Accepted Manuscript

Microbeam, Timing and Signal-Resolved Studies of Nuclear Materials with Synchrotron X-ray Sources

Gene E. Ice, Eliot D. Specht

PII: S0022-3115(11)00932-9
DOI: 10.1016/j.jnucmat.2011.10.038
Reference: NUMA 46161

To appear in: *Journal of Nuclear Materials*

Received Date: 1 March 2011
Accepted Date: 22 October 2011

Please cite this article as: G.E. Ice, E.D. Specht, Microbeam, Timing and Signal-Resolved Studies of Nuclear Materials with Synchrotron X-ray Sources, *Journal of Nuclear Materials* (2011), doi: 10.1016/j.jnucmat.2011.10.038

This is a PDF file of an unedited manuscript that has been accepted for publication. As a service to our customers we are providing this early version of the manuscript. The manuscript will undergo copyediting, typesetting, and review of the resulting proof before it is published in its final form. Please note that during the production process errors may be discovered which could affect the content, and all legal disclaimers that apply to the journal pertain.

ACCEPTED MANUSCRIPT

Microbeam, Timing and Signal-Resolved Studies of Nuclear Materials with Synchrotron X-ray Sources

Gene E. Ice, and Eliot D. Specht
Materials Science and Technology Division
Oak Ridge National Laboratory, Oak Ridge, TN, 37831

Abstract
The development of ultra-brilliant synchrotron x-ray sources enables characterization methods that are particularly important for nuclear materials. Here we discuss emerging synchrotron methods with unprecedented signal-to-noise, spatial and time resolution. Microprobe methods are discussed that extend virtually any x-ray characterization measurement to ultra-small sample volumes. This ability is critical to resolve heterogeneities in nuclear materials and for studies on volumes with vastly lower activity than are needed for traditional x-ray characterization. Specific methods discussed include microdiffraction for the characterization of local crystal structure and micro-spectroscopy techniques that allow for characterization of elemental distributions with sensitivity for daughter products, oxidation states and diffusion through buffer layers. Opportunities are also discussed that exploit the high brilliance and pulsed nature of synchrotron radiation to reduce backgrounds from sample radiation and to study materials dynamics.

1.0 Introduction
Despite the recent Fukushima accident, there is widespread –if grudging- acceptance that nuclear power offers advantages that cannot be matched by alternatives. As a result there is an urgent need to reimagine nuclear power to exploit modern materials that simply did not exist when current nuclear plants were designed. Busby [1] has pointed out, that modern materials offer the potential for lower lifetime costs, higher efficiency, more flexibility, simplified designs and improved safety. A major obstacle to the deployment of new materials and concepts however, is the difficulty of certifying materials and systems. Fortunately, a range of characterization tools developed since the accidents at Three Mile Island and Chernobyl offer hope for unprecedented materials characterization to accelerate deployment of new nuclear technologies. Scanning surface probes [2], electron microscopy including electron beam backscattering diffraction [3], and nano-indentation [4], have all either emerged as important new characterization tools or have made major advances in the last three decades.

The development of ultra-brilliant 3rd and now 4th generation synchrotron sources [5,6] is particularly significant for nuclear materials. Synchrotron X-ray characterization methods can provide unprecedented nondestructive measurements of local chemistry, oxidation state, crystal structure and defect densities. Moreover, synchrotron sources now routinely provide 12-14 orders-of-magnitude better time-average performance than conventional laboratory X-ray sources, with an additional 3 to 4 orders of magnitude in pulsed performance [7]. Novel 4th generation linear accelerator-based X-ray sources [6] offer an additional 10-12 orders of magnitude pulsed brilliance and generate coherent beams with new characterization capabilities.

2.0 Synchrotron-based Characterization Opportunities for Nuclear Materials

ACCEPTED MANUSCRIPT

As a result of the opportunities afforded by ultra-brilliant synchrotron radiation sources, at least three nuclear-materials research directions have recently emerged. A first approach is through the use of dedicated instrumentation designed to contain radioactive materials and maximize the signal-to-background (Section 2.1). For example, highly intense and collimated beams can be used to overwhelm sample-generated backgrounds and wavelength-dispersive optics can be used to shield sensitive detectors and to provide high resolution (Fig. 1A). The pulsed nature of synchrotron sources can also be exploited to improve signal-to-noise (Fig. 1B) or to follow materials evolution on ultra-fast time scales (Section 2.3). Through the use of spatially-resolving microprobes, or full-field imaging methods, heterogeneities can be resolved in nuclear materials (Fig. 1C).

