likely to be used in reactor operations. The data will be used to form a database to establish a code case for using composites in control rod applications. These composites will use fiber preform architectures and optimal materials (i.e. Hi-Nicalon Type-S SiC fibers) that will be used in actual control rod structures.

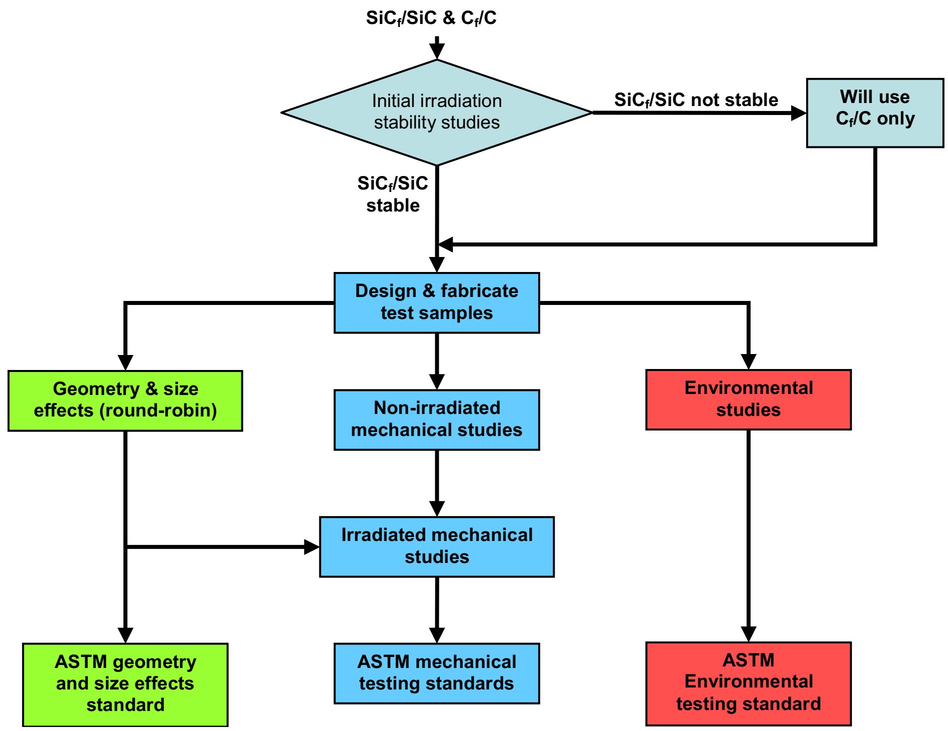


Figure 3. Path forward diagram of composite testing program. The end result will be ASTM test standards for mechanical, environmental, and irradiation testing.

2. *Geometry and size effects*: Anticipating the need for small, flat irradiation specimens that will actually fit within the reduced volume of a material test reactor core a series of ASTM round robin tests (both tensile strength and creep tests) will be performed to demonstrate that these smaller test samples adequately represent the true response of larger composite tubes used for control rod applications. These tests do not require using the more expensive Hi-Nicalon Type-S SiC fibers required for the mechanical tests described above.

A large number of mechanical and environmental test samples (*Section 5 – Design and fabrication of test samples*) for both composite types will be required for all mechanical testing. A series of tubular and flat test specimens will be fabricated for test methodology validation and for mechanical property testing.

Finally, standardized test specimen design, specimen dimensions, fabrication issues, and testing methodology will need to be agreed upon to develop national and international standards (*Section 7 – ASTM standards development*). Various national laboratories have been designated as the lead institution for each specific task outlined previously, Table 1. However, the data from each task will be compiled and analyzed by all participating laboratories to create a comprehensive model of the composite response in this specific application. This close collaboration between all the laboratories will culminate in the development of new ASTM (or equivalent) testing standards applicable for composite component systems in a nuclear application. An ASTM working group with all laboratories present is essential in the development of both national and international standards for these unique material systems.

Table 1. Tasks for composite research

Task	Principal Laboratory
Irradiation stability studies	ORNL
Design & fabrication of SiC_f/SiC test samples	ORNL/PNNL
Design & fabrication of C_f/C test samples	ORNL/INL
Environmental effects on SiC_f/SiC	PNNL
Thermo-mechanical studies	ORNL/INL
Irradiation creep studies	INL
ASTM standards development	All laboratories

3. IRRADIATION STABILITY STUDIES

To date neither C_f/C nor SiC_f/SiC composites have been exposed to irradiation doses equivalent to an expected VHTR full lifetime dose, around 30 dpa. The maximum dose applied to either composite is around 8 dpa where C_f/C composites have been shown to become compromised.

