ANNUAL RESEARCH REPORT

2017

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The Annual Research Report

This report summarizes the principal research activities in the Michigan Ion Beam Laboratory during the past calendar year. Eighty researchers conducted 32 projects at MIBL that accounted for 5704 hours of instrument usage. The programs included participation from researchers at the University, corporate research laboratories, private companies, government laboratories, and other universities across the United States. The extent of participation of the laboratory in these programs ranged from routine surface analysis to ion assisted film formation. Experiments included Rutherford backscattering spectrometry, elastic recoil spectroscopy, nuclear reaction analysis, direct ion implantation, ion beam mixing, ion beam assisted deposition, and radiation damage by proton bombardment. The following pages contain a synopsis of the research conducted in the Michigan Ion Beam Laboratory during the 2016 calendar year.

About the Laboratory

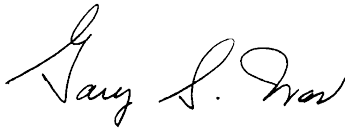
The Michigan Ion Beam Laboratory for Surface Modification and Analysis was completed in October of 1986. The laboratory was established for the purpose of advancing our understanding of ion-solid interactions by providing up-to-date equipment with unique and extensive facilities to support research at the cutting edge of science. Researchers from the University of Michigan as well as industry and other universities are encouraged to participate in this effort.

The lab houses a 3 MV Pelletron accelerator, a 1.7 MV tandem ion accelerator, and a 400 kV ion implanter that are configured to provide for a range of ion irradiation and ion beam analysis capabilities. The control of the parameters and the operation of these systems are mostly done by computers and are interconnected through a local area network, allowing for complete control of irradiations from the control room as well as off-site monitoring and control.

In 2010, MIBL became a Partner Facility of the National Scientific User Facility (NSUF), based at Idaho National Laboratory, providing additional opportunities for researchers across the US to access the capabilities of the laboratory.

On December 20, 2013, the lab was closed for a major expansion that included the addition of a 3 MV Pelletron accelerator, reconfiguration of the accelerator room, establishment of a target room and a control room. The refurbished lab consists of 3 accelerators, 8 beam lines, 5 target chambers and the capability to conduct dual and triple beam irradiations. The expansion was completed in the Fall of 2015 and the lab was rededicated on October 26, 2015.

Respectfully submitted,



Gary S. Was, Director

Research Projects

GRAIN ORIENTATION EFFECT ON IRRADIATION ACCELERATED CORROSION OF 316L STAINLESS STEEL

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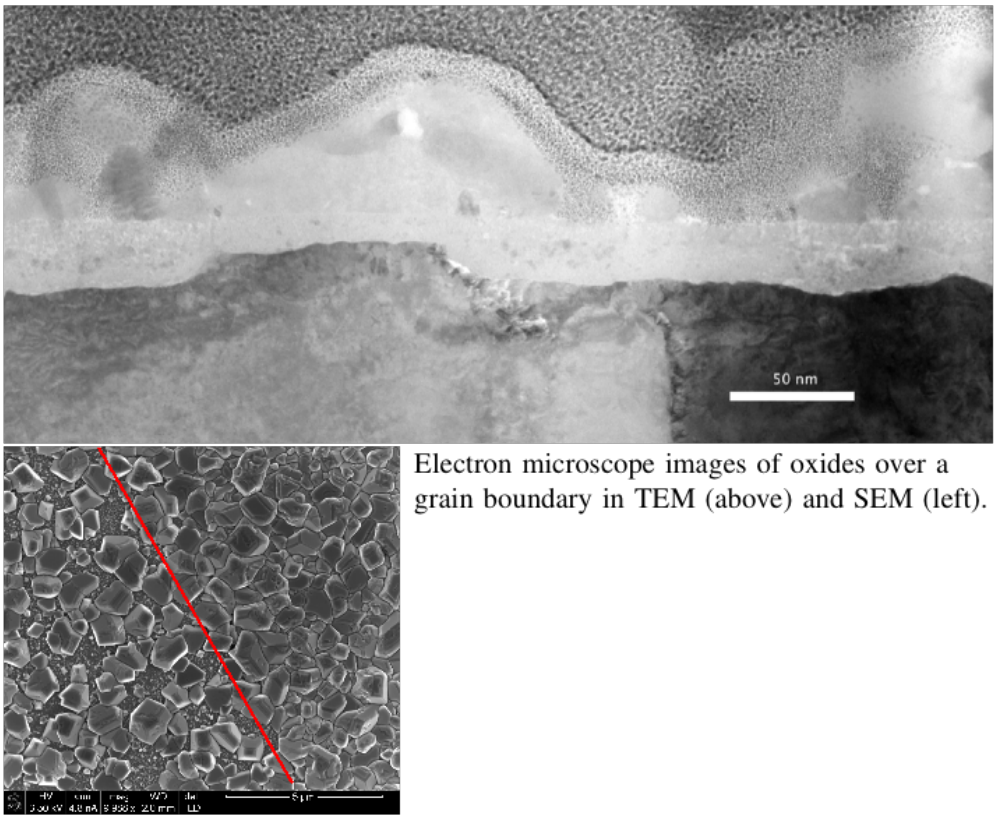
Irradiation assisted stress corrosion cracking (IASCC) is a major concern in light water reactors. IASCC is a phenomenon that depends on complex interactions between radiation, material, and the environment that are not well understood. By simulating the in-reactor conditions with proton irradiation, the process of irradiation accelerated corrosion (IAC) can be studied to better understand the core processes behind IASCC. By using the sample as a barrier between the beamline and the water, radiation damage occurs in both the water and the metal in contact with the water.

Previous work has shown that the grain orientation of the oxidized surface can have an impact on corrosion behavior. A redesign of sample mounts for the IAC cell in MIBL utilizes 5.4 MeV protons to perform corrosion experiments with minimal deformation and residual activity. Two activation experiments were performed to validate this sample redesign.

IAC experiments were conducted on 36 μm 316L stainless steel samples backed by 40 μm 17-4 PH stainless steel in 320 °C high purity water with 3 wppm hydrogen for 24 hours. Sample surfaces were pre-characterized by EBSD to obtain a map of grain orientations that can be correlated with oxide properties afterwards. Characterization of the oxide was accomplished with Raman spectroscopy and mapping, SEM and TEM.

This research is supported by EDF contract number 8610-5920005571.



Electron microscope images of oxides over a grain boundary in TEM (above) and SEM (left).

