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In-Situ Investigation of Advanced Structural Coatings and Composites

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Overview

The premise of this project is a comprehensive study that involves the *in-situ* characterization of advanced coatings and composites by employing both neutron and X-ray diffraction techniques in a complementary manner. The diffraction data would then be interpreted and used in developing or validating advanced micromechanics models with life prediction capability. In the period covered by this report, basic work was conducted to establish the experimental conditions for various specimens and techniques. In addition, equipment was developed that will allow the *in-situ* studies under a range of conditions (stress, temperature, atmosphere and so on). The details are described below.

X-Ray Diffraction Studies of TBCs: Preliminary heating experiments were performed on several TBC specimens obtained from D. R. Clarke (UCSB). A collaboration was established with Dr. Clarke to study the residual stresses in TBCs. His extensive background in the microstructural characterization of TBC systems together with his expertise in the use of piezospectroscopy to measure stresses in some oxides effectively complemented our expertise on XRD. The first experiment involved heating a TBC while monitoring its cross section with a 25x100 μm^2 X-ray beam. Due to the failure of the furnace, the experiment could not be carried out beyond 700°C. The results can be summarized as follows: (1) High-energy XRD can be used to study the *in-situ* residual stress evolution during the high-temperature anneal and/or cycling of a TBC. (2) Spatial resolution reaching 1 μm is possible allowing the differentiation of the behavior of various layers in a typical TBC system. (3) Cross-sectional investigations are better than plan view studies. This is due to the fact that the latter yields a very strong response from the superalloy substrate and does not distinguish between the various TBC layers. (4) Due to absorption effects, model specimens are needed to effectively study the cross section of a TBC. Specifically, samples should be rectangular prisms of depths no more than 5 mm. (5) A special furnace is needed to allow a successful experiment. Such a furnace (with IR heaters) has been procured and tested.

X-Ray Diffraction Studies of CMCs: Using specimens provided by NASA Glenn, preliminary studies were performed at APS to identify critical experimental variables. The first specimen was a 'microcomposite' of a single SiC fiber in a SiC matrix. It was sampled with a 100x100 μm^2 X-ray beam to show that the fiber and the matrix could be distinguished although both have the same crystallographic phase. The second sample was plate-like and had a Si_3N_4 matrix and SiC fibers. Despite significant peak overlap, it was possible to identify individual reflections from each phase using an analyzer crystal. Both specimens will be run extensively soon based on the experimental parameters identified so far. It was determined, however, that the most powerful method to study damage evolution in CMCs is a combined use of X-ray diffraction and imaging. To achieve this, a special loading fixture was designed and built that will allow loading experiments while tomographic images (e.g., crack propagation) and crystallographic data (lattice strain, texture, phase information) are collected simultaneously. This fixture was tested at APS over the summer and the first extensive studies are scheduled for November 2002.

Neutron Diffraction Studies of Ceramics and CMCs: Using a new neutron spectrometer at LANSCE (SMARTS), some tensile creep experiments were performed on an *in-situ*-reinforced β-Si_3N_4 and a Si_3N_4-SiC_p composite. These experiments reached 1400°C, the highest temperature ever reached with neutron diffraction under loading, and 175 MPa. Although the beam time was not long enough to observe creep, the feasibility of the technique was demonstrated and the high temperature elastic properties (CTE and stiffness tensors) of the phases were obtained. The results are described in three papers submitted for publication. New ND experiments with longer beam time to assure creep are currently underway.

ADDITIONAL EXPLANATION and FUTURE STUDIES*

1. X-Ray Diffraction Studies of TBCs

a) Our main approach has been to develop capability to conduct an *in-situ* test whereby we monitor residual stress evolution in a TBC during a high-temperature exposure and/or while it is cycled between room and high temperature. This would be a unique test and would provide critical information about the failure mechanisms in TBCs.

b) Through discussions with David Clarke and literature search it became apparent to us that the growth of the TGO layer is one of the critical processes that determine the lifetime of a TBC. Residual stress measurements using piezospectroscopy (see e.g., V.K. Tolpygo, J.R. Dryden and D.R. Clarke, *Acta Mater*., **46**(3), 927-937 (1998)) have revealed very high growth stresses (around 5 GPa) in some TGOs. However, no clear understanding could be reached as to how these high residual stresses are generated since the stress measurements were conducted at room temperature and *ex situ*. Factors such as creep in the substrate and TGO, uncertainty about thermal residual stresses and so on could all influence the final stress value. In other words, there is an urgent need to understand the mechanisms of stress generation (and relaxation) in TBCs during high temperature exposure and cycling.