To determine the actual irradiation limit for these two different composites an irradiation study conducted within the High Flux Irradiation Reactor (HFIR) is currently being conducted.[3] This study will expose both C_f/C and SiC_f/SiC composites to 10, 20, and 30 dpa levels, successively. At each dose level representative samples will be removed from reactor and analyzed to determine physical and mechanical property changes within the composite structures.

This study is designed to be a "go/no-go" test for the SiC_f/SiC composites. Since the only advantage SiC_f/SiC has over C_f/C composites is superior irradiation stability the actual dose limit for SiC_f/SiC must be determined. If the SiC_f/SiC composites do not demonstrate sufficient irradiation stability to allow the control rods to become a lifetime component then C_f/C composites will be used as the material system for these components.

As of June 2005, three 10 dpa capsules containing SiC_f/SiC and C_f/C composite samples have completed irradiation and have been disassembled. One capsule which contains FMI-222 samples failed a leak test after the first assembly. That capsule was then rebuilt and is in a process of safety review. All other capsules started irradiation in early 2005 and will be irradiated until year 2006 or 2007, depending on the total required dose.

The irradiated specimens will be tested for fracture strength in a four-point-bend configuration. A true flexural strength value for these composites will not be completely valid since the sample size falls short of the minimum recommended in ASTM standard C1341 (Standard Test Method for Flexural Properties of Continuous Fiber-Reinforced Advanced Ceramic Composites). However, since these tests are designed as a "go-no go" test to determine the material's general irradiation stability this test method will provide evidence of a major change in mechanical properties and will be sufficient. In addition, adopting the present specimen geometry will be beneficial in order to maintain consistency with the previous series of experiment. Moreover, this geometry will yield the required comparative results.

Thermal diffusivity coupons will be cut out of the end sections of the broken bend bar specimens. Thermal diffusivity will be determined from room temperature to the irradiation temperature at 50°C intervals by the *t1/2* method. A specimen holder that accommodates multiple coupon samples will be prepared for this experiment. Thermal conductivity of the irradiated specimens will be calculated using the measured irradiated thermal diffusivity, measured mass density, and the theoretical values for heat capacity calculated for the nominal chemical composition.

4. MECHANICAL TESTING

Assuming that the composite systems are shown to be stable at the required doses it then becomes necessary to determine if they are structurally suitable as control rod materials. Extensive thermo-mechanical testing will be required to determine whether these materials are truly viable for this type of application. Standardized tests will be developed from these studies to provide the necessary data required for codification of these materials for use in a nuclear environment (see ASTM standards development section below). This data, even though it is recognized as preliminary only, will most likely be used in support of a code case for use of composite materials as control rod tubes.

Traditionally, it is standard practice to use small, representative test samples in place of full-size components. However, a real problem exists for scale-up of composite materials. Unlike monolithic materials these are composites engineered from two distinct materials using complicated infiltration techniques to provide full density and maximum mechanical properties. The material properties may be significantly affected when the component geometry or size is changed. This is a major consideration since small sample sizes and more suitable geometries are required for test samples especially for irradiated sample studies where the material must be placed within the limited space of a reactor. It was decided that it must be shown that the test samples adequately represent the true response of larger composite tubes used for control rod applications.

To fit into any nuclear reactor, test samples much smaller than the actual control rod diameters (~ 100-mm dia.) will be required. In addition, to further reduce the test sample volume and provide a larger number of irradiated samples, flat, “dog-bone”-shaped tensile specimens are considered to be an optimal geometry for test specimens. However, before these smaller, flat tensile specimens can be used it needs to be established that they are truly representative of large tubes, which would be used for the control rods.

Geometry effects: Tensile tests will be conducted to determine if flat samples accurately represent right cylindrical tubular samples for these composite architectures. Tensile tests over a range of temperature will be conducted for each sample type. The results will be compared and any effects resulting from geometry changes will be noted. Sizes between the tubular and flat plate samples will be similar (approximately 125-mm long x 9-mm wide).

Size effects: Once the geometry effects have been accounted for, the effects resulting from sample size on the mechanical response will be investigated. Round tubular specimens with diameter sizes ranging from 9-mm – 50-mm will be tested and compared over a range of temperatures. Flat tensile specimens will not be used.

