## 3.1 LOW ACTIVATION JOINING OF SiC/SiC COMPOSITES FOR FUSION APPLICATIONS: MODELING DUAL-PHASE MICROSTRUCTURES AND DISSIMILAR MATERIAL JOINTS

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### OBJECTIVE

Finite element continuum damage models (FE-CDM) have been developed to simulate and model dual-phase joints and cracked joints for improved analysis of SiC materials in nuclear environments. This report extends the analysis from the last reporting cycle by including results from dual-phase models and from cracked joint models.

### SUMMARY

The international fusion materials community designed miniature torsion specimens for joint testing and irradiation in test reactors with limited irradiation volumes since SiC and SiC-composites used in fission or fusion environments require joining methods for assembling systems. HFIR irradiation results indicated two broad types of joint damage due to irradiation at either 500˚C or 800˚C to 3 dpa or 5 dpa. One type of damage can be categorized as microcracking within multi-phase joints at the micron length scale. The other type of damage can be categorized as cracking within the miniature torsion sample and within the joint where the cracks are now on the scale of the joint thickness and are not considered to be microcracks. This report discusses methods to model both types of cracking due to differential properties within the joint due to multiple phases or due to the fact that the joint itself is dissimilar from the joined CVD-SiC material. This analysis requires two different models to account for these effects.

### PROGRESS AND STATUS

#### Introduction

The international fusion materials community has irradiated and is currently irradiating several SiC joint types and compositions in the HFIR reactor at ORNL [1]. PNNL is working with Politecnico di Torino (POLITO) and ORNL using miniature torsion specimens that have been specifically designed for pre- and post-irradiation joint shear strength testing [2]. To elucidate how cracks initiate and propagate in the torsion joint specimens, finite element analyses of these specimens subjected to torsion were performed using a continuum damage mechanics (CDM) model previously developed at PNNL for elastic materials for which any nonlinearity in stress/strain response is due solely to damage and not to other irreversible processes such as plasticity [3-5]. The CDM model was implemented in the ABAQUS® finite element code via user defined subroutines. This CDM model [3] was now applied at the submicron scale by creating a dual-phase FE mesh within the joint region. The constitutive behavior of each phase in the modeling domain was described by the CDM model to explore the effects of differential material responses within the joint to external forces, such as thermal expansion, swelling, and applied loads. In addition, the previous model was also exercised but now including pre-cracked joints to simulate observed HFIR cracking patterns [1]. Although the joints survived the HFIR irradiation with reasonable properties, there was degradation observed in SEM images taken post-irradiation at ORNL. This report discusses methods to understand the sources for the observed cracking and subsequent joint degradation so that improved joints can be fabricated for fusion applications.