c) The basic experiment would then be the scanning of a cross-section of a TBC while it is heated using a small X-ray beam. Currently at APS, 10 μm beams are routinely generated; in a very near future 1 μm will be possible. The first experiment was attempted at APS in November 2001. The specimens were obtained from D. Clarke. All the runs were conducted in transmission mode using a digital image plate as an area detector. First, plan view runs were tried at room temperature. The experiment geometry is shown schematically in Fig. 1. The plan view results were not very useful since the very strong response of the substrate 'drowned' the data from the TBC (see Fig. 2). Next, a cross-sectional run was tried while the specimen was heated in air. This experiment was more successful in that different layers of the TBC system could be distinguished. Unfortunately, the furnace failed around 700°C. However, it was possible to obtain information about the CTE of the TBC layer (see Fig. 3). Here, the TBC patterns were fit as a tetragonal ZrO_2 with Y doping.

d) The main problem with the cross-sectional experiments is that, due to the circular sample geometry, the beam paths in the sample are not identical (see the marked beam paths in Fig. 1 for this geometry – the red line). This leads to differences in beam absorption. As a result, large errors in strain data are obtained. To correct this problem, specimens with a constant depth are needed so that the beam will traverse equal paths in every location. Such specimens are currently being made by D. Clarke. Note that, due to possible damage during handling, circular specimen cannot be easily cut into desired geometries to be used in these XRD runs. Instead, it is necessary to start with substrates of desired geometry and then grow the TBC on top of them.

e) Future Plans:

- A new furnace with IR heaters was procured by APS. IR heaters were chosen to avoid oxidation of the heating elements (as experienced with the previous run) and provide enough open space

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* Acronyms: ANL: Argonne National Laboratory; APS: Advanced Photon Source (at ANL); CIT: California Institute of Technology; CMC: ceramic matrix composite; CTE: coefficient of thermal expansion; FEM: finite element method (or modeling); LANL: Los Alamos National Laboratory; LANSCE: Los Alamos Neutron Science Center; NPD: neutron powder diffraction or the Neutron Powder Diffractometer (at LANSCE); ORNL: Oak Ridge National Laboratory; SMARTS: Spectrometer for Materials Research at Temperature and Stress (at LANSCE); SR: synchrotron radiation; TBC: thermal barrier coating; TGO: thermally grown oxide (in a TBC); XRD: X-ray diffraction.

around the specimen for X-rays to access it. The new furnace was fully characterized for temperature uniformity. It is now being adapted for TBC experiments.

- Cross-sectional XRD runs on 'model' TBC systems will be conducted at APS in November 2002. Several specimen types are envisioned: (i) complete TBC systems (with or without TGO present and at various stages of their lifetime) for residual stress measurements as a function of temperature; (ii) a bond coat specimen to be oxidized *in situ* while under X-rays (to monitor the growth of the TGO).
- Room temperature measurements of surface residual stresses using low-energy XRD (at Caltech) and piezospectroscopy at UCSB. These will complement the APS data.
- Modeling of residual stresses in TBCs. Both analytical and finite element modeling will be attempted to interpret the experimental data and obtain further predictions.
- One of the critical parameters in determining the residual stresses is the value of the elastic constants of various phases present in a TBC system. Simple loading (tension/compression or bending) experiments are planned at Caltech and APS while exposing specimens to X-rays. This will yield the diffraction elastic constants (incorporating the intergranular effects). Such data is more valuable in calculating residual stresses from the lattice strain data obtained by XRD.
- We also found that small angle X-ray scattering (SAXS) can provide information about pore structure, texture and size in TBCs. SAXS can be conducted simultaneously with a 'regular' XRD run to see how porosity evolves while the TBC is exposed to high temperature. Such an experiment will be attempted soon.
- Similar studies are also possible with samples to be provided by NASA Glenn.

2. X-Ray Diffraction Studies of CMCs

a) The main impediment in studying deformation and damage evolution in CMCs has been the lack of suitable model specimens. So far two different CMCs were obtained from NASA Glenn. The first sample was a 'microcomposite' with a single SiC (SCS-6) fiber embedded in a SiC matrix (provided by G. Morscher). It was run at APS using a 100x100 μm^2 X-ray beam in transmission mode so that matrix and fiber could be distinguished (as they have the same phase). The only way this could be done was by using diffracted beam slits allowing the definition of a three-dimensional sampling volume. This was demonstrated in spring; the extensive experiments are planned for the fall.