A round robin testing program will be initiated for all labs (ORNL, INL, PNNL, and University of Bordeaux-France) with the appropriate number of tubular and flat plate specimens. Once the sample matrix has been established the participating laboratories will test the samples using similar testing methods. The results will be fed back to the appropriate ASTM subcommittee (or working group) and analyzed as discussed later. Experts from all labs must work within ASTM guidelines and methods to produce a defensible test matrix and testing procedures for ceramic composite tubes.

4.1 Strength Testing (Room and High Temperature)

Tensile strength testing has focused primarily upon establishing test specimen parameters, test fixture requirements, testing parameters, and the design of tubular and flat plate specimen dimensions. Strength tests will be performed over a range of temperatures (RT-1600º C) to determine failure response, high temperature “yield strength” or tensile matrix-cracking stress, and geometry effects for both tubular and flat plate specimens. The high temperature failure response and matrix-cracking stresses will be used to determine the optimal stress loads for long-term creep studies.

Design of test specimens: The final size of the fabricated test specimens dictated how the samples would be gripped, loaded, and tested. Since the Hi-Nicalon Type S fibers are extremely expensive it was decided that tubular samples as small as possible should be manufactured for both composite types. Final dimensions of approximately 125-mm long by 15-mm wide were selected as the most appropriate size (see next section for detailed description of sample dimensions).

Room-temperature studies: Room temperature tensile tests of both tubular and flat plate specimens are primarily designed to investigate the geometry effects study. The quantitative geometry effects between the tubular and flat plate specimens will be determined using a series of head-to-head comparison tests between the flat "dog-bone" and tubular tensile specimens. These tests will be conducted both at the national laboratories and with our French collaborators as part of the international test standards development for structural ceramics in nuclear applications.

In addition, the tensile strength results will also provide a comparative study to previous work in these composite systems for the fusion materials program. The fusion materials program used specimens fabricated from different fiber preform architectures and different geometries (i.e. flat, loom-woven plate material). A comparison of the new tensile strength results to the previous results used in the fusion work will illustrate the fabricability of the new tubular geometry components. Any dramatic changes from the expected strength levels would affect the viability of these composites.

To date, new load grips designed for both tubular and flat specimens have been designed (see next section) as well as an ASTM test matrix for both geometries (see section 7). Tensile tests and comparison studies on both composite types will begin next year.

High temperature studies: High temperature testing will include both tensile strength (for geometry effects studies) and long term creep tests of the Hi Nicalon Type-S tubular and flat plate samples. The tests will be conducted from 900 ºC to 1600º C over a range of times to provide a non-irradiated baseline of tensile strength and creep data for these ceramic composite systems.

Similar to the room temperature studies, both tubular and flat tensile specimens will be tested. Results from both geometry types will be analyzed and the geometry effects determined. Both geometry samples will be tested within the high temperature load frames using vertical clam-shell heaters and a static load. However, due to the anticipated service in the VHTR, the test frames will necessarily be outfitted with an environmental chamber allowing the samples to be tested at temperature in a He atmosphere, Figures 4 and 5.