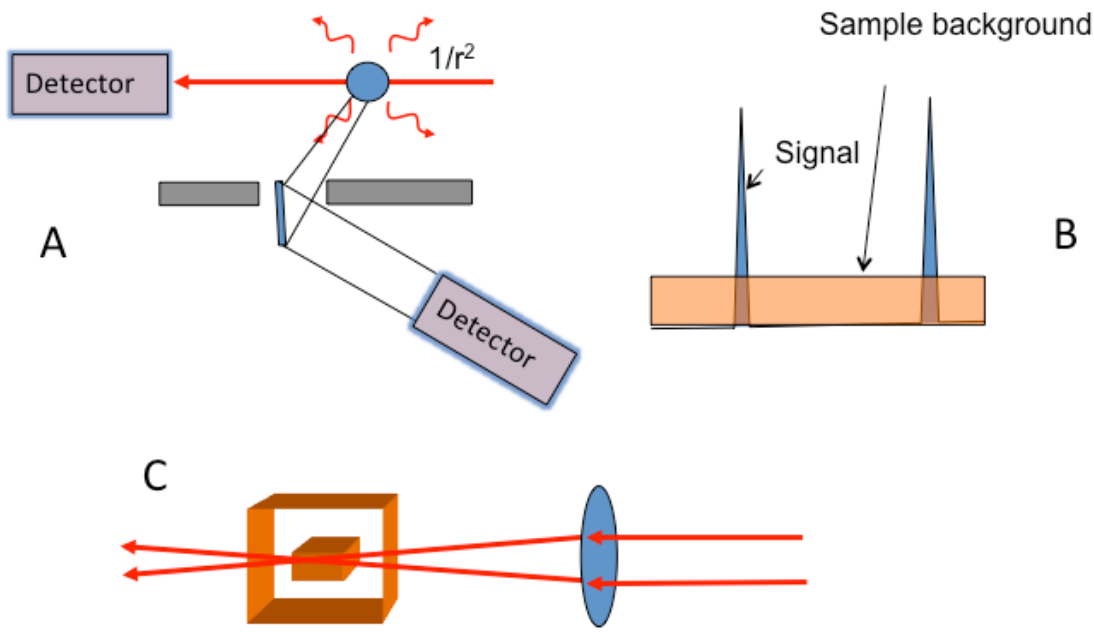

Fig. 1. Schematic of the advantages offered by ultra-brilliant synchrotron hard X-ray beams. (A) High signal to overwhelm sample-introduced backgrounds and to efficient use crystal optics to shield detectors and gain the best signal-to-noise; (B) timing to improve background and time resolution; and (C) focused beams to resolve heterogeneities and for characterization of small samples.

A second direction for synchrotron studies of nuclear materials is with microbeam analysis of tiny irradiated samples (Section 2.2). With activated materials, the adoption of tiny samples has the important advantage that it can vastly lower total activity compared to traditional specimens (Fig. 2). This will simplify and accelerate sample preparation and handling, allow for combinatorial studies with reduced waste management issues, will reduce sample-generated backgrounds and will greatly expand access to synchrotron characterization tools. The use of tiny samples however, brings important technical concerns about size effects in materials behavior. Fortunately, as described in section 2.2, a consensus about the role of size and defect density is emerging that can help advance the use of tiny samples and can help build confidence in extending the results from tiny specimens to actual materials systems.

ACCEPTED MANUSCRIPT

Time-resolved measurements are a third direction made possible by ultra-brilliant synchrotron sources. Indeed synchrotron sources enable completely novel experimental studies of defect interactions and dynamics. This realization is at the core of the Center for Defect Physics [8], an Energy Frontier Research Center funded by the United States Department of Energy. Within this center are two signature experiments only now possible due to the availability of new synchrotron capabilities. In one experiment, the interactions of discrete defects will be characterized and in another experiment the dynamics of the first picoseconds of radiation cascade events will be characterized. Both experiments are of fundamental importance for understanding how nuclear materials behave and evolve with time.