ION BEAM ANALYSIS OF INTERSTITIAL COMPLEXES IN GaAs(Bi)N ALLOYS

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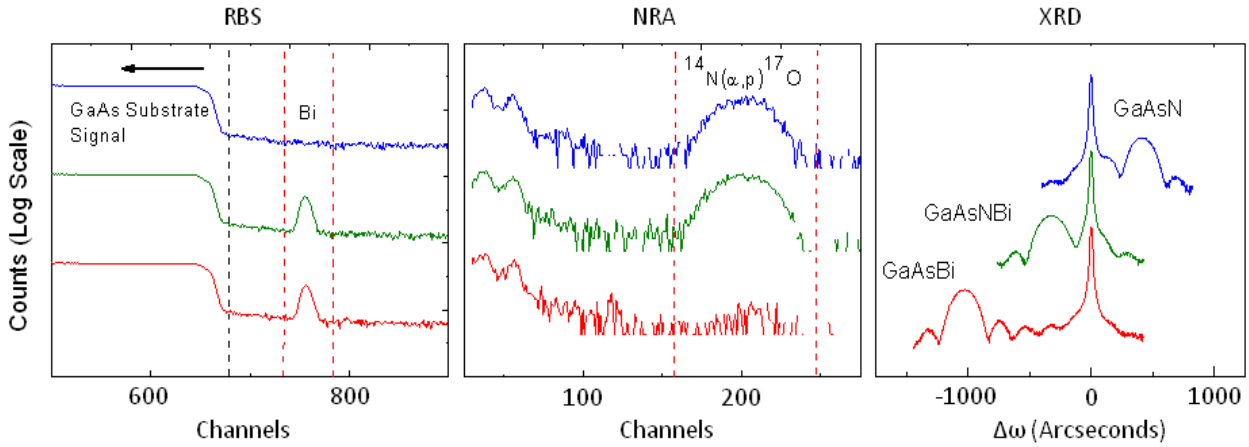
Due to their significant band-gap narrowing with minimal change in lattice parameter, dilute nitride semiconductor alloys are useful for a variety of applications, including long-wavelength lasers and detectors, ultra-high-efficiency solar cells, and high performance heterojunction bipolar transistors. However, N-related point defects often contribute to carrier scattering and recombination, leading to degraded carrier mobilities and optical efficiencies. For GaAsN and related alloys, co-alloying with larger group V elements such as Sb or Bi is expected to lead to significant energy band gap narrowing using a substantially lower N fraction, and a correspondingly lower concentration of N-related defects. For GaAsN, several groups have suggested that N shares an arsenic site with either arsenic or another N atom, often termed $(N\text{-}As)_{As}$ or $(N\text{-}N)_{As}$ split interstitials. In the case of GaAsNBi, the published experimental work has focused primarily on growth parameters and optical properties, without addressing the mechanisms for N and Bi co-incorporation during epitaxy. To identify N-related defects, we compare channeling Rutherford backscattering and nuclear reaction analysis spectra with Monte Carlo-Molecular Dynamics simulations along the [100], [110], and [111] directions. Using this combined computational-experimental approach, we have identified $(N\text{-}As)_{As}$ is the dominant interstitial GaAsN [1]. For GaAsNBi, a comparison of RBS, NRA and x-ray rocking curve analyses reveals enhanced N incorporation in the presence of Bi. Furthermore, ion channeling measurements reveal non-substitutional incorporation of the extra N. We are currently considering the nature of the N interstitial complexes in GaAsNBi, including the relative concentrations of $(N\text{-}N)_{As}$, $(N\text{-}As)_{As}$, and $N_{tetrahedral}$.

This work is supported support by the National Science Foundation (Grant No. DMR 1410282) and the U.S. Department of Energy Office of Science Graduate Student Research (SCGSR) Program.

[1] T. Jen, G. Vardar, Y. Q. Wang, and R. S. Goldman. Appl. Phys. Lett. **107**, 221904 (2015)



Sample spectra of RBS (left), NRA (center), and XRD (right). Each plot has three samples – GaAsN (top), GaAsNBi (middle), and GaAsBi (bottom). Region of interest in RBS and NRA are marked in between two dashed line. As expected, GaAsN sample does not have Bi signal in the RBS plot while GaAsBi sample does not have N signal in the NRA plot. In XRD plot, N (Bi) atoms will introduce compressive (tensile) strain, therefore, the epilayer will shift to right (left) while GaAsNBi epilayer peak location will be determined by sum of compressive and tensile strain.

FORMATION OF α′ IN ION-IRRADIATED F-M ALLOYS

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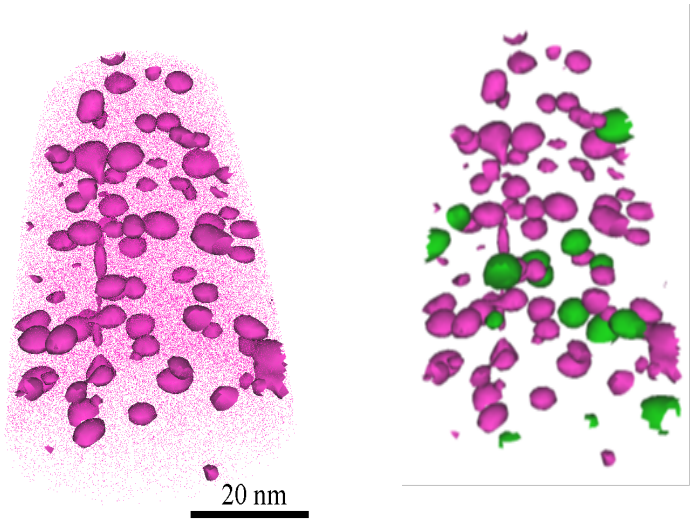
The formation of α′ phase in F-M alloys during irradiation may cause significant hardening and embrittlement leading to property degradation. The formation of α′ phase has been reported in reactor-irradiated and electron-irradiated Fe-Cr alloys. However, the α′ phase has yet to be positively identified in ion irradiated Fe-Cr alloys, even though some Cr-rich precipitates containing C,N, Si or P were reported. The objective is to investigate the formation of α′ phase in high dose rate ion irradiated F-M alloys.

F-M alloys HCM12A and HT9 were used in this study. Ion irradiations were conducted at MIBL using 2 MeV protons at 400°C to 3 and 7 dpa with a dose rate of 3.5×10^{-5} dpa/s (Full cascade), and 5 MeV Fe^{++} at 400°C to 7 and 100 dpa with a dose rate of ~1×10^{-3} dpa/s, and to 3 dpa with a dose rate of 3.4×10^{-5} dpa/s (Full cascade). α′ or precursors formed in proton-irradiated HCM12A and HT9 at 400°C with a dose rate of 3.5×10^{-5} dpa/s, but were not observed in Fe^{++} irradiation up to 100 dpa with a dose rate of 10^{-3} dpa/s. α′ or precursors did not form in Fe^{++} irradiated samples at the same dpa and damage rate as proton irradiation, indicating that damage rate is not the sole factor controlling α′ formation in self- ion irradiated F-M alloys.