b) The second CMC had a Si_3N_4 matrix and SiC fibers (provided by S. Farmer). It was plate-like with five rows of unidirectional fibers. Unfortunately, both the α and β phases of Si_3N_4 appear to be present in the matrix leading to significant overlap with the SiC reflections. However, it was possible to isolate several SiC peaks by the use of an analyzer crystal at APS. The challenge now is to cut small samples out of the plate to use them in tension experiments at APS. Ideally, it would be good to obtain a sample with a single row of fibers; however, this will not be an easy task as the fiber rows are closely spaced. Laser cutting will be tried due to its good spatial resolution. In sum, there is still a strong need for model CMC samples either with a single fiber or with a single row of fibers, but definitely with different phases in fibers and matrix. These will be relatively easy to study in terms of damage evolution.

c) In the meantime, additional XRD techniques were explored. An especially promising one was identified in microtomography combined with diffraction. Microtomography is basically the process of obtaining a number of radiographic images from a specimen [1,2,3]. Using special software, these images are re-constructed to obtain a three-dimensional internal structure of the specimen. The high coherence of synchrotron X-ray beams allows dramatic increases in spatial resolution (currently down to 0.5 μm) since both phase and absorption contrast can be combined. This method has recently been combined with strain measurements to obtain a detailed description and quantification of damage

evolution in a Ti-SiC composite [2]. It offers a tremendous potential for damage evolution studies in coatings and CMCs. Using this technique, one can obtain information about matrix cracking, debonding at interfaces, crack opening displacement, bridging stresses, strain profiles along fibers (shear lag profiles), void formation, etc. in CMCs as well internal porosity and cracking in TBCs. Although this technique is currently used in Europe, no equipment exists in the US to perform similar studies. In collaboration with APS personnel, a special loading fixture (see Fig. 4) was designed and built at Caltech that will allow the first combined microtomography and diffraction experiments in this country. This fixture is already adapted to the setup at APS and first CMC experiments are planned for the later part of November.

d) Future Plans:

- The emphasis with XRD studies of CMCs will be on microtomography + diffraction. The first experiments in July demonstrated the potential of the technique (Fig. 5). Additional beam time is scheduled for late November to continue with this study. The above mentioned CMCs will be the first to study. A typical experiment will involve imaging a composite under tensile stress. Applied stress levels will be increased systematically in small steps. For a given specimen, several loading-unloading cycles will be needed. This way, data about evolving hystereses can be collected so that a direct comparison with 'traditional' methods can be made while additional *in-situ* data is obtained from imaging and diffraction. Based on preliminary studies at Sector 2 of APS, it is estimated that a complete damage evolution study for one sample will last about a day. This will also include detailed strain measurements around some critical regions identified by microtomography. From the appearance and growth of cracks, it will be known whether the cracks deflect along the interface between fiber and matrix. The amount of crack opening, debond lengths along interface and the spacing of cracks will indicate whether the interface is strong or weak. In addition to mechanics models, such information will be of utmost use for refinement of processing methods, for example, if samples processed in different ways possess different interface characteristics which require tailoring based on the ultimate application. A long term goal in this study is to perform all of the above investigations at higher temperatures. This is important since deformation mechanisms strongly depend on temperature, e.g., the BN and C coatings applied to fiber surfaces to control interface strength degrade at higher temperatures drastically modifying the composites behavior. There are plans to later add infrared heating elements to the loading fixture designed for this study.
- Depending on the outcome of the microtomography experiments, additional XRD runs are planned with the current CMCs for this fall using small X-ray beams and diffracted beam slits and/or analyzer crystals. These experiments are estimated to allow a sampling volume of 50x50x100 μm^2 since the grain size in both phases present in those CMCs is rather small. Again, tensile loading will be applied at the same time to monitor damage evolution. It is also possible to employ an area detector which will yield the two-dimensional strain tensor. These will be the first ever damage evolution studies in CMCs with such a small spatial resolution while investigating all phases present. (There have been some optical studies, e.g., with micro Raman, that reached a smaller sampling volume, but these studies did not allow the investigation of all phases.)