#### Model Formulation

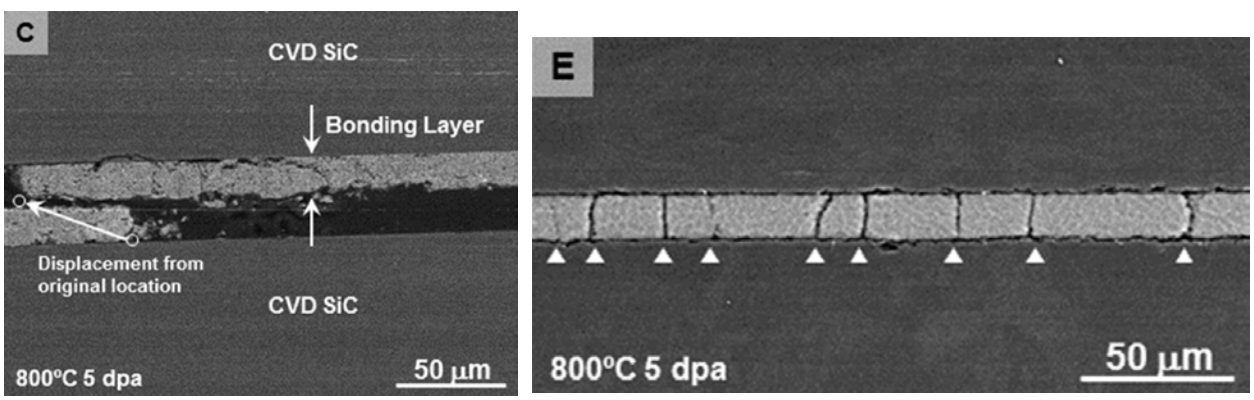
<u>Approach</u>

This section summarizes the damage model and method for modeling the joints [3-7] in the joined CVD-SiC that has been previously reported. Damage in an elastic and damageable material, for which any nonlinearity in stress/strain response is due solely to damage and not to other irreversible processes, can be described by a scalar variable, $D$, that can be related to the level of damage accumulation in the material [6]. Damage affects the material stiffness according to a stiffness reduction law. Using the concepts of thermodynamics of continuous media [6, 8], a thermodynamic potential is defined to derive

the constitutive relations and the thermodynamic force associated with the damage variable. This damage model uses the density of the elastic deformation energy as the thermodynamic potential that provides a coupling between damage and elasticity. A damage criterion dependent on a damage threshold function is defined and the damage evolution law is obtained. Damage evolves with the deformation according to the damage evolution law until a critical (saturation) state at which $D = D_{cr}$ $(0 < D_{cr} < 1)$ and failure occurs. $D_{cr}$ is small for brittle materials, and this is the case for ceramic materials studied in this work. In this work, failure at damage saturation ($D = D_{cr}$) leading to crack initiation and propagation is modeled by a vanishing finite element technique [9] that reduces the stiffness and stresses of the failed element to zero [3, 10]. We choose to implement the damage model for brittle ceramics in shear loading. The thermodynamic and CDM approach adopted is phenomenological and it does not describe the detailed frictional sliding of crack ligaments but the magnitude and effect of shear damage on the material response are *phenomenologically* captured by the damage variable, *D,* driven by the associated thermodynamic force, *F*(*D*), and the damage evolution law.

As discussed in Ref. [1] miniature torsion joints were irradiated in HFIR at ORNL and examined post-irradiation using SEM and then tested in torsion. Archival joints were imaged and tested pre-irradiation for comparison. Joint images showed two types of damage that is assumed to have originated from the neutron irradiation in HFIR for several of the joints (but not all). One type of damage can be categorized as microcracking within multi-phase joints at the micron and sub-micron length scale. The other type of damage can be categorized as cracking within the miniature torsion sample and within the joint where the cracks are now on the scale of the joint thickness and are not considered to be microcracks. This FE-CDM requires two different models at two different length scales to account for these observed effects.

First, a pattern of cracks at the joint/specimen scale are created within the joined sample so that the joint region consists of a homogenized layer containing incipient damage in the form of either planar cracks, which are cracks parallel to the joint plane, or transverse cracks perpendicular to the joint plane. These two types of cracks are shown in Figure 1. The homogenization method uses the previously described Eshelby-Mori-Tanaka approach (EMTA) [11] to model cracks as elongated inclusions with zero stiffness and provide a tensor description of defect orientations for the homogenized material. We anticipate that these two types of crack patterns will behave differently due to this directionality with regard to the applied torsion strains. A crack volume fraction of 25% is assumed as an estimate.