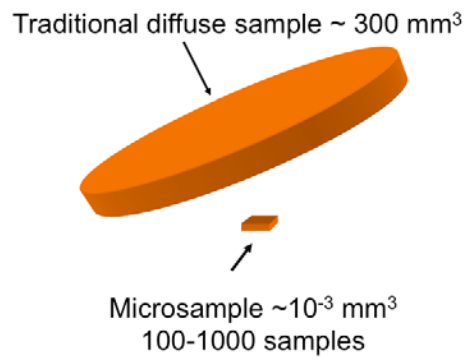

Fig. 2. With samples on the order of 10^{-3} mm^3 the activity of irradiated materials is reduced by 6 orders of magnitude compared to cm^3 samples. For example, diffuse X-ray scattering samples are typically 300 mm^3 or larger. With microbeams however, a $10^{=3}$ mm^3 sample can have hundreds to thousands of useful sample volumes.

Below we describe these three approaches and present some example characterization experiments on nuclear and surrogate nuclear materials made using third-generation synchrotron source and speculate about future experimental directions made possible by high-brilliance synchrotron sources including new 4th generation synchrotron sources.

2.1 Synchrotron Instrumentation for the Study of Engineering-Scale Samples

One straightforward solution for characterizing nuclear materials at a synchrotron source is through the construction of specially-designed beamlines with the necessary infrastructure for handling radioactive samples and for containment of the activated materials from the rest of the synchrotron source. A first example of this approach is the MARS beamline at SOLEI [9]. This beamline has a specially-designed air filtration system and instrumentation optimized to suppress sample-generated backgrounds. The beamline is designed to handle a total sample activity (including stored samples) of up to 185 GBq with a maximum of 18.5 GBq (0.5 Ci) per sample. The beamline also includes a suite of instrumentation needed for handling and preparing radioactive specimens including glove boxes, cryostats, electrochemical cells, furnaces, high pressure diamond anvil-type cells, specially shielded containment and multi-barrier confinement.

The initial experimental capabilities include high-resolution x-ray powder diffraction (HRXRD), transmission X-ray Diffraction (XRD) and x-ray absorption spectroscopy (XAS). The high-resolution powder diffraction instrument uses multiplexed crystal optics similar to the crystal optics illustrated in Fig. 1A to provide high-resolution with low background. The XAS instrument has absorption detectors, which are located far from the sample (Fig. 1A). This exploits the highly collimated incident beam and results in the detectors subtending very small solid angles from the sample position.

ACCEPTED MANUSCRIPT

At MARS, the beam size can be adjusted from ~15 μm to 300 μm. This makes the beamline ideal for ensemble-average measurements of materials with sensitivity to how average composition, chemistry and phase content change from point-to-point. First measurements have shown extraordinary sensitivity to minor phases, texture and defect broadening of the diffraction lines [10]. Of course, the importance of synchrotron characterization of nuclear materials has also been recognized elsewhere and several recent symposia have been held to explore synchrotron-enabled options for nuclear materials studies [11,12].

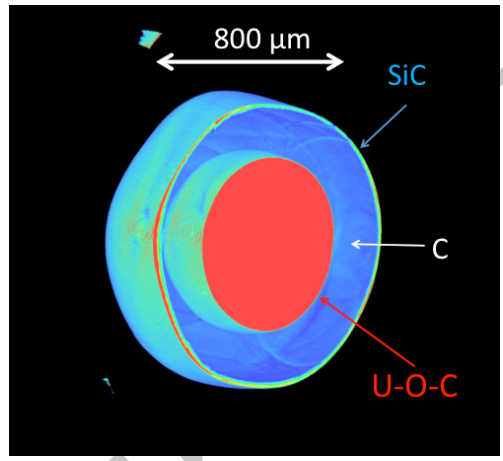

Fig. 3. Absorption and edge contrast tomography used to measure the structure of a TRISO fuel particle. Note that these measurements were made on a laboratory source with ~1.2 mm resolution. They can can now be made in the same time with 30 nm resolution at a synchrotron source.

Beyond ensemble averages, materials are inherently inhomogeneous with structure on many different length scales. At the atomic scale, point defects including interstitials, vacancies and site substitutions are critical to materials properties including diffusion and dislocation mobility. At the mesoscale multiple phases, precipitates, voids, dislocation networks, grains, grain-boundaries and layered structures all effect materials behavior. Characterization with spatial resolution is therefore essential to unravel the role of heterogeneity on materials behavior and x-ray characterization methods that include spatial resolution are particularly important since they allow for nondestructive characterization with minimum sample preparation.