Support was provided by DOE under contract DE-FG07-07ID14894.



Formation of α′ or precursors (magenta) in HT9 proton-irradiated to 7 dpa at 400°C and their spatial relation with the Ni/Si precipitates (green).

LOCALIZED DEFORMATION AND INTERGRANULAR FRACTURE OF IRRADIATED ALLOYS UNDER EXTREME ENVIRONMENTAL CONDITIONS

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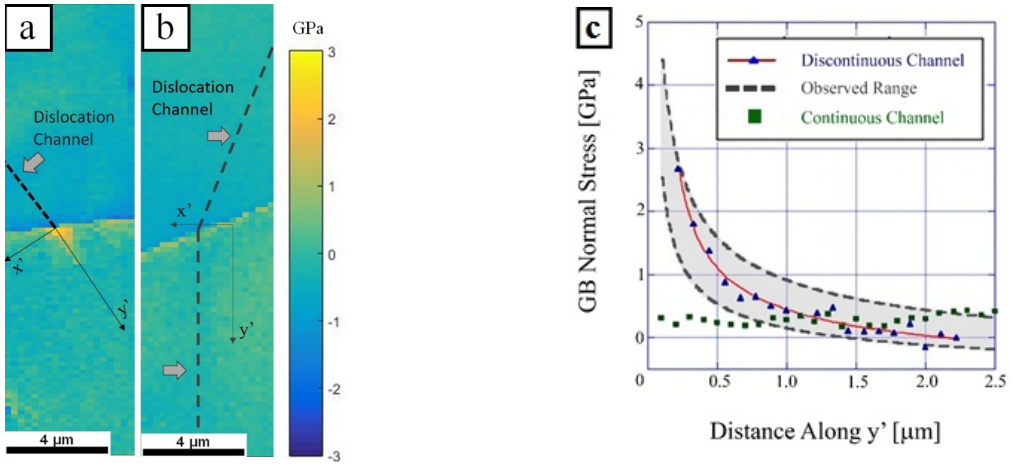
The goal of this project is to determine the role of localized deformation in austenitic steel during irradiation assisted stress corrosion cracking (IASCC). The project is a collaboration between the University of Michigan, University of Wisconsin, and Virginia Tech with the purpose of obtaining better understanding of the mechanisms involved in IASCC. Samples are being irradiated at the University of Michigan and strained in constant extension rate tensile (CERT) tests to study the cracking behavior. Atomistic models of the experiments are being developed by Dr. Farkas's group at Virginia Tech, and irradiated samples will be strained in-situ in a TEM by Dr. Robertson's group at the University of Wisconsin.

This year, two irradiations were performed using 2 MeV protons at 360 °C in the Wolverine Tandem Accelerator located in the Michigan Ion Beam Laboratory. Tensile samples from this irradiation will be used to quantify the stress component normal to grain boundaries at discontinuous dislocation channel – grain boundary interaction sites, which will be related to cracking. Samples will be strained in high temperature argon (288°C) to produce dislocation channels. These channels are either arrested at the grain boundary or transmit into the adjacent grain. In both cases, residual elastic stress values will be calculated using High Resolution Electron Backscatter Diffraction, as shown in the figure. Once stress has been characterized, the samples will be strained further in water, and cracking behavior will be characterized, with respect to the known stress levels in the dislocation channel/grain boundary intersections. A Plasma Focused Ion Beam (PFIB) will then be used to determine the orientation of grain boundaries in 3D, and allow the measured stresses to be resolved in a direction normal to the grain boundary.

This research has been supported by the Basic Energy Science office of the U.S. Department of Energy under grant DE-FG02-08ER46525.



Stress profile near a discontinuous channel (a) and near a continuous channel (b). GB normal stress has been plotted as a function of distance from the GB in both cases (c).

EFFECT OF PROTON AND HEAVY ION IRRADIATION ON WSe_2 AND SiC HETEROSTRUCTURES

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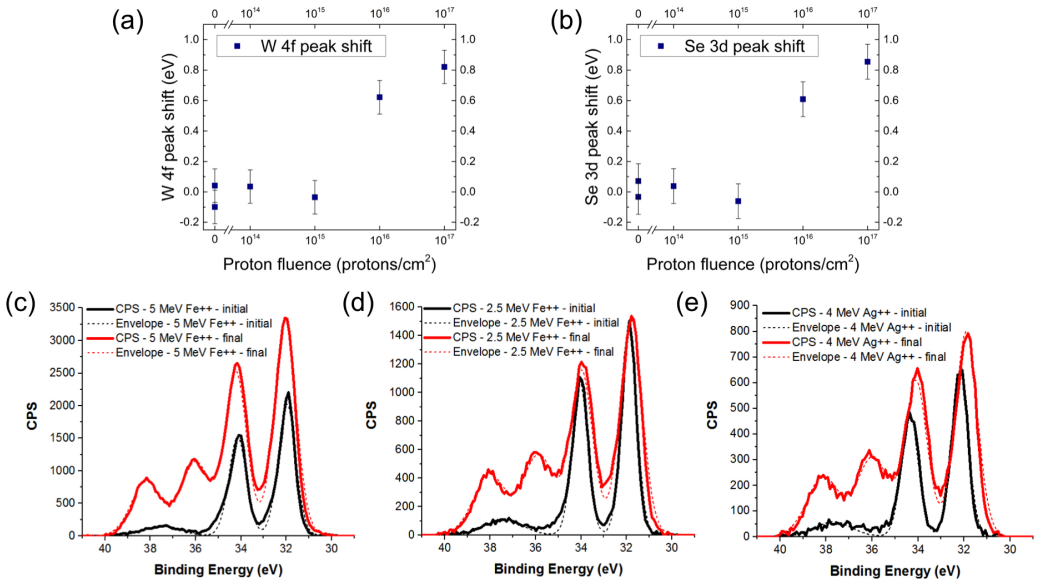
Transition metal dichalcogenides (TMDs) have received much attention in recent years because of their outstanding material, optical and electronic properties (sizable band gap, high mobility, etc.) and atomically-thick dimensions. Among various TMDs, tungsten diselenide (WSe_2) exhibits particularly attractive electronic properties. For the purpose of investigating the potential for use of TMD-based devices in radiation-harsh environment and space applications, we have studied the chemical modifications of WSe_2/SiC heterostructure induced by proton and heavy ion irradiation using UV-visible-NIR spectroscopy and X-ray photoelectron spectroscopy. Irradiation via a beam of 2 MeV protons was carried out at different fluence levels (10^{14}-10^{17} protons/cm^2). The deterioration of WSe_2/SiC heterostructure was investigated with heavy ions (2.5 MeV Fe ions, 5 MeV Fe ions and 4 MeV Ag ions) at a fluence of 10^{16} ions/cm^2.