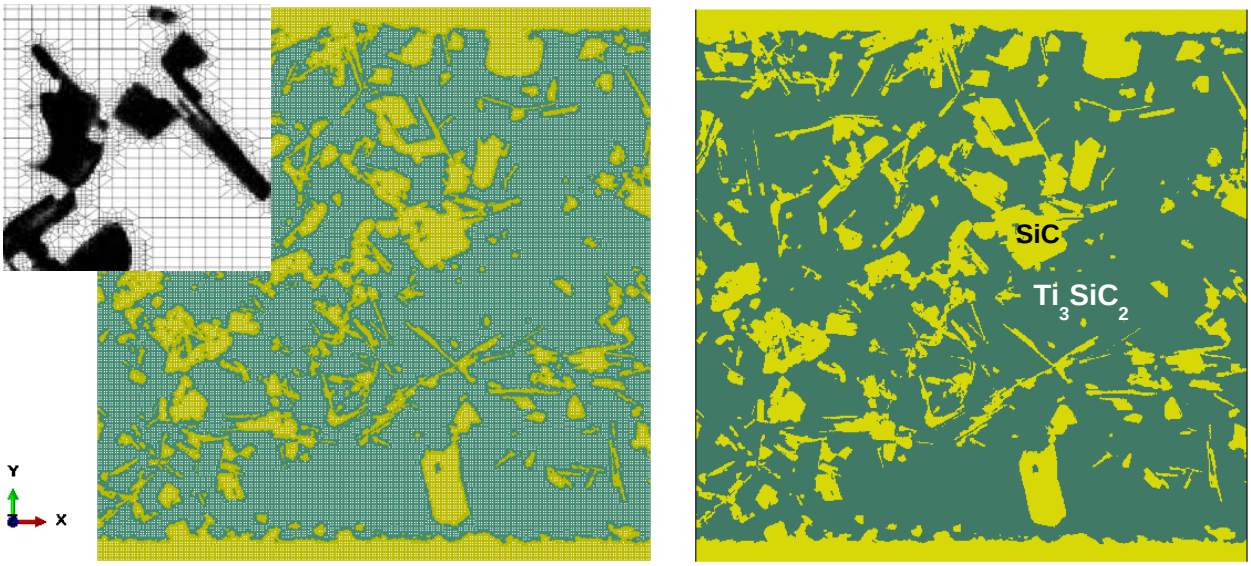

(a) (b)

**Figure 1.** SEM images from Ref. [1] showing planar cracking in (a) between the joint and the CVD-SiC and transverse cracking in (b) within the glass-ceramic joint region. Both joints were irradiated at 800˚C to 5 dpa in HFIR and examined at ORNL post-irradiation prior to torsion testing.

Second, the origin of the intra-joint microcracking was investigated by creating a finite element mesh of the joint microstructure using the OOF2[1] public domain software. A digital image that was 14.1 µm on a side (910 x 910 pixels) was input and OOF2 was used to create a two-phase FE mesh consisting of 230k

[1] Software developed at the National Institute of Standards and Technology

FE nodes, both triangular and quadrilateral elements. This allowed a high resolution meshing of the microstructure at the phase boundaries and provided the ability for ABAQUS to assign individual constitutive laws to each phase. The meshed microstructure and image are shown in Figure 2.




(a) (b)

**Figure 2.** The FE mesh is shown in (a) superimposed on the image of the microstructure. Inset in (a) shows the high-resolution mesh at the interfaces between the SiC (black) and the ternary MAX $Ti_3SiC_2$ (white) phase. Shown in (b) is the image with the two phases identified within the joint.

**Results**

Model Results

The damage models are implemented in ABAQUS® as a FE analysis of the miniature torsion specimen containing 1) a homogenized layer with incipient cracks in two orientations and 2) a region within the joint containing regions of SiC and regions of $Ti_3SiC_2$ meshed from a digital image. The assumed mechanical properties of the CVD-SiC for both models are shown in Figure 3.

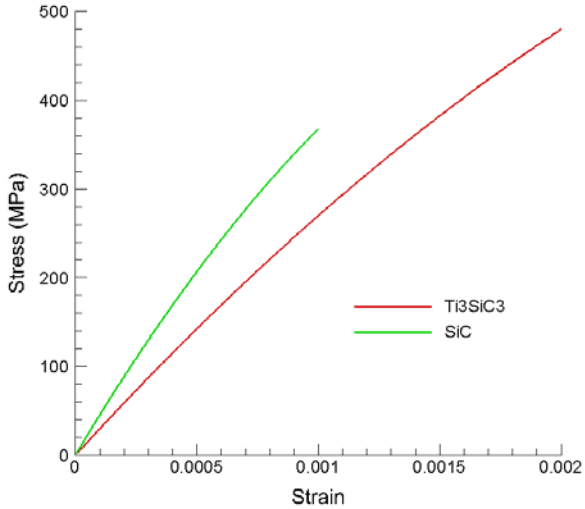



**Figure 3.** Assumed constitutive laws for CVD-SiC and $Ti_3SiC_2$ for this report. Failure occurs for SiC at a strain of 0.001 and 0.002 for $Ti_3SiC_2$.