A good example of the potential of synchrotron radiation to contribute to our understanding of inhomogeneity in nuclear materials, is some recent work at ORNL where *laboratory-based* x-ray tomographic methods have been used to study the as-built structure of TRISO fuel particles and more recently, changes in the structure of a TRISO fuel particle during irradiation. The laboratory-based instrumentation provides spatial resolution of about 1.2 μm with good sensitivity to defects in the SiC shell and some sensitivity to defects in the carbon buffer layers. The enabling technology for working with irradiated particles is a special transport mechanism designed to provide shielding from the particle during transport and to be easily mounted and opened on the tomography instrument to allow the x-ray beam to pass through the sample and onto the detector. Measurements show important changes in the TRISO elemental distribution and shell structure after irradiation. A typical absorption

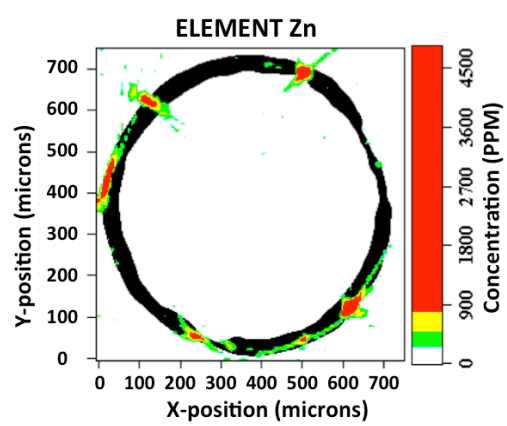

Fig. 4 Elemental map of Zn in the shell of a TRISO SiC shell. The figure is adapted from Naghedolofeizi et al. [17].

ACCEPTED MANUSCRIPT

tomography reconstruction of an unirradiated TRISO fuel particle is shown in Fig. 3.

At a synchrotron, tomographic methods can be greatly extended beyond what is possible with a laboratory source due to the intensity of the synchrotron source and the opportunity to tune wavelength. For example, if the incident beam wavelength is adjusted above and below an elemental absorption edge, information about the local chemistry can be determined [13]. Spatial resolution of about 30 nm has been demonstrated with a synchrotron source and recent measurements have also shown the ability to map the oxidation state distribution in three dimensions using x-ray near edge tomography [14]. At a synchrotron, the partial coherence of the beam makes tomography sensitive to subtle changes in the refractive index (phase contrast). This is particularly useful for observing edges in low Z materials and can be used to simultaneously observe structure in low Z and high Z materials [15]. As a consequence, enhanced sensitivity to changes in the carbon buffer layers is possible.

Another technique that is only practical on a synchrotron is microfluorescence tomography. This is an extension of x-ray fluorescence tomography [16], which provides 3D information on trace element distributions. An example experiment is illustrated in Fig. 4. In this experiment, irradiated TRISO fuel particles were laser drilled and then acid etched to remove the nuclear core and the graphitic buffer layers [17]. This greatly reduced sample activity, which made it possible to study the remaining SiC shells on a standard x-ray microprobe beamline. The trace element content in the remaining SiC shells were characterized using a synchrotron submicron x-ray beam to test if daughter products were migrating through the shells. The measurements *did not* detect daughter products in the shells, but did find unexpected metal particles-presumably from the fabrication process. This sensitive technique can detect trace elements at the sub ppm level, but is relatively slow compared to full-field tomographic methods. With a specially designed beamline, the removal of the core and graphite would not be required to characterize trace elements in the SiC shell.

In addition to information on elemental composition and chemistry, X-rays are powerful probes of crystallographic structure with much better resolution than either neutron or electron probes. One method that has recently emerged from advanced synchrotron sources, is called 3D X-ray Diffraction Microscopy [18]. This method uses a singly focused beam to isolate crystal grains in the plane of the radiation. The sample is rotated to reach the Bragg condition multiple times for each grain. The Bragg spots observed at the detector are projections through the sample grain and the shapes and intensities of each Bragg reflection carries information about the grain. The Bragg reflections are reconciled by either triangulation or by space-filling algorithms to determine the crystal structure in the plane of the singly-focused radiation. The sample is then translated to build up a three-dimensional view of the crystal structure. Three-dimensional grain maps with a resolution of ~3 μm have been demonstrated, and because the method is relatively fast, the grain structure network can be observed as it evolves due to the sample environment. This method is well suited to nuclear materials, because the high-energy x-ray beam scatters in the forward direction which minimizes the solid-angle subtended by the detector which reducing the overall background arising from activated samples.