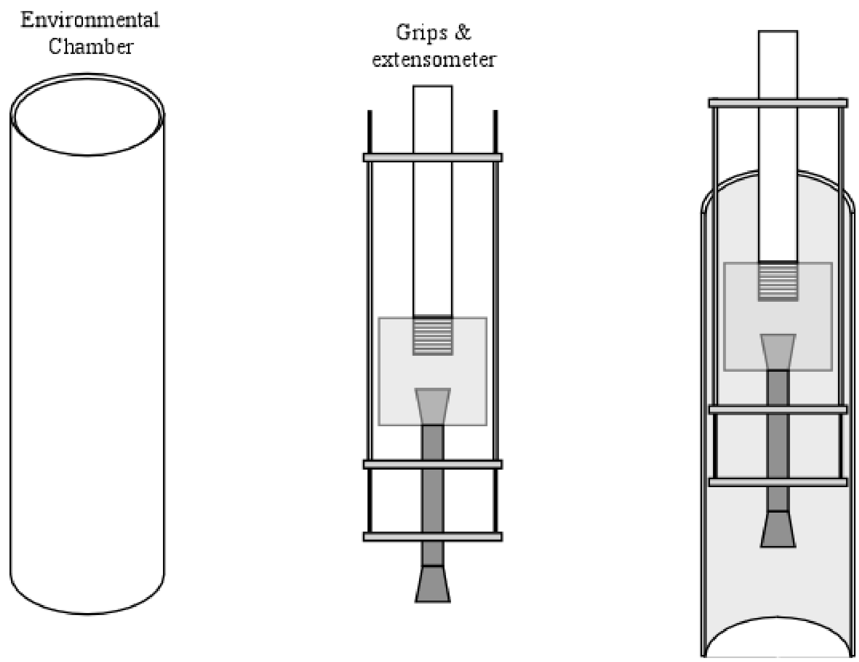


Figure 4. Schematic illustrations of a) environmental chamber surrounding sample and grip assembly b) high temperature grips and extensometers with sample, and c) grip assembly inside cut-away environmental chamber (retort).

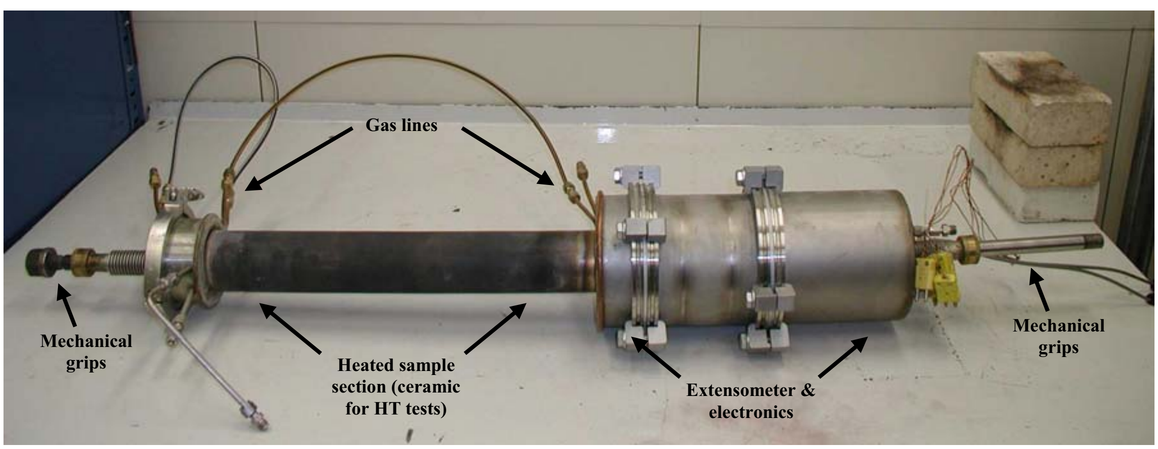


Figure 5. Typical environmental chamber housing required electronics, mechanical grips, and extensometers inside an inconel chamber capable of withstanding test temperatures of 1000° C.

Currently, load frames capable of very high temperature operation (<1700º C), using compatible environmental chambers, and appropriate controllers have been modified in support of the high temperature tests. The new furnaces will be capable of sustained temperatures in excess of 1600º C to envelope the anticipated operating temperatures of the control rods while the ceramic retort environmental chambers will provide the necessary environmental conditions.

Ceramic environmental chambers with an inside diameter of 75-mm will be used for the very high test temperatures (up to 1600º C). The relatively small sample sizes for both the tubular and the flat plate specimens should be accommodated within the limited volume of the environmental chambers, see Figure 4. An ultra-high purity helium gas environment will be used within the chambers to simulate the

VHTR reactor environment. High temperature tests and comparison studies on both composite types will begin next year.

High temperature grip and extensometer design: Appropriate high temperature grips, extensometers, and insulation requirements inside the chamber were also addressed. At the expected high test temperatures actively loaded grips will not be possible for long-term creep studies (i.e. mechanically tightened grips will creep and relax inside the environmental chamber). Therefore, a shoulder-mounted gripping system was designed to allow passive gripping for high temperature testing. This required the tensile specimens to have a tapered end, or shoulder, fabricated on each end of the specimen.

High temperature grips capable of being used for both tubular and flat tensile specimens are being designed and fabricated. These are passive grips that use the flared ends of the test samples to load the specimens in tension, as opposed to active grips which are spring loaded and may lose their gripping force if exposed to high temperatures over long periods of time, Figure 6.