**Table 1.** Material properties for CVD-SiC and $Ti_3SiC_2$ phases

| Material | Elastic Modulus (GPa) | Poisson Ratio | Strength (MPa) | Failure Strain | Damage Variable Critical Value |
|---|---|---|---|---|---|
| **SiC** | **460** | **0.2** | **368** | **0.001** | **0.2** |
| **$Ti_3SiC_2$** | **300** | **0.2** | **480** | **0.002** | **0.2** |

The FE-CDM was exercised for both the planar and transverse cracked joint and the damage fracture pattern is shown in Figure 4 for both cases. The CDM model predicts that the planar cracks are more deleterious to the joint degradation compared to the transverse and nearly vertical cracks, which is a consequence of the crack orientation with respect to the applied torque. Also, in comparison to assumed porous joints that were previously modeled the planar cracks are quite efficient in reducing the joint failure strength. Joints that prior to irradiation would be predicted to fail within the CVD-SiC are now predicted to fail in the joint plane [1, 4, 5].

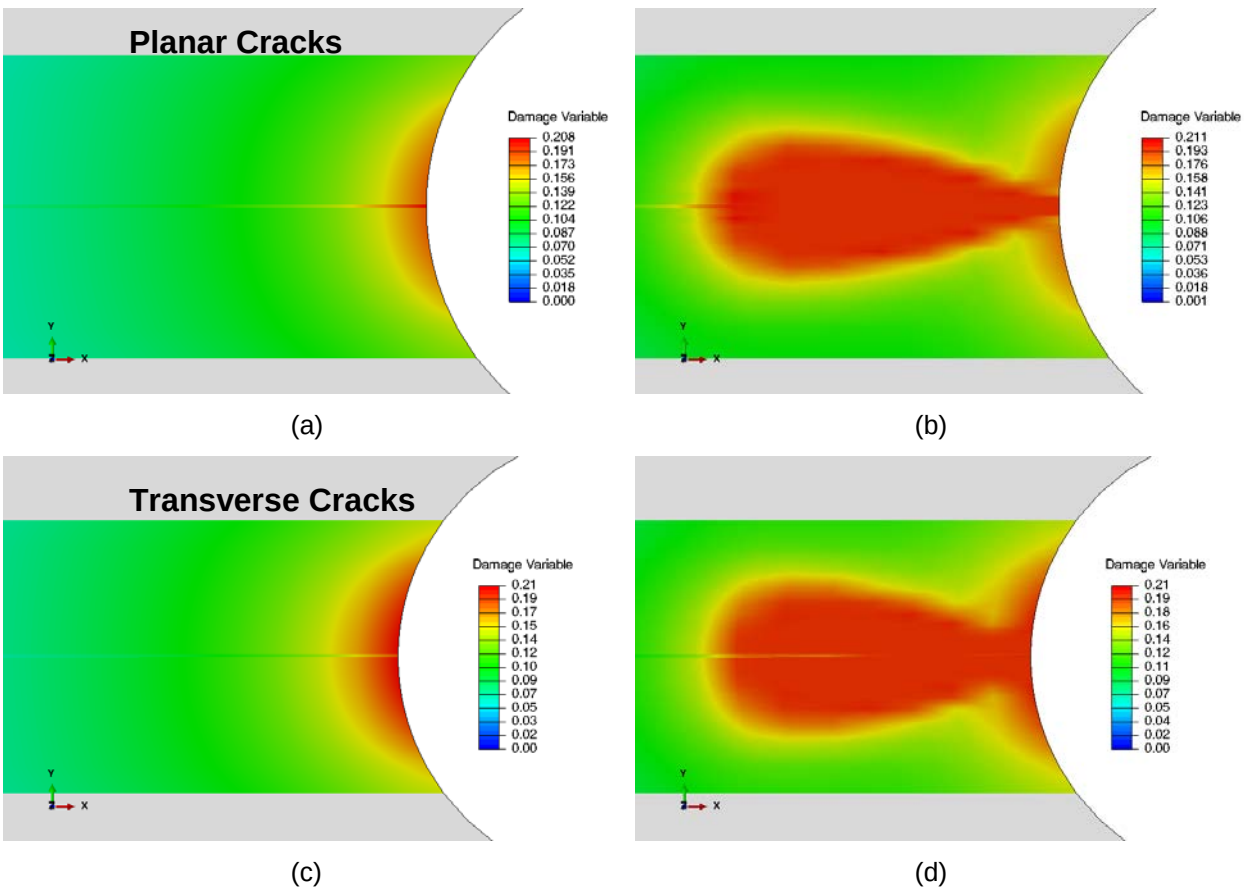
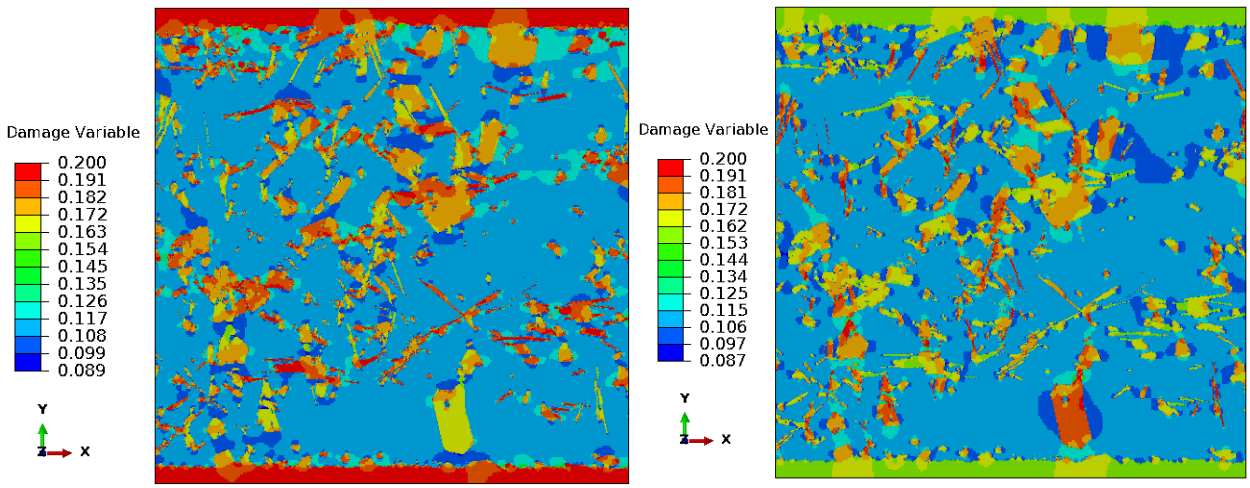



**Figure 4.** Predicted fracture patterns (red regions) using an elastic damage model for the torsion joint specimens containing 25% volume fraction of planar cracks in (a) and (b) and 25% volume fraction of transverse cracks in (c) and (d). In each case an initial damage and final damage pattern are shown.

Figure 5 shows results from the micromechanical two-phase model under bi-axial loading. For this case, the left/bottom boundaries are fixed and the right/top boundaries are displaced with either $u_x/u_y$ = 3 or 1/3 displacement ratio. The displacements $u_x$ and $u_y$ are expressed in the xyz coordinate system shown in Figure 5. Bi-axial tension can be considered as a substitute for volumetric swelling in the absence of a swelling model for $Ti_3SiC_2$. The hypothesis being explored here is that a dual-phase microstructure has some liabilities when it comes to incompatibility strains within the joint. This behavior can account for the observation of microcracking within the joint after HFIR irradiation exposure [1]. The model clearly shows that differential strain within the joint can account for cracking at the microscale in the joint, which qualitatively agrees with observations for this joint following HFIR irradiation.