3. Neutron Diffraction Studies of Ceramics and CMCs

a) As part of the commissioning of the SMARTS diffractometer at LANSCE, some tensile creep experiments were performed on a commercial *in-situ*-reinforced β-Si_3N_4 (AS800 from Honeywell) and a Si_3N_4-SiC_p composite (also from Honeywell). These experiments reached 1375°C for AS800 and 1400°C, the highest temperature ever reached with neutron diffraction under loading, for the composite. The maximum tensile stress was 175 MPa. Although the beam time was not long enough

to observe creep, the feasibility of the technique was demonstrated and the high temperature elastic properties (CTE and stiffness tensors) of the phases were obtained (see Figs. 6-9). First, the heating data was used to calculate the CTE tensors of phases using individual reflections in a redundant manner. The calculation of CTE is more accurate this way (compared to the calculation of the lattice constants first) since less error is propagated [4]. The result for AS800 is shown below:

$$\alpha_{ij} = \begin{bmatrix} 3.50 & 0 & 0 \\ 0 & 3.50 & 0 \\ 0 & 0 & 4.06 \end{bmatrix} (x10^{-6} 1/K)$$

b) The high-temperature hold under stress was not apparently long enough to observe creep in each specimen (Figs. 7, 9). However, the lattice strain data obtained under loading at high temperature was used in modeling the elastic behavior of phases. For AS800, an elastic self-consistent model was developed that allowed the calculation of its stiffness tensor at 1375°C as shown below:

$$C_{ij} = \begin{bmatrix} 460 & 160 & 240 & 0 & 0 & 0 \\ 160 & 460 & 240 & 0 & 0 & 0 \\ 240 & 240 & 310 & 0 & 0 & 0 \\ 0 & 0 & 0 & 140 & 0 & 0 \\ 0 & 0 & 0 & 0 & 140 & 0 \\ 0 & 0 & 0 & 0 & 0 & 150 \end{bmatrix} (GPa)$$

As far as we know, this is the first time the high-temperature stiffness of Si_3N_4 has been determined. It is also the first time self-consistent modeling has been applied to this material. The potential of the model has been shown. Once new data is collected with enough creep (in the fall), another version of the model, the viscoplastic self-consistent model, will be applied. This model is more appropriate for texture evolution and creep deformation. The current data from AS800 is described in two publications recently submitted [5,6].

c) Similar results were also obtained from the composite. Unfortunately, the peak intensity was not high enough for every reflection; therefore, the self-consistent modeling cannot be applied here. However, since the deformation was basically elastic in all phases, a simple Eshelby model was developed and it is seen to fit the data well (Fig. 9). The details are described in two papers [6,7].

d) Future Plans:

1. Currently, new neutron experiments are being performed attempting to increase the high-temperature hold times so that creep can be observed. Another grade of Si_3N_4 (GS44) is used as it is easier to creep than AS800. In addition, several specimens were crept outside the neutron beam for long periods and then a loading/unloading experiment will be performed to obtain their elastic stiffness as well as to measure creep-induced texture. This way, the neutron beam time will not be prohibitively long.
2. Collaborations were established with leading researchers in the field of ceramic creep: S. Wiederhorn at NIST and D. Wilkinson at McMaster University. Dr. Wiederhorn is interested in creep studies of monolithic Si_3N_4 and is offering different grades. Dr. Wilkinson is studying the creep of an Al_2O_3-matrix / SiC-whisker composite. His samples will be tested as well if there is enough beam time.
3. Complementary ultra-small angle X-ray scattering (USAXS) [8] experiments are also planned on select specimens to quantify creep cavitation evolution. These will be done at Argonne, at the APS. The first experiment is planned for the fall. We note that the recently developed USAXS method can "see" cavities in the 100 Å to 5 μm range. As a result of these investigations, we expect to obtain detailed knowledge about the deformation mechanisms in structural ceramics

combined with new "realistic" models with predictive capability. For instance, it will be interesting to compare the deformation mechanisms in ceramics with "dry" grain boundaries [9]. to those in ceramics with viscous amorphous films.

Publications

1. G. A. Swift, E. Üstündag, B. Clausen, M.A.M. Bourke, H. T. Lin and C. W. Li, "High-Temperature Elastic Strain Evolution in Si_3N_4-Based Ceramics," in print: *Adv. X-Ray Anal*., **46** (2002).
2. G. A. Swift, E. Üstündag, B. Clausen, M.A.M. Bourke, H. T. Lin and C. W. Li, "High-Temperature Deformation of Silicon Nitride and Its Composites," submitted to *Ceram. Trans*., (2002).
3. G. A. Swift, E. Üstündag, B. Clausen, M.A.M. Bourke and H. T. Lin, "High-Temperature Elastic Properties of an *In-Situ*-Reinforced Si_3N_4," submitted to *Appl. Phys. Lett.* (2002).