2.2 Small Sample Methods

ACCEPTED MANUSCRIPT

To synchrotron scientists, the obvious way to mitigate the special handling requirements of nuclear samples is by making the samples smaller. As illustrated in Fig. 2, small samples can have activities orders of magnitude lower than those of similarly prepared samples with typical engineering dimensions. Such low activity samples can be handled on virtually any beamline, which opens up hundreds of synchrotron beamlines for experiments with nuclear materials. The main issue with small samples is whether they are representative of actual materials used in nuclear applications. This issue is of great importance not only for nuclear materials, but also for all of materials science, where our understanding of materials is evolving from an understanding based on ensemble average properties to one based on more realistic models that include local fluctuations in chemistry, local structure and defect density.

Of particular importance is the emergence of an understanding of the transition from "small-sample" or "near-surface" behavior to "bulk-like" behavior. For example, Pang and co-workers [19] have recently reported measurements of dislocation activity as a function of distance from the sample surface. They find a transition from a universal surface behavior, to a crystallographically-sensitive bulk behavior that occurs a few microns below the sample surface. This measurement provides guidance as to when the sample size becomes so small that free surface effects becomes import.

Similarly, George and co-workers [20] have provided important insights into the transition from bulk-like properties to extraordinary small-sample properties based on sample size and defect density. They find that small single-crystal pillars with high defect densities behave very much like bulk specimens, whereas those same pillars with low defect densities can have strength approaching the theoretical strength of the material (without dislocations). Here again a clearer understanding is emerging about when small sample results can be generalized to large samples, and also how small samples might be prepared in such a way as to simulate their behavior in larger specimens. With these caveats, we describe some simple opportunities for studying small specimens with synchrotron radiation.

Intense small x-ray beams can be used to study small samples using most of the popular synchrotron characterization methods. For example absorption spectroscopy methods are well suited to microbeam analysis, and are included in the MARS beamline (Section 2.1). Absorption spectroscopy measurements are a set of well-established techniques for characterizing the local environment around specific elements. The so-called near-edge region which includes the X-ray absorption region within ~-+/-50 eV of an elemental absorption edge, is sensitive to of the oxidation state of the element. The extended edge region from 50-250 eV above the edge, provides information about the co-ordination number and bond distances. These techniques can be used to identify the local chemistry with specialized beamlines designed with achromatic focusing optics and tunable beams that localizes the measurements to small volumes, which can separate the contributions from distinct phases and/or identify chemistry changes across boundary layers.

Microdiffraction is another way to determine local crystal phase, orientation (texture), elastic strain and defect density. With one method capable of submicron 3D resolution, small X-ray beams are focused onto the sample, and the scattering from along the incident beam is resolved using a method called differential aperture microscopy [21]. This is a knife edge method that passes an absorbing wire near to the sample surface and uses triangulation to reconstruct the origin of intensity collected in an x-ray sensitive area

ACCEPTED MANUSCRIPT

detector. Three-dimensional images are built up by rastering the sample under the small probe beam and by scanning the differential aperture. This method is ideal for example, for identifying nanoscale phases at borders between fuel materials and cladding. With nanophase materials the diffraction patterns are similar to traditional powder diffraction- but with micron real-space resolution of the evolving phase and texture fractions. With larger grains, individual Bragg reflections are isolated and single-crystal methods can be used for data analysis [22].

As an extreme example, Specht and co-workers [23] have recently demonstrated the use of microdiffraction for single-crystal *diffuse* x-ray scattering measurements of individual grains in a polycrystalline sample. In this experiment, a submicron x-ray beam was used to probe a single grain in a 10 mm x 10 mm x 0.1 mm Fe foil. The sample was prepared remotely by ion deposition at liquid nitrogen temperatures. A specially designed sample stage allowed the sample to be transported to the beamline and mounted at liquid nitrogen nitrogen temperatures. This stage used a microfluidic gas expansion system and was practical in part because of the small sample size.