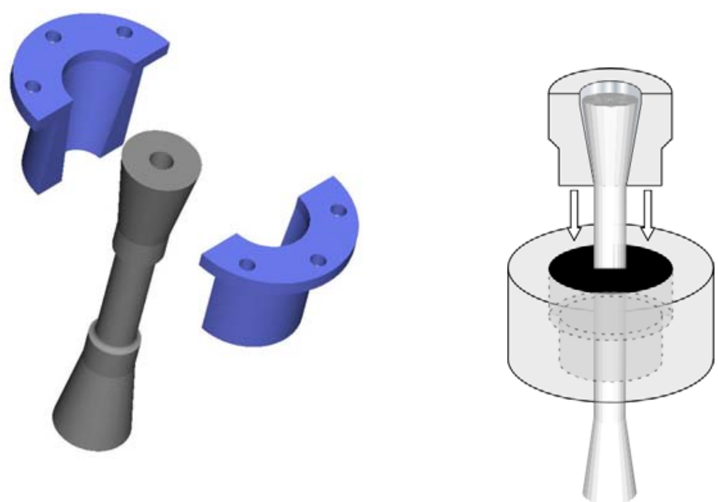


Figure 6. High temperature grip design for passive loading of tubular test specimen.

4.2 Irradiation Creep Studies

A primary degradation mechanism identified for composite control rod components is irradiation creep. A study to determine the creep rate of these composite systems under irradiation is necessary. Preliminary discussions focusing on sample dimensions, loading methods, and the design of an irradiation canister for insertion into a reactor have been conducted.

As discussed previously, irradiation samples will necessarily need to be small and compact to minimize volume within the reactor core. Using previous experience and sample designs for creep studies of metals and monolithic materials a general sample size approximately 50-mm long and 12-mm wide (at the ends) has been determined. No detailed dimensions for the samples have been decided to date. A final design for load grips or the irradiation canister has not been determined. Detailed discussions of the sample dimensions and canister design will be decided next year.

5. DESIGN AND FABRICATION OF COMPOSITE TEST SPECIMENS

Test samples for both composite types are currently being designed and fabricated.[4] Since these are engineered material systems the proper design of the composites is critical. Key fabrication parameters include the fiber type, fiber preform structure, weave/braid techniques, weave/braid angles, composite thickness, infiltration method, and material selections. Previous experience[5] and the in-service conditions expected in the reactor were used to narrow the composite design parameters to a manageable list for each composite type. Based upon current control rod design information the expected in-service conditions are given in Table 2.[1]

Table 2. Anticipated operating parameters for composite control rods.

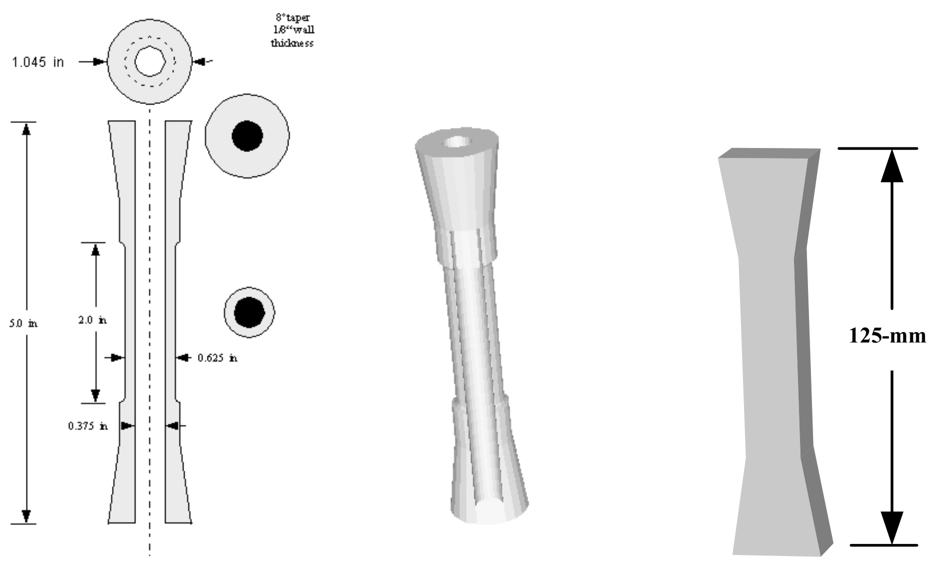
Parameters	Assumed values
Control rod dimensions	
Diameter	90 – 101-mm
Thickness	2 – 3 mm
Length (section)	~ 90 mm
Length (core length)	~ 10 m
Static load	3-5 MPa
Dynamic load (stuck rod)	~ 6-10 MPa
Operating temperatures	
Normal operation	1250 ºC
Off-normal	1600 ºC
Max. temp. gradient	800 ºC – 1250 ºC
Expected dose	0.5 dpa/yr (30 dpa total)
Operating environment	He (min. impurities)