Presentations

1. G. A. Swift, "STRAIN AND MICROSTRUCTURE EVOLUTION IN SI_3N_4 DURING HIGH-TEMPERATURE DEFORMATION," 26th Cocoa Beach Conference, Cocoa Beach, FL, January 2002.
2. E. Ustundag, "HIGH-TEMPERATURE DEFORMATION OF SILICON NITRIDE AND ITS COMPOSITES," Annual Meeting of the American Ceramic Society, St. Louis, MO, May 2002.
3. E. Ustundag, "HIGH TEMPERATURE ELASTIC STRAIN EVOLUTION IN Si_3N_4-BASED CERAMICS," 51st Denver X-Ray Conference, Colorado Springs, CO, August 2002.
4. G. A. Swift and R.C. Rogan, "MULTISCALE CHARACTERIZATION OF CERAMICS USING DIFFRACTION," invited poster, Gordon Conference on Ceramics, August 2002.
5. E. Ustundag, "INVESTIGATION OF COMPOSITE DEFORMATION USING DIFFRACTION," invited seminar, Dept. of Materials Science and Engineering, University of Illinois, Urbana, IL, September 2002.

Key Personnel

Dr. Ersan Ustundag (PI): Assistant professor of Materials Science, California Institute of Technology.

Dr. Irene J. Beyerlein: Technical staff member, Theoretical Division, Los Alamos National Laboratory. Dr. Beyerlein has developed many of the micromechanics models to be used in this work.

Dr. Bjorn Clausen: Senior postdoctoral scholar, Department of Materials Science, California Institute of Technology. Dr. Clausen has a profound background in the use of ND and XRD in internal stress studies, structural analysis, FEM and self-consistent modeling.

Geoffrey A. Swift: Third year graduate student, Department of Materials Science, California Institute of Technology.

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5. G. A. Swift, E. Üstündag, B. Clausen, M. A. M. Bourke and H. T. Lin, submitted to *Appl. Phys. Lett*. (2002).
6. G. A. Swift, E. Üstündag, B. Clausen, M. A. M. Bourke, C. W. Li and H. T. Lin, submitted to *Ceram. Trans.* (2002).
7. G. A. Swift, E. Üstündag, B. Clausen, M.A.M. Bourke, H. T. Lin and C. W. Li, "High-Temperature Elastic Strain Evolution in Si_3N_4-Based Ceramics," in print: *Adv. X-Ray Anal*., **46** (2002).
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9. S.M. Wiederhorn, B.J. Hockey and J.D. French, *J. Eur. Ceram. Soc*., **19**, 2273-2284 (1999).

FIGURES

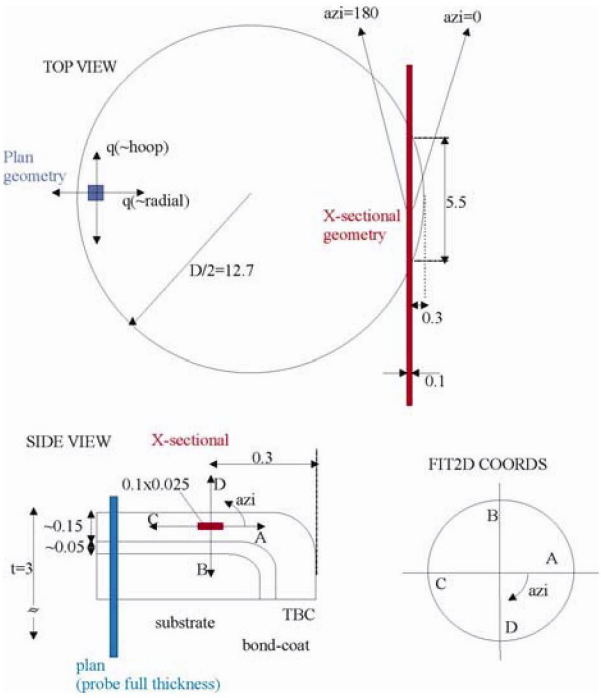

Figure 1. Schematic of experimental geometries used in high-energy XRD runs of TBCs at APS. The X-ray beam (blue in plan view experiment, red in cross-sectional run) is shown with respect to the specimen orientation. Since the area detector measures the two-dimensional strain tensor, the directions of the strain components are also indicated for each case. All dimensions are in mm.

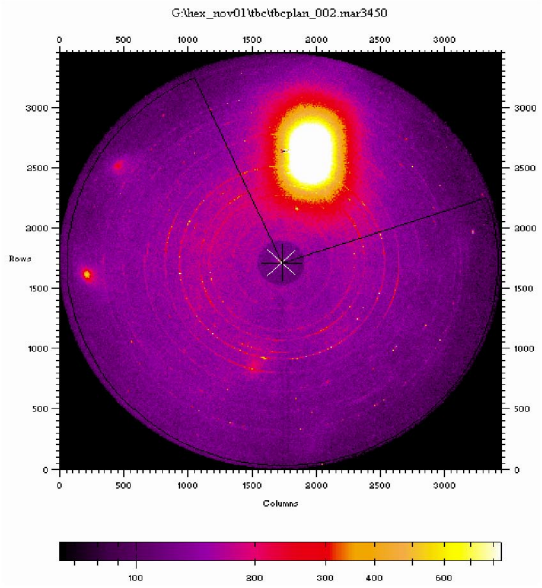

Figure 2. XRD data collected on digital image plate of a TBC sample in plan view; here we see that the large-grain substrate peaks (the large bright spots) dominate the pattern.