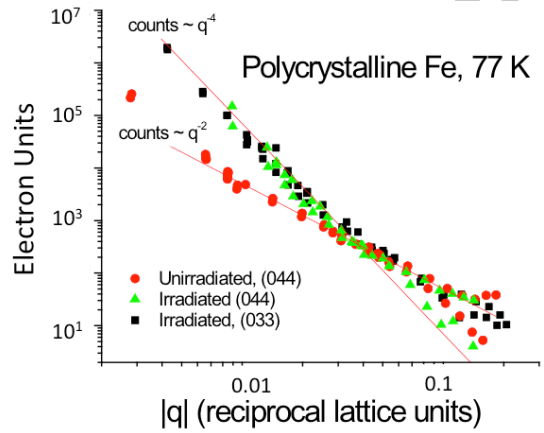

Fig. 5. Measurements of diffuse scattering in Fe grain with a submicron beam. The irradiated sample has significantly increased diffuse scattering (after Specht et al. [23].

A reciprocal-space volume was collected by varying x-ray energy while collecting images on an x-ray sensitive area detector. The x-ray probe sampled to a depth of about 17 microns. As shown in Fig. 5, the diffuse contribution introduced by defects is clearly resolved even with a tiny X-ray probe beam.

2.3 Time-resolved studies

Intense X-ray beams from synchrotron sources not only enable *spatially-resolved* studies of nuclear materials, but also enable *time-resolved* measurements. The time-scales of measurements made possible extend from femtosecond studies made possible by new 4th generation sources to real-time studies of evolving phases during materials processing with resolution at the second-to-hour time scale. Two interesting examples are studies of phase evolution during annealing, and the potential for radiation cascade studies that capture the first few picoseconds of evolving defect dynamics.

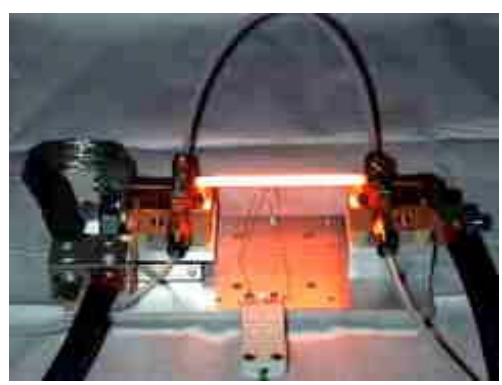

Fig. 6. In-situ measurement of oxide scale formation and stress development.

2.31 Slowly evolving structure

Nuclear materials evolve during initial processing and in service. It is essential to understand how phases, microstructures and defects evolve to suggest new approaches for improved properties and longevity. Recent in-situ experiments with synchrotron radiation demonstrate how intense X-ray beams can be

ACCEPTED MANUSCRIPT

used to advance our understanding of nuclear materials [24,25]. The research by Mayr et al. demonstrates how the influence of processing conditions can be followed at important time scales in steels using synchrotron radiation. This is but one example of a growing body of studies of the dynamics of phase evolution in materials. A 2003 study by Specht and co-workers [25] shows how in-situ measurements can help understand materials evolution and performance during service conditions. In these measurements, scale formation and residual stress evolution was observed at temperatures ranging from 1000-1200° C over time scales from minutes to hours (Fig. 6). The observed tensile stresses and stress dynamics challenged conventional models of growth stresses.

2.32 Sub-Picosecond Radiation Cascade Dynamics

Models of radiation cascades, predict the multiplication of defects in the first few picoseconds after an ion enters a sample, followed by recombination and evolution of the defect structure into a much less defective state. Although models have been used to predict the behavior of defects for more than 50 years, and although there is an extensive literature on post mortem studies of residual defects in materials after irradiation, there have been no tests of the critical first few picoseconds. The development of the Linear Coherent Light Source at Stanford now opens up femtosecond science with the ability to discern structural changes on the time scales needed to provide first-of-their kind tests of cascade theories. One of the signature experiments of the Center for Defect Physics [8] is a measurement of the diffuse scattering arising from single cascade events within the first few picoseconds. In this experiment, a single ion is directed onto a single-crystal sample. The ultra-intense x-ray pulse with less than 100 femtosecond pulse length captures a snap shot of the atomic lattice structure before atoms in the sample volume are disturbed by the x-ray pulse. Because of the power deposition over a very short time, the area probed by the x-ray beam is ablated and the single crystal must be moved to a fresh position where the next measurement can be made. Modeling has shown that such measurements are feasible at the LCLS and the necessary instrumentation has been assembled and is currently undergoing testing. These measurements will follow the development of the radiation cascade with sub 100 femtosecond resolution.