It was found that a dose of 10^{16} protons/cm^2 was necessary to induce a charging effect in the heterostructure, which led to significant shifts of the core level binding energies of WSe_2. Meanwhile, the SiC undergoes a color change from transparent to black with a corresponding feature appearing in the absorption spectra around 1.1 eV. This was attributed to displacement damage that led to the formation of a region rich with single vacancies. Heavy ions damage WSe_2 and convert it into a mixture of WO_x and Se-poor WSe_2. The substrate 6H-SiC is also damaged, becoming a mixture of amorphous sp^3 carbon, SiO_x and C-poor SiC. The valence band of this heterostructure is heavily altered due to the oxidation of WSe_2 and amorphization of SiC, which is expected to have deleterious impact on device operation.

This work is supported by the Defense Threat Reduction Agency (DTRA) under grant number HDTRA1-14-1-0037.



The binding energy shift of (a) W 4f peak and (b) Se 3d peak as a function of 2 MeV proton fluence. The change of W4f peak spectrum after irradiation with c) 5 MeV Fe, (d) 2.5 MeV Fe, and (c) 4 MeV Ag ions at a fluence of 10^{16} ions/cm^2. The appearance of two additional peaks after the irradiation indicates the oxidation of tungsten.

INVESTIGATION OF ION IRRADIATION BEHAVIORS OF SILICON CARBIDE (SiC) AND NANOSTRUCTURED FERRITIC ALLOY (NFA) COMPOSITES

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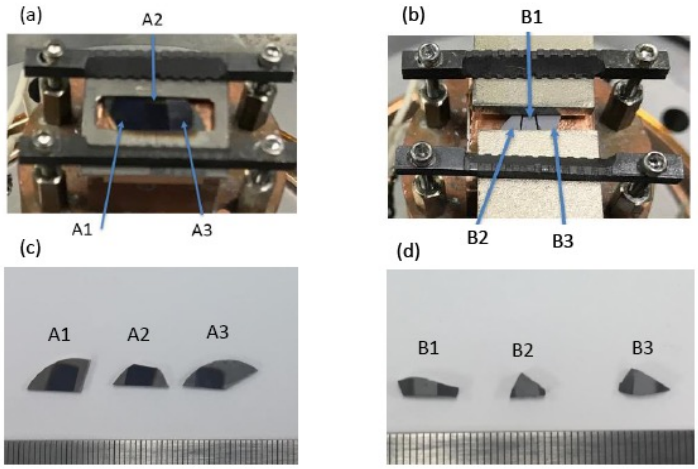
Cladding materials in nuclear fission reactors are expected to withstand high dose of irradiation at high temperatures of 500-1000^{o}C for long periods of time. Composites of silicon carbide (SiC) and nanostructured ferritic alloy (NFA) are proposed to be considered as a new candidate for such applications. SiC has high thermal stability, mechanical strength, and superior irradiation resistance, and has also been considered as breeding blanket in fusion reactors to reduce diffusion of radiation products, due to its excellent chemical stability at elevated temperatures. Recently developed 9-Cr nanostructured ferritic alloy (NFA) is another promising material for radiation shielding applications due to its very high strength, creep and irradiation resistance. The NFA derives its superior property from the Y-Ti-O-Cr-Fe enriched nanoclusters, which not only improve mechanical strength and capability of trapping helium atoms but also reduce the irradiation induced point defects.

The new SiC-NFA composites, by combining desirable properties from both SiC and NFA, are expected to be a promising nuclear cladding material with high corrosion resistance, strength, creep and irradiation resistance. In this work, spark plasma sintered (SPS) SiC-NFA composites with different compositions were irradiated by Fe^{++} and Si^{++} ions at room temperature to explore their ion irradiation resistance.

The high NFA samples (A1: 0 vol% SiC-100 vol% NFA, A2: 2.5 vol% SiC-97.5 vol% NFA, A3: 5 vol% SiC-95 vol% NFA) were irradiated to 103 dpa using Fe^{++} ions with ion beam energy of 5 MeV at room temperature. While high SiC samples (B1: 100 vol% SiC-0 vol% NFA, B2: 97.5 vol% SiC-2.5 vol% NFA, B3: 95 vol% SiC-5 vol% NFA) were irradiated to 97dpa using Si^{++} ions with ion beam energy of 5 MeV at room temperature. These samples are being characterized using SEM, EBSD and cross-sectional TEM to evaluate the irradiation behaviors.

This work is supported by the Office of Nuclear Energy of Department of Energy under grant #DE-NE0008264.



The setup of ion irradiation for samples of A1: 0 vol% SiC-100 vol% NFA, A2: 2.5 vol% SiC-97.5 vol% NFA, A3: 5 vol% SiC-95 vol% NFA (a) and B1: 100 vol% SiC-0 vol% NFA, B2: 97.5 vol% SiC-2.5 vol% NFA, B3: 95 vol% SiC-5 vol% NFA (b); after ion irradiation the high NFA samples showing the induced dark color (c) and the high SiC showing the induced white color (d).

DEVELOPMENT OF MICRO-CHARACTERIZATION TECHNIQUES FOR PROTON IRRADIATED NUCLEAR MATERIALS

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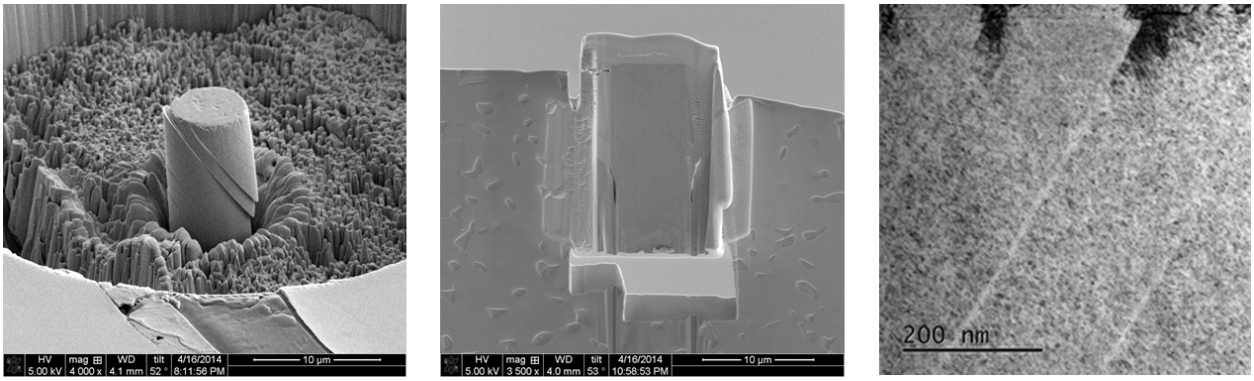
Degradation of materials properties under neutron irradiation is one of the key issues limiting the lifetime of nuclear reactors, their continued operation and the development of next-generation nuclear systems. Evaluating the property changes of materials due to irradiation and understanding the role of microstructural evolution in mechanical property changes are required for ensuring the safe and reliable operation of nuclear reactors. Ion beam irradiation can be used to simulate neutron irradiation of materials in a controlled way but has the main limitation of small penetration depth in the scale of micro meters. On the other hand, handling of neutron-irradiated materials is restricted due to activation. To overcome these disadvantages, proton irradiation technique and small-scale material testing are applied in characterizing irradiated materials using micro-sized samples.

Proton irradiation of commercial austenitic SS was performed using 2 MeV protons at 360 °C in the Michigan Ion Beam Laboratory. Two kinds of samples were obtained with different damage by controlling the irradiation time, which are low-dose (1 dpa) and high-dose (5 dpa) sample. Micro-compression tests on the irradiated samples were carried out with a nanoindenter (NHT2, CSM instruments). The engineering stress-strain curves of low-dose and high-dose proton irradiated samples were obtained using the micro compression test. The average critical resolved shear stress for low- and high-dose samples were measured to be 227 and 458 MPa, respectively. We characterized microstructure changes in the proton irradiated samples using transmission electron microscopy.

This work is supported by a National Research Foundation of Korea (NRF) grant funded by the Korea government (MSIP).



SEM image (left) of micro-pillar in the proton irradiated SS after micro-compression test. TEM sample (center) fabricated from the micro-pillar, and deformed microstructure (right) obtained from the TEM sample.

IRRADIATION ASSISTED STRESS CORROSION CRACKING (IASCC) BEHAVIOR OF ALLOY APMT (PLATE) AND ALUCHROM YHF

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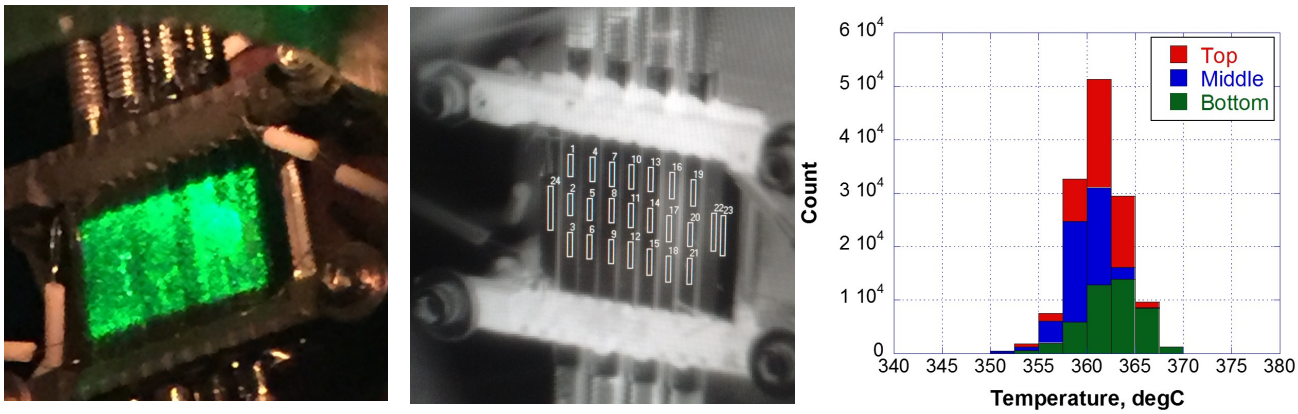
The research work carried out in this project is a part of the work aimed at development of advanced alloys for accident tolerant fuel (ATF) clad application. In this context, three different alloys, namely, alloy 33, alloy T91 and APMT forged ingots were studied first, followed by super ferritic alloys 446 and 4C54. The IASCC behavior and oxidation performance were demonstrated to be best in the case of APMT alloy forged ingot.

In continuation of this project, an APMT alloy plate material and Aluchrom YHF (a FeCrAl alloy processed through traditional melting route) are being investigated for ATF clad application. A systematic investigation has been carried out to assess their IASCC in boiling water reactor (BWR) normal water chemistry (NWC) environment at the University of Michigan.

Both alloys APMT and Aluchrom YHF were subjected to irradiation using 2 MeV protons at a temperature of 360 °C. A total area of 18 mm x 10 mm was irradiated, which included two tensile specimens of each alloy and three TEM bars each of APMT and Aluchrom. The arrangement of specimens on the irradiation stage with laser beam alignment before irradiation is presented in part (a) of the figure. A total dose of 5 dpa was achieved through the irradiation. A stable beam current of ~ 35 μA was achieved on the stage. The temperature and pressure through the irradiation experiment remained stable. The thermal image indicating 24 areas of interest (AOIs) is shown in the figure, part (b); while the typical temperature distribution on top, middle and bottom AOIs (1-3) for the first tensile bar is given in part (c). The irradiated tensile specimens of both alloys were subsequently subjected to stress corrosion cracking (SCC) tests in BWR-NWC simulated environment.

Research supported by NEUP from the Office of Nuclear Energy, U. S. Department of Energy.



(a) (b) (c)

Irradiation stage having APMT and Aluchrom with laser alignment (a), thermal image with AOIs (b), and temperature distribution on top, middle and bottom AOIs for first tensile specimen (c).

SURFACE VERSUS BULK CHEMISTRY OF WATER FILTRATION MEMBRANES

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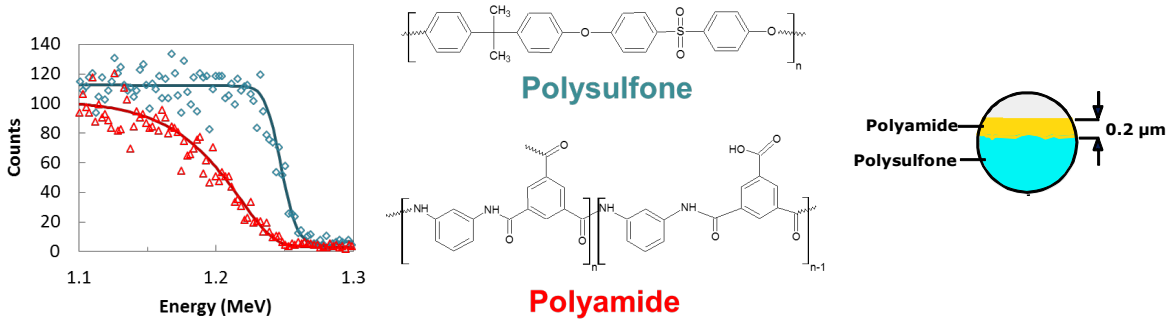
The Dow Chemical Company

Safe and pure water is essential to life and critical to human progress. At Dow Chemical we have the most complete portfolio in the industry today and a global presence second to none with #1 positions in reverse osmosis and ion exchange. Access to the Michigan Ion Beam Laboratory has allowed Dow to further the development of advanced membrane chemistries. The polyamide layer of reverse osmosis thin-film composite membranes is <200 nm thick. Separation of this thin layer from the supporting layers is a complex process and can only be done chemically, which results in a fragile polyamide layer and makes characterization challenging. X-ray photoelectron spectroscopy (XPS, near-surface) and Rutherford backscattering spectrometry (RBS, bulk) have been applied to characterize the polyamide layer, without the need to separate polyamide from the supporting layers. Using the automatic stage controls on the Maize IB9 chamber, the beam can be rastered to keep a low He^+ ion fluence to prevent damage to the polymer.

RBS allows determination of the thin-film thickness of the active polymer layer in the membrane, surface roughness, and elemental composition, as shown in the figure below. The combination of RBS and XPS analysis also allows the comparison of bulk vs. near-surface carboxylic acid content, which is a driver in filtration performance. We have leveraged these analyses for current and new product development for increased filtration performance.

- Unique element, S, in polysulfone makes thickness determination possible
- Shape of S edge is due to roughness



Left: RBS spectra of the sulfur edge between 1.1 – 1.3 MeV showing edge shift due to polyamide thickness. Center: chemistry of polyamide active layer. Right: Thin film composite of reverse osmosis water filtration membrane.

IRRADIATION ASSISTED STRESS CORROSION CRACKING BEHAVIOR STUDY OF ALLOY 718

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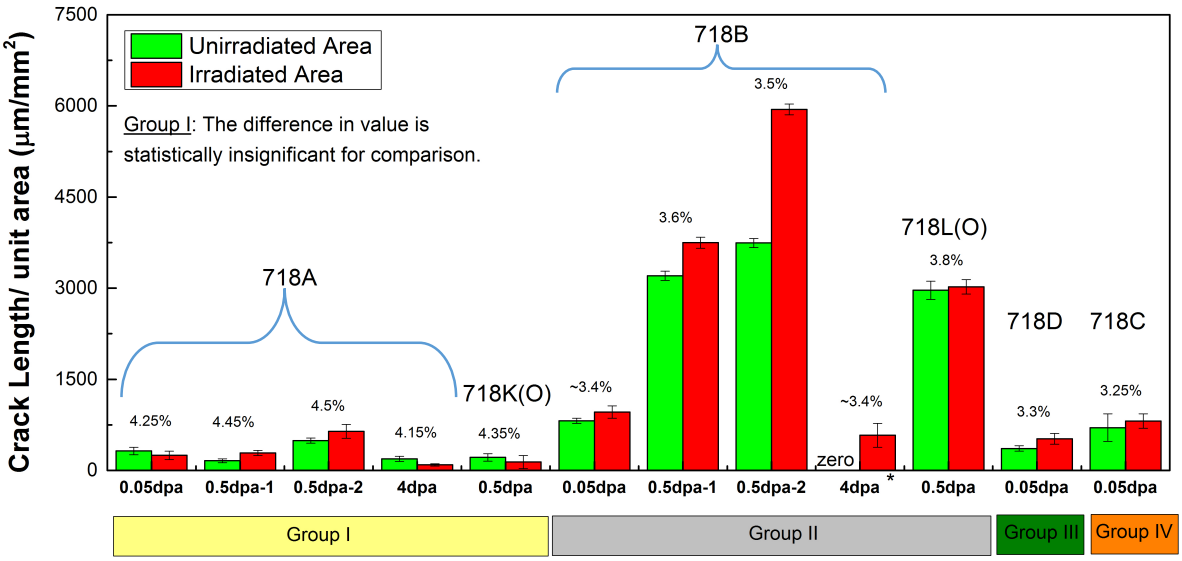
Inconel 718 is an age hardenable nickel-based alloy used in fuel assembly of PWRs because of its high strength and resistance to irradiation-induced growth and corrosion. There have been instances of cracking and deleterious effects of components manufactured with this particular alloy, leading to early assembly discharge, and possibly, fuel failures. This project focuses on studying the irradiation assisted stress corrosion cracking (IASCC) behavior of Alloy 718 in PWR primary environment. The stress corrosion cracking (SCC) behavior of alloy 718 subject to different thermal mechanical histories was studied in both the irradiated and non-irradiated conditions. Four tensile and three TEM samples were irradiated with 2 MeV protons to doses of 0.05 dpa, 0.5 dpa, and 4 dpa at a temperature of 360°C as listed in the Table. The tensile samples were used to evaluate IASCC behavior and the TEM samples were used for microstructural characterization. The figure illustrates the IASCC susceptibility of alloy 718 with different conditions. Group II conditions (B and L) were most susceptible, group I conditions (A and K) were least susceptible, and groups III and IV (conditions C and D) were of intermediate susceptibility.

This research is supported by Electric Power Research Institute (EPRI, contract *MA10001593*).

Proton irradiations completed at MIBL.

Dose (dpa)	Date	Tensile samples	TEM samples
0.05	Nov. 2014	A, B, C, D	A, B
0.5	Oct. 2014	K(O), L(O)	A, B, C, D, K(O)
4	Sept 2015	A, B	A, B
0.5	Sept 2016	A, B	none



Cracking susceptibility of irradiated 718 with different conditions straining to 3-5% in PWR primary water.

IRRADIATION INDUCED LONG RANGE ORDER PHASE IN NICKEL BASE ALLOYS

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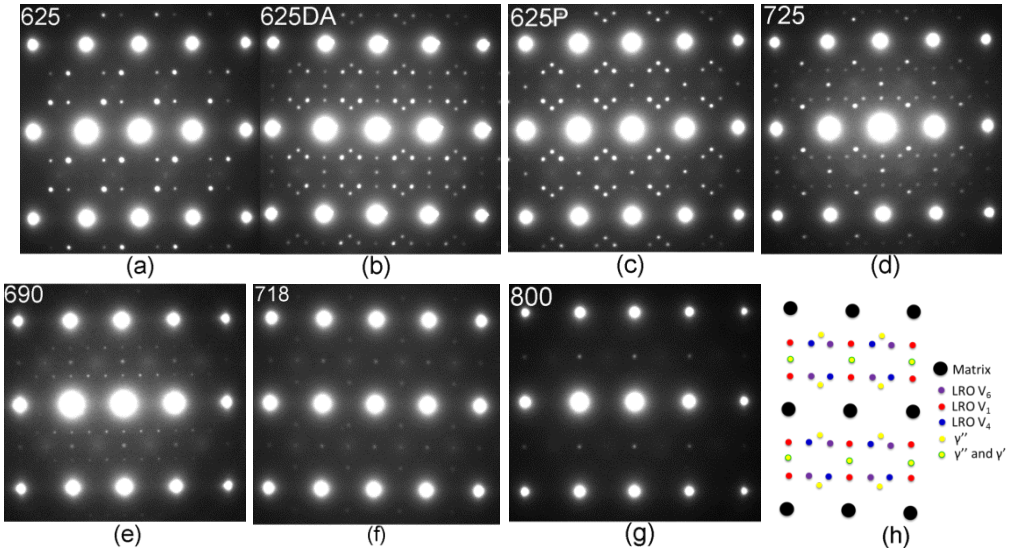
Department of Nuclear Engineering and Radiological Sciences, University of Michigan

The Advanced Radiation Resistant Materials (ARRM) project is aimed at identifying promising candidate alloys that can replace austenitic stainless steels, which suffer from serious irradiation-assisted stress corrosion cracking in light water reactors environments. Austenitic steel and nickel based alloys were commonly recognized as high performance structure materials in harsh environments. However, the irradiation induced microstructures of these alloys are poorly known.

Several alloys were irradiated with 2 MeV protons to 5dpa at 360°C. The long range ordered (LRO) phase (Ni_2Cr), which requires at least 10^4 hr to form under thermal conditions, was identified in alloys 625, 625DA, 625Plus, 725 and 690, but absent in alloys 718 and 800 as shown in the figure. According to diffraction theory, the intensity of the diffraction spots relative to transmission spots can be an indicator of the relative amount of each phase. Thus, a strong composition dependence of this phase was observed. As the Fe content increased, the diffraction spots of the LRO phase became weaker (from (a) to (e)). In alloy 718, there was no diffraction spot that could be related to the LRO phase. As the composition becomes more iron rich, the γ' phase becomes prevalent as shown for alloy 800 in (g). The key for the diffraction pattern is given in part (h). It is proposed that Fe may reduce the binding energy of the Ni-Cr bond and increase the Gibbs free energy of Ni_2Cr when Fe is present. So as Fe is increased, the Ni_2Cr phase become unstable and finally ceases to form.

The ARRM project is supported by EPRI (contracts 10002164 and 10002154) and DOE (contract 4000136101).



Diffraction patterns of the long range ordered phase after 5 dpa: (a) Alloy 625, (b) direct aged 625, (c) alloy 625Plus, (d) alloy 725, (e) alloy 690, (f) alloy 718 (after 4dpa), (g) alloy 800, and (h) key to the diffraciton patterns.

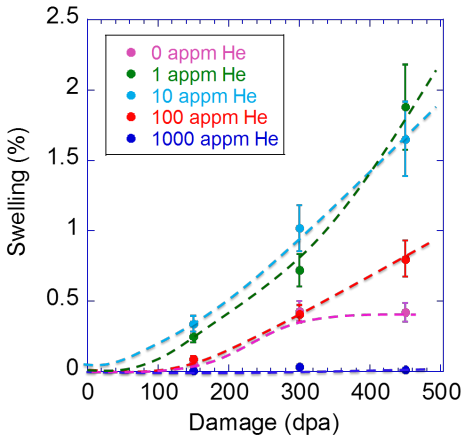
ACCELERATED IRRADIATIONS FOR EMULATION OF HIGH DAMAGE MICROSTRUCTURE IN FERRITIC-MARTENSITIC STEELS

A. M. Monterrosa, Z. Jiao, G. S. Was
Department of Nuclear Engineering and Radiological Sciences, University of Michigan

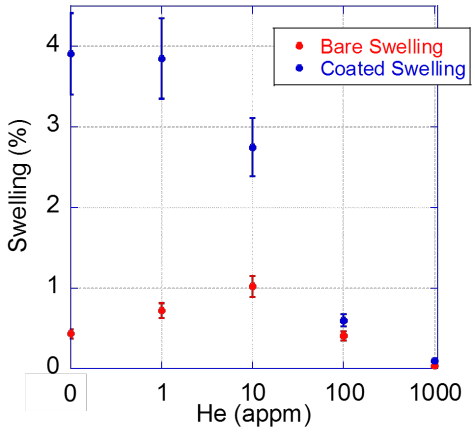
The new generation of faster reactors will push damage levels in structural materials to very high values, in excess of 500 dpa. Using fast reactors to irradiate materials to such high damage is prohibitively expensive and time-consuming. This project will study how effectively self-ion irradiations can be used to emulate microstructural features (voids and precipitates) seen in neutron-irradiated HT9 and other ferritic-martensitic (F-M) steels.

Self-ion irradiation experiments have been performed on ferritic-martensitic alloys T91 to determine swelling behavior at 460°C, at damage levels up to 450 dpa with various levels of helium pre-implanted (0, 1, 10, 100, and 1000 atomic parts per million, appm). The irradiations were performed using the 3MV Wolverine accelerator at the Michigan Ion Beam Laboratory. The effects of damage on void swelling were analyzed using a transmission electron microscope in scanning mode (STEM). Irradiations were performed on samples with and without alumina coatings that were used to prevent carbon contamination. Cavities were observed in all conditions though coated samples exhibited enhanced cavity nucleation and growth with no observable carbide formation. It is likely that the presence of carbon (whether in solution or in the form of carbides) serves to trap vacancies and promote recombination. This results in a microstructure which suppresses nucleation of cavities. In the bare samples, higher helium levels promoted a higher density of cavity nucleation. At the high helium levels of 100 and 1000 appm, swelling is suppressed due to the formation of many small cavities. Intermediate levels of helium (1 and 10 appm) show enhanced swelling due to increased nucleation, without suppressing growth. In the coated samples this trend is not observed. High nucleation occurs in the 0 appm helium case, and further additions of helium only serve to suppress swelling. It appears that in the absence of carbon brought in from the vacuum, the material does not require helium to swell appreciably. These results suggest that in highly nucleation resistant microstructures, additions of helium will result in a peaked swelling behavior. However, in a less nucleation resistant microstructure, helium is not needed to promote swelling and can actually cause it to decrease.

This work is supported by DOE NEUP award DE-AC07-05ID14517.



Swelling as a function of damage in uncoated T91 at a variety of helium pre-implantation levels.



Swelling as a function of helium pre-implantation level for uncoated and coated T91 at 300 dpa.

ION-SLICED LITHIUM NIOBATE THIN FILMS

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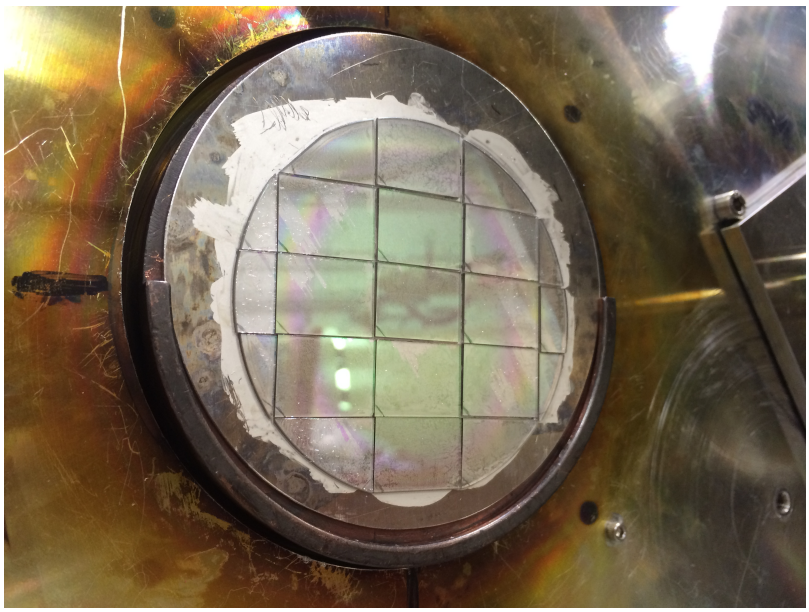
Electroscience Laboratory, Department of Electrical and Computer Engineering,
The Ohio State University

Lithium niobate ($LiNbO_3$) thin films with sub-micron thickness are useful for on-chip optical devices. These films are ion-sliced from a bulk wafer by implanting with He^+ ions and subsequently heat treating. The ions form a damage layer localized at the implantation depth, which is determined by the ion energy. The heat treatment causes stress at this damage layer and causes a thin film to split off.

We started with a 7.62mm x 1mm thick 5% magnesium oxide doped lithium niobate wafer, coated with 500nm of silicon dioxide (SiO_2) on one surface and diced into approximately 1.5cm samples. The samples were mounted to a 4" aluminum carrier with colloidal silver paste. The aluminum carrier was then mounted to a copper fixture provided by MIBL with doubled sided copper tape. Implantation occurred on the surface with the SiO_2. The implant dose was $4x10^{16}$ He^+/cm^2 and the energy was 380kV. SRIM modeling predicts 380kV will produce a 790nm thick $LiNbO_3$ film. The current density was set to $0.25uA/cm^2$ resulting in a total implant time of approximately 8 hours. Thin films were successfully split and the thickness was measured with a Dektak stylus profilometer and found to be near 800nm, in good agreement with simulations.

This material is based upon work supported by the National Science Foundation under Grant No. 1436414



$LiNbO_3$ samples after ion implantation. Samples are mount to an aluminum carrier with silver paste and mounted to copper fixture with double sided copper tape. Fixture is attached to implantation chamber door.

CHARACTERIZATION OF SILICON CARBIDE DIFFUSION COUPLE

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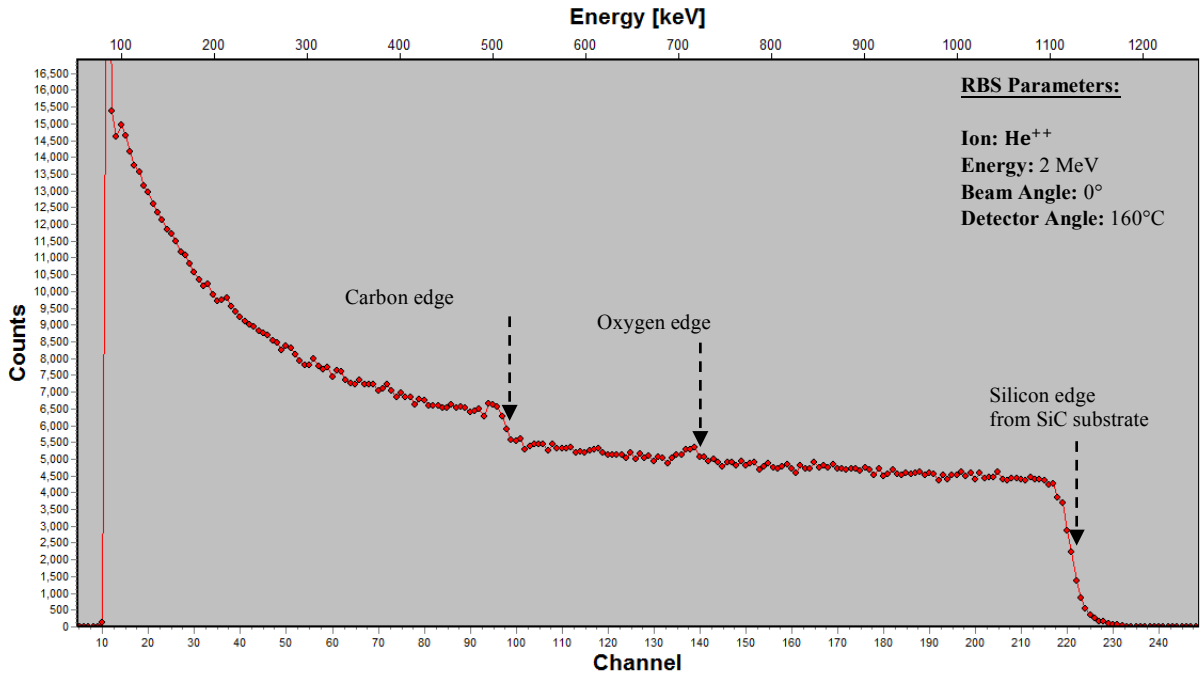
This project focuses on identifying the mechanism for fission product diffusion into silicon carbide (SiC), the main component in TRISO fuel used in the very high temperature reactor (VHTR). Multi-layered diffusion couples consisting of a SiC substrate, a thin pyrocarbon layer, and another thin SiC cap are characterized at various steps in the fabrication process at the Michigan Ion Beam Laboratory (MIBL). Rutherford backscattering spectroscopy (RBS) was performed at MIBL to measure the pyrocarbon layer to verify deposition and measure thickness. A 2 MeV He^{++} beam is used in RBS analysis with a detector angle of 160°. The figure shows a RBS spectrum taken at MIBL for one of four SiC substrates previously coated with pyrocarbon. Analysis of the spectrum revealed that minimal carbon was deposited on the SiC substrate as well as indicating a small silicon oxide layer formed on the substrate.

This research is supported by the Department of Energy under NEUP.



RBS spectrum showing a deposited carbon layer in fabrication of a novel layered diffusion couple for identifying the transport mechanism for fission products in silicon carbide.

IRRADIATION ASSISTED CORROSION AND STRESS CORROSION CRACKING OF NUCLEAR GRADE STAINLESS STEELS

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