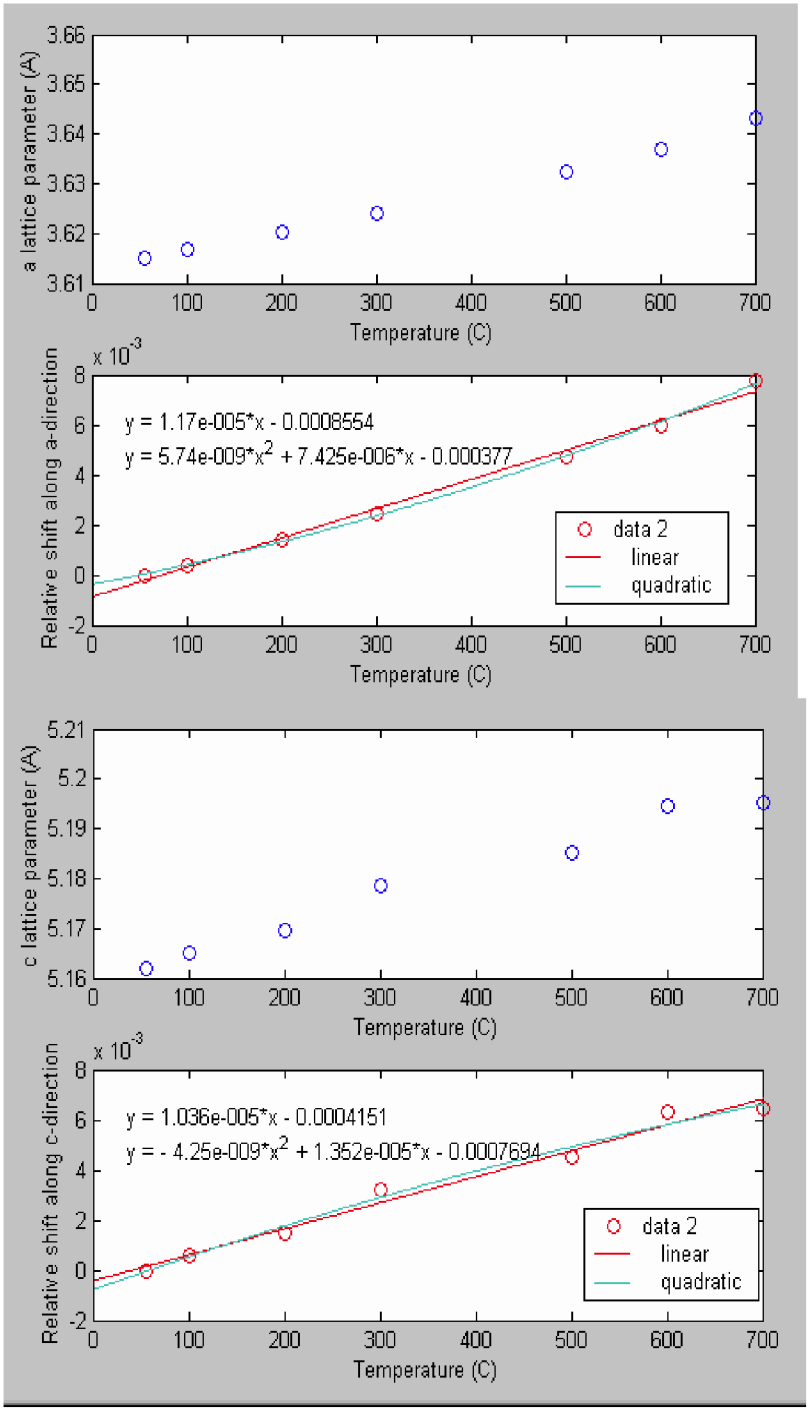

Figure 3. Derived lattice parameters of TBC vs. temperature: the top is *a*, the bottom is *c* of tetragonal ZrO_2 (doped with Y). The linear CTE values were found to be around 10 to 12 x 10^{-6} 1/K for each parameter.