3.0 Conclusion

As described above, emerging synchrotron characterization tools offer important new opportunities for the study of nuclear materials. Specially-designed beamlines will allow for nondestructive measurements on nuclear materials with sensitivity to chemical, structural and defect distributions. These beamlines will include the infrastructure to handle irradiated samples and the instrumentation to suppress sample-generated backgrounds. In addition, because x-rays are penetrating probes, experiments can often be done on samples with little sample preparation and with the ability to resolve sample heterogeneity. Intense x-ray beams will also allow for characterization of small samples that would be impractical to study at engineering sizes. Here the orders-of-magnitude lower activity of micron-sized samples will allow for experiments on synchrotron beamlines with a range of techniques. Finally, timing experiments can be used to watch nuclear materials evolve over time scales ranging from sub picoseconds to hours. Collectively the new characterization opportunities made possible by ultra-brilliant synchrotron sources will provide guidance as new materials systems are evaluated for deployment into the nuclear infrastructure.

ACCEPTED MANUSCRIPT

ACCEPTED MANUSCRIPT

4.0 Acknowledgments

Research supported by the U.S. Department of Energy, Basic Energy Sciences, Materials Sciences and Engineering Division and performed in part on beamlines 2-ID, 7-ID and 34-ID at the Advanced Photon Source, (APS). The APS is a DOE-BES user facility.

5.0 References

[1] J. T. Busby, Economic benefits of advanced materials in nuclear power systems, J. Nucl. Mat. 392 (2009) 301-306.

[2] T.C. Shen, Role of scanning probes in nanoelectronics: A critical review, Surf. Rev. and Lett. 7 (2000) 683-688.

[3] A.J. Schwartz, M. Kumar, and B.L. Adams, Electron Backscatter Diffraction in Materials Science, Kluwer Academic New York, NY (2000).

[4] W. C. Oliver, G. M Pharr, Nanoindentation in materials research: Past, present and future, MRS Bulletin **35** (2010) 897-907.

[5] D.M. Mills, Third Generation Hard X-ray Synchrotron Radiation Sources, John Wiley & Sons, New York, 2002.

[6] W. A. Barletta, J. Bisognano, J.N. Corlett , P. Emma, Z. Huang, K.J. Kim, R. Linberg, J. B. Murphy, G.G. Neil, D.C. Nguyen, C. Pellegrini, R.A. Rimmer, F. Sannibale, G. Stupakov, R.P. Walker and A.A. Zholents, Free electron lasers: Present status and future challenges, Nuc. Inst. And Meth. A 618 (2010) 69-96.

[7] A. Beswick, H. Baumgartel, C. R. A. Catlow, P. Decleva, H. Dexpert, E. Geissler, J. Goulon, M. J. Hubinfranskin, J. Lajzerowicz, Time-resolved X-ray Studies using 3rd generation synchrotron radiation sources, in Synchrotron Radiation and Dynamic Phenomena AIP Conf. Proc. **258** (1992) 483-495.

[8] Website of the Center for Defect Physics, a United States Department of Energy Energy Frontier Research Center: http://www.ms.ornl.gov/cdp/index.shtml

[9] P. L. Solari,, S. Schluti, H. Hermange, B. Sitaud, Mars, a new beamline for radioactive matter studies at SOLEIL, 14th International Conf. on X-ray Absorption Fine Structure, J. of Phys Conf. Series 190 (2009) 012042.

[10] First results from the mars beamline: http://www.synchrotron-soleil.fr/Recherche/LignesLumiere/MARS

[11] T. Holden, M. Bourke and S. Maloy, Research Needs and Opportunities for Characterization of Activated Samples at X-ray and Neutron User Facilities", Report of Sept. 20-22 2009 Workshop, 2010.

ACCEPTED MANUSCRIPT

ACCEPTED MANUSCRIPT

[12] Workshop on the Role of Synchrotron Radiation in Solving Scientific Challenges in Advanced Nuclear Energy Systems 27 to 28 January 2010 at Argonne's Advanced Photon Source: http://www.aps.anl.gov/News/APS_Today/Announcements/APS_workshop_LS.pdf.

[13] H. Toda, K. Shimizu, K. Uesugi, Y. Suzuki, M. Kobayashi, Application of Dual-Energy K-Edge Subtraction Imaging to Assessment of Heat Treatments in Al-Cu Alloys", Mat. Trans. **51** (2010) 2045-2048.

[14] F. Meirer, J. Cabana, Y. Liu, A. Mehta, J.C. Andrews and P. Pianetta, Three-dimensional imaging of chemical phase transformations at the nanoscale with full-field transmission X-ray microscopy", J. Synch. Rad. 18 (2011) 773-781.

[15] M. Herbig, A. King, P. Reischig, H. Proudhon, E. M. Lauridsen, J. Marrow, J. Y. Buffiere, W. Ludwig, 3D growth of a short fatigue crack within a polycrystalline microstructure studied using combined diffraction and phase-contrast X-ray tomography", Acta Mater. 59 (2011) 590-601.

[16] C. J. Sparks Jr., X-ray fluorescence microprobe for chemical analysis, in: H. Winick and S. Doniach (Eds.), Synchrotron Radiation Research, Plenum, New York, 1980 pp. 459–512.

[17] M. Naghedolfeizi, J. S. Chung, R. Morris, G.E. Ice, W. B. Yun, Z. Cai, B. Lai. X-ray fluorescence microtomography study of trace elements in a SiC nuclear fuel shell, J. Nucl. Mat. 312 (2003)146-155.

[18] H. F. Poulsen, W. Ludwig, S. Schmidt S., 3D X-ray Diffraction Microscopy, in: W. Reimers, A.R. Pyzalla, A. Schreyer and H. Clemens (Eds.), Neutrons and Synchrotron Radiation in Engineering Materials Science, Wiley-VCH 2006, pp. 335-351.

[19] J. W. L. Pang, G.E. Ice, W.-J. Liu The role of crystal orientation and surface proximity in the self-similar behavior of deformed Cu single crystals, Mat. Sci. and Eng. A 528 (2010) 28-31.

[20] H. Bei, S. Shim, E. P. George, M. K. Miller, E. G. Herbert, G. M. Pharr, Compressive strengths of molybdenum alloy micro-pillars prepared using a new technique, Scripta Mater. 57 (2007) 397-400.

[21] B.C. Larson, W. Yang, G. E. Ice, J. D. Budai, J. Z. Tischler, Three-Dimensional X-Ray Structural Microscopy with Submicrometre Resolution, Nature 415 (2002) 887-891.

[22] E. D. Specht, F. J. Walker, W.-J. Liu, X-ray microdiffraction analysis of radiation-induced defects in single grains of polycrystalline Fe, J. Sync. Rad., 17 (2010) 250-256.

[23] G. E. Ice, and J. W. L. Pang, Tutorial on x-ray microLaue diffraction, Mat. Charact. 60 (2009) 1191-1201.

ACCEPTED MANUSCRIPT

ACCEPTED MANUSCRIPT

[24] P. Mayr, T. A. Palmer, J. W. Elmer, E. D. Specht, S. M. Allen Formation of Delta Ferrite in 9 Wt Pct Cr Steel Investigated by In-Situ X-Ray Diffraction Using Synchrotron Radiation, Met. and Mat. Trans. A 41A (2010) 2462-2465.

[25] E. D. Specht, P. F. Tortorelli, P. Zschack, In-situ measurement of growth stress in alumina scale, Powder Diffraction, 19 (2003) 69-73.

6.0 Figure Captions

Fig. 1. Schematic of the advantages offered by ultra-brilliant synchrotron hard X-ray beams. (A) High signal to overwhelm sample-introduced backgrounds and to efficient use crystal optics to shield detectors and gain the best signal-to-noise; (B) timing to improve background and time resolution; and (C) focused beams to resolve heterogeneities and for characterization of small samples.

Fig. 2. With samples on the order of 10^{-3} mm^3 the activity of irradiated materials is reduced by 6 orders of magnitude compared to cm^3 samples. For example, diffuse X-ray scattering samples are typically 300 mm^3 or larger. With microbeams however, a 10^{-3} mm^3 sample can have hundreds to thousands of useful sample volumes.

Fig. 3. Absorption and edge contrast tomography used to measure the structure of a TRISO fuel particle. Note that these measurements were made on a laboratory source with ~1.2 mm resolution. They can now be made in the same time with 30 nm resolution at a synchrotron source.

Fig. 4 Elemental map of Zn in the shell of a TRISO SiC shell. The figure is adapted from Naghedolofeizi et al. [17].

Fig. 5. Measurements of diffuse scattering in Fe grain with a submicron beam. The irradiated sample has significantly increased diffuse scattering (after Specht et al. [23].

Fig. 6. In-situ measurement of oxide scale formation and stress development.

ACCEPTED MANUSCRIPT