(a) (b)

**Figure 5.** Damage accumulation within the joint using the mesh shown in Figure 2 and the constitutive properties in Table 1 and Figure 3. Shown in (a) is the case for $u_x/u_y$=3 and in (b) the ratio is 1/3. The red regions of damage saturation are considered to be crack nuclei or microcracked regions. Note that this cracking occurs adjacent to the SiC phase within the joint and also at the joint interface with the CVD-SiC. This result supports the thesis that a dual-phase joint might develop a system of microcracks due to differential strain response to either thermomechanical loads or volumetric swelling.

**Discussion**

Model Predictions and Comparisons

The FE-CDM model appears to be able to account for two key observations from the HFIR joint irradiation study reported in Ref. [1]. One observation was the change in torsion sample failure mode from CVD-SiC body failure to in-plane joint failure. As shown in Figure 1, planar cracks were observed in the irradiated joint between the dual-phase joint and the CVD-SiC specimen body. This was thought to be the cause for the observed transition from out-of-plane to in-plane torsion test failure mode. Results from the FE-CDM model including an assumed 25% planar crack distribution in the joint and homogenized using EMTA support this hypothesis. As shown in Figure 4a and Figure 4b, planar cracks act to concentrate crack damage in the plane of the joined specimen, which shifts the failure location from the CVD-SiC base material to within the joint material, or out-of-plane to in-plane fracture[2]. Further, as shown in Ref. [1], the presence of transverse cracks does not quite have the same effect since that joint retained some portion of failure in the CVD-SiC material.

Microcracking within the dual-phase PNNL joint was also observed following HFIR irradiation at 800˚C to 5 dpa. Figure 6 shows an image from Ref. 1 that reveals microcracking within the dual-phase joint material and that the cracking appears to coincide with the dark features in the joint, which are the SiC phase regions. The FE-CDM model constructed here, even though it does not contain all the detailed

[2] In Ref. [1] this is referred to as base material (B) or joint plane (J) fracture.

deformation modes experienced by joints irradiated in HFIR, such as thermomechanical and swelling loads, demonstrates that deformation of the joint regions might cause microcracking in the joint and that the origin of the microcracking is probably associated with the $SiC/Ti_3SiC_2$ incompatibility strains at the phase interfaces. This is shown clearly in Figure 5 where the damage accumulation in the form of crack nuclei is predicted to occur at the interfaces between the SiC and $Ti_3SiC_2$ phases within the joint at micron or submicron length scales. This illustrates a potentially key weakness of multi-phase joints where the mechanical properties of constituent phases are significantly different.