Figure 4. Special loading fixture for microtomography experiments. It was designed by G. Swift and built at Caltech. The right grip is pulled by turning the nut shown on the right end of the fixture. The main load-bearing component is a plexiglass tube since it has to allow the penetration of X-rays from all directions while the specimen is rotated. This fixture is already adapted to the goniometer at Sector 2 of APS where the preliminary experiments were performed in late July 2002.

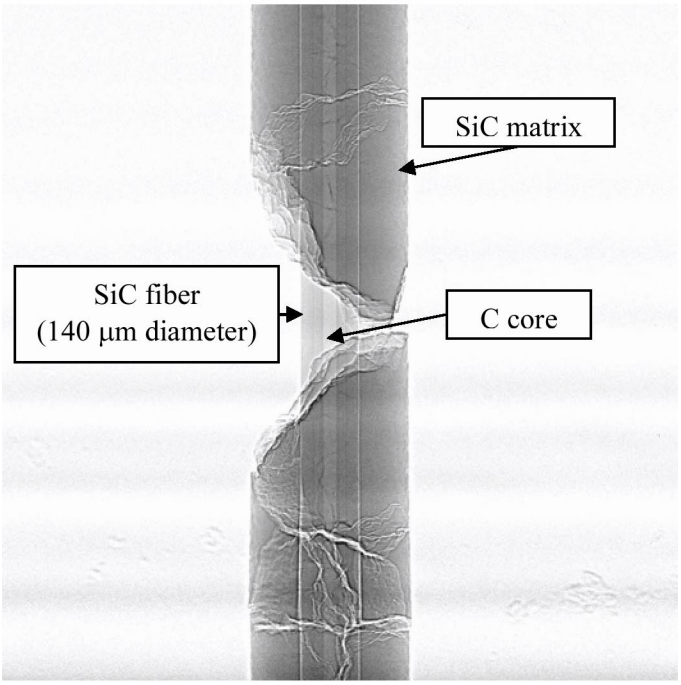

Figure 5. X-ray tomograph of a SiC/SiC 'microcomposite' collected at APS.

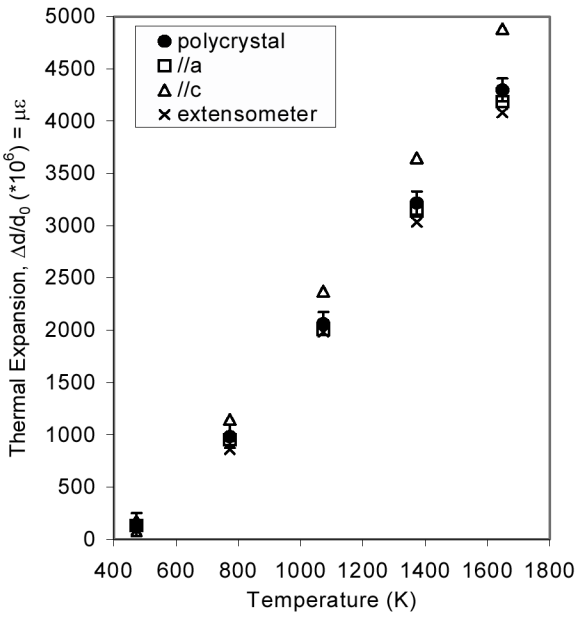

Figure 6. Thermal expansion of β-Si_3N_4 (AS800 from Honeywell). The lattice constants were determined from Rietveld analysis of neutron diffraction patterns. The polycrystal 'average' was obtained from *(2a+c)/3*.