Due to the relatively low operating stresses and lack of hermetic requirements it was decided that a simple ±45° bi-axial braiding architecture was sufficient for both composite fiber preforms. This greatly simplified the braiding process, reduced the amount of reinforcing fiber needed, and still provided sufficient strength to meet the in-service requirements. However, both the infiltration methods and materials used for each composite type were distinctly different. Test samples for both SiC_f/SiC and C_f/C composites are currently being fabricated. Details are given in Table 3.

Table 3. Fabrication parameters and test specimen dimensions for composite samples.

SiC_f/SiC composites	C_f/C composites
Simple tube braid preform architecture	Simple tube braid preform architecture
Hi Nicalon Type-S fibers	Mesophase pitch
CVI (chemical vapor infiltration)	Both fibers & matrix material
Beta-SiC	Superior irradiation stability
Multiple infiltration cycles	Liquid infiltration
~ 85 %TD	Fully graphitized @ 2500 - 3000 ºC
High temperature anneal between cycles	~ 91 %TD
20 – 127-mm x 9.5-mm diameter tubes	1 – full-sized prototype control rod section
4 – 254 x 76 x 2.5-mm thick plates	5 – 100-mm diameter tubes
40 – non-Type S tubes for ASTM round robin testing (127-mm x 9.5-mm diameter)	3 – 30-cm x 30-cm x 2-mm thick plates

Flat, dog-bone shaped tensile specimens will be machined from the larger flat plates. These flat specimens will be approximately the same size as the tubular specimens (i.e. same height and wall thickness), Figure 7. High temperature grips capable of being used for both tubular and flat tensile specimens are being designed, as discussed previously.



a. b.

Figure 7. Schematic of a) tubular test samples and b) flat, “dog-bone” tensile test specimen.

6. ENVIRONMENTAL EFFECTS ON SiC_f/SiC COMPOSITES

It is assumed that the fundamental irradiation response of the microstructure will be similar for all preform architectures and component geometries. However, using different preform architectures (i.e. weave angles, fiber tow counts, weave structures, etc.) can lead to differences in the macroscopic mechanical responses in the composite structure due to infiltration efficiency, fiber bending stresses, or matrix/fiber interface characteristics. The environmental conditions these materials will be subjected to may also change the overall creep response of the composite (i.e. creep crack growth for fiber reinforced materials).

PNNL has extensive experience in environmental degradation of SiC. They have developed a creep crack growth model to predict the environmental factors on the overall creep of the SiC_f/SiC composite structures. This model is currently being expanded to include flat, thin specimens (i.e. to simulate flat dog-bone shaped tensile specimens). It is anticipated that the model may be further expanded to include the 3-dimensional tubular geometry if applicable/desirable at a future time.

To improve the accuracy of the model predictions a limiting "reactor environment" for elevated temperature tests must be determined. Most likely, the limiting environmental species in the He loop will be the H_2/H_2O ratio. Assuming these species are the most damaging to the composites PNNL will determine the degradation potential for various H_2/H_2O ratios using both modeling and experimental tests.

Slow crack creep growth results (experimental and modeling): Slow crack growth tests have been performed in high purity Argon (expected to be no different than He) at 1100º C, 1150º C, 1200º C, and 1300º C. These tests require analysis for crack growth rates but we observed that failure for these Type-S fiber composites at 1300º C was very rapid, which suggests an upper temperature limit below 1300º C for this composite system.

PNNL is using materials that were on hand and purchased in 2004 from GE Power Systems. The SiC_f/SiC materials are 8-harness satin weave, 8 ply, Hi-Nicalon Type-S fiber composites. They are intended to be a surrogate until the newer Hyper-Therm materials arrive. The 4-point bend slow crack growth tests were all performed on un-notched bend bars and can be analyzed to give crack growth rates in Argon due to fiber creep. An activation energy analysis will be performed and compared to creep of single Type-S SiC fibers.

Studies will continue up to 1400˚C in pure Argon or pure He. Then, testing will begin using impure He that is tailored to simulate actual VHTR operational environments. A crack growth model will be developed to explore crack growth and time-dependent bridging in Type-S materials.