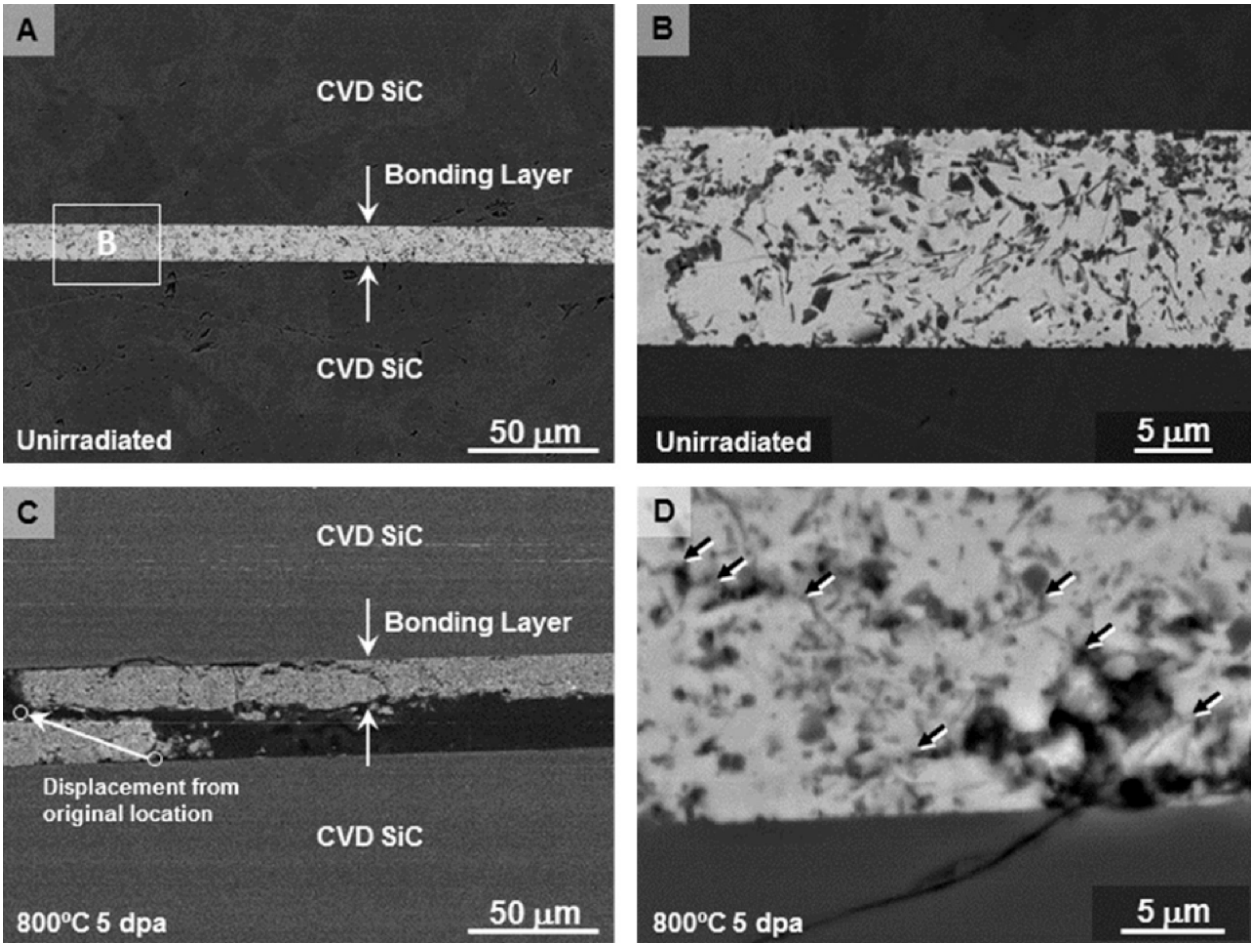



**Figure 6.** SEM images taken from Ref. [1] showing the pre-irradiated and post-irradiated joint microstructures. The larger planar cracks are shown in C and the micron-scale microcracks are shown in D. The model discussed here captures both types of cracking using the FE-CDM approach on a dual-phase FE mesh (See Figure 2 and Figure 5).

**Conclusion**

A modification of the PNNL FE-CDM model [4, 5, 11] implemented in the ABAQUS FE package has been shown to provide qualitative agreement with experimental observations following SiC joint irradiations in HFIR. Both in-plane and transverse cracks are simulated and the overall response of the torsion sample with this type of incipient damage agrees well with torsion test data obtained at ORNL following HFIR irradiation. In addition, SEM observations of irradiated joints indicated microcracks were nucleating within the dual-phase joint region at $SiC/Ti_3SiC_2$ interface boundaries and the PNNL FE-CDM model captures this type of damage when a microstructural scale FE-CDM model containing two phases is strained in bi-axial loading.

**Future Work**

The developed FE-CDM model will be modified to account for thermophysical and swelling loads consistent with the HFIR irradiation conditions.

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