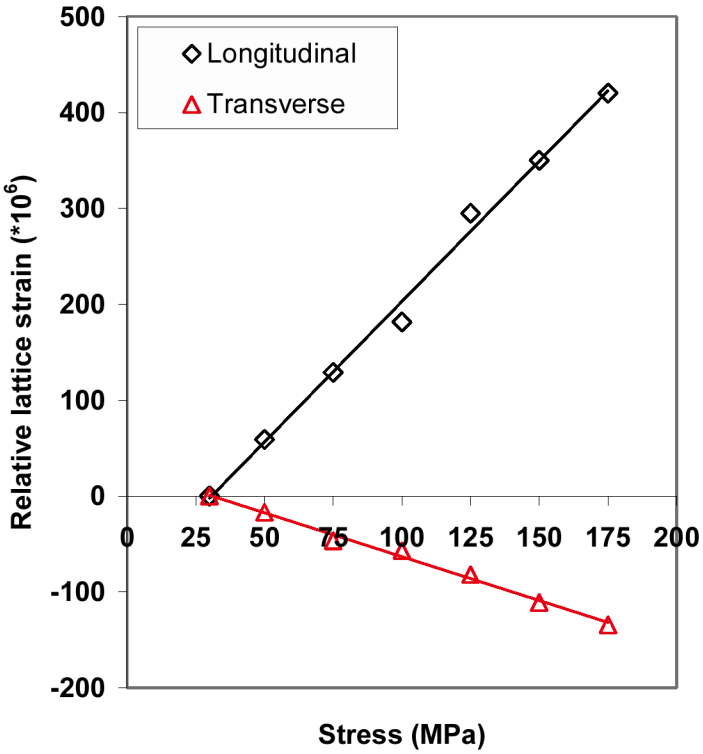

Figure 7. Tensile stress-strain curve of β-Si_3N_4 (AS800 from Honeywell) at 1375°C obtained from neutron diffraction. A nearly elastic behavior is observed.

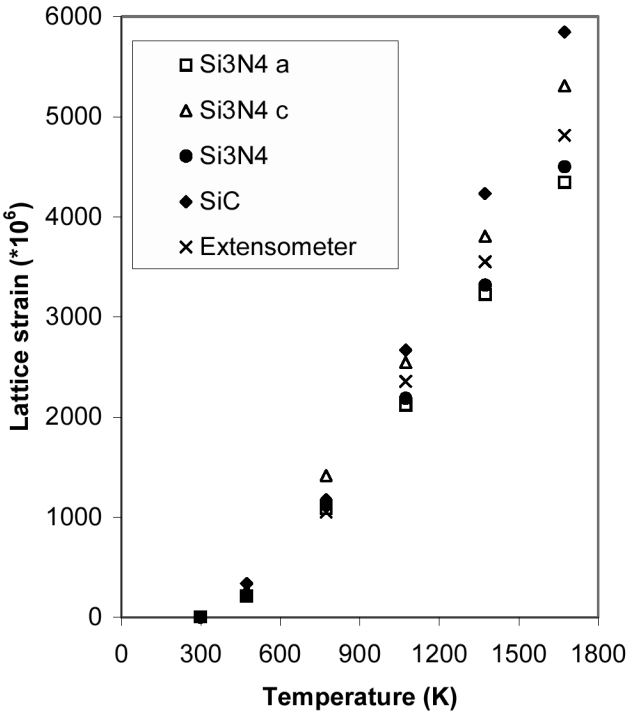

Figure 8. Thermal expansion of a β-Si_3N_4–SiC_p composite (from Honeywell). The lattice constants were determined from Rietveld analysis of neutron diffraction patterns. The polycrystal 'average' of Si_3N_4 was obtained from *(2a+c)/3*.

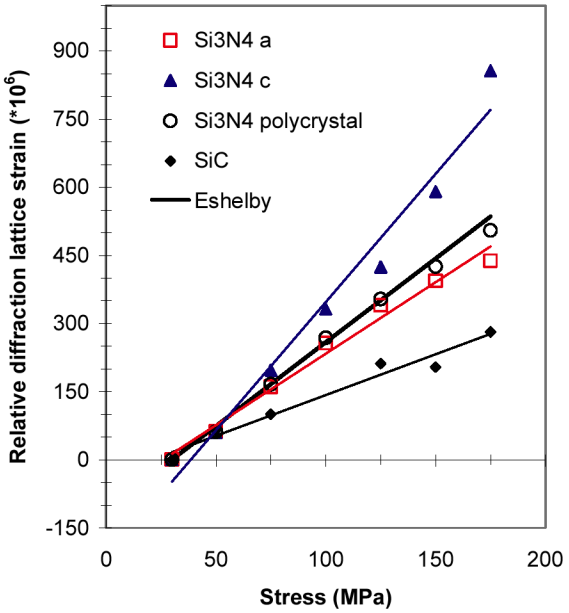

Figure 9. Tensile stress-strain curve of a β-Si_3N_4–SiC_p composite (from Honeywell) at 1400°C obtained from neutron diffraction. A nearly elastic behavior is observed. The polycrystal average behavior of both phases can be described with an elastic Eshelby model.

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The premise of this project is a comprehensive study that involves the in-situ characterization of advanced coatings and composites by employing both neutron and x-ray diffraction techniques in a complementary manner. The diffraction data would then be interpreted and used in developing or validating advanced micromechanics models with life prediction capability. In the period covered by this report, basic work was conducted to establish the experimental conditions for various specimens and techniques. In addition, equipment was developed that will allow the in-situ studies under a range of conditions (stress, temperature, atmosphere, etc.).

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