7. ASTM STANDARDS DEVELOPMENT

Unlike other structural materials, initial standardization efforts for SiC_f/SiC composites were concurrent with their development because it was recognized that their commercial diffusion and industrial acceptance could be hampered by lack of standard test methods, databases or design codes.[6,7]

Numerous standardized mechanical testing methodologies have been developed for characterizing the mechanical properties of engineering materials. Noteworthy are the standards developed for the American Society for Testing and Materials (ASTM). Typically these standards are based on testing experience including both independent research and round-robin evaluations. Such standards, so developed, are the result of consensus on the part of ASTM participants and, therefore, address the needs of the participants at the time the standards are developed. In the United States, sub-committee C28.07 on Ceramic Matrix Composites of the American Society for Testing and Materials (ASTM) has spearheaded the widespread introduction of standard test methods for SiC_f/SiC and other ceramic matrix composites [2].

These standards have primarily concentrated on the evaluation of test coupons to determine the intrinsic mechanical properties of these materials and little work has been focused on the development of standards for the evaluation of ceramic matrix composite components. The potential use of SiC_f/SiC composites in the VHTR will require the existence of:

- design codes, which list "rules" and guidelines for designing and testing SiC_f/SiC composite components and incorporating them into advanced designs;

- design codes which regulate the certification procedures for processing materials, fabricating components, and assembling final designs; and

- databases that provide statistically significant and complete material properties and performance.

Since 1995, one noteworthy national effort has been initiated in design codes for advanced ceramics: ASME Boiler and Pressure Vessel Code. Of particular importance for the Next Generation Nuclear Power (Gen IV) applications (such as control rod cladding and guide tubes) are acceptance of aspects of codes (including standards) by the Nuclear Regulatory Commission (NRC).

The primary technical objectives of this project are:

1. To coordinate efforts that lead to the introduction of national (ASTM) and international (ISO) test standards for the thermo-mechanical evaluation of SiC_f/SiC composites and components fabricated with these materials;

2. To coordinate round-robin testing programs for establishing precision and bias statements for the new standards;

3. To carry on efforts for developing national design codes that address the use of SiC_f/SiC composites as part of such national efforts as the ASME Boiler and Pressure Vessel Code; and

4. To facilitate efforts for development and expansion of databases for SiC_f/SiC composites.

This project addresses specific needs in the characterization of SiC_f/SiC composites for ultimate use in the engineering design and fabrication of control rod cladding and guide tubes in nuclear power plants. This work has been prioritized based on the expected modes of failure of these components.

8. I-NERI COLLABORATIONS

International Nuclear Engineering Research Initiatives (I-NERI) are designed to allow a free exchange of ideas and data between U.S. and international researchers working in similar research areas. This international agreement encourages strong collaborations between research institutions where a benefit to both countries is anticipated. Two I-NERI collaborations have been proposed between the U.S. and France and the U.S. and Japan.

8.1 France

A three-year I-NERI grant between U.S. - French research institutions (INL, ORNL, PNNL, CEA, and University of Bordeaux) has been approved for research and development of SiC/SiC composites. The proposed research will investigate the issues surrounding the development of tubular geometry SiC/SiC composite material for control rod and guide tube applications. Mechanical, thermal, and radiation-damage response of the French fabricated tubular composites will be studied during this time.

The project is designed to take full advantage of the innovative SiC/SiC technologies developed by our French collaborators (Prof. Jacques Lamon at the Universite de Bordeaux, Apessac, France). This research group has pioneered the use of 2D woven SiC/SiC composites and also nanoscale-multilayered pyrolytic carbon/silicon carbide interphases.

The French will benefit from the U.S.'s full-scale composite testing and irradiation program. The U.S. research program is much more focused upon application oriented testing and verification. Thus, both programs compliment each other with little to no overlap of research. Initial meetings have discussed data exchange, sharing modeling experience, and test sample exchanges between the two programs. Further meetings in the coming months will provide detailed schedules for these exchanges.

8.2 Japan

A U.S.-Japan I-NERI is currently being discussed and negotiated. The proposed research will investigate development issues surrounding tubular C/C composite material for control rod and guide tube applications. Similar to the SiC/SiC composite research, the mechanical, thermal, and radiation-damage response of both the U.S. and the Japanese fabricated tubular composites will be studied.[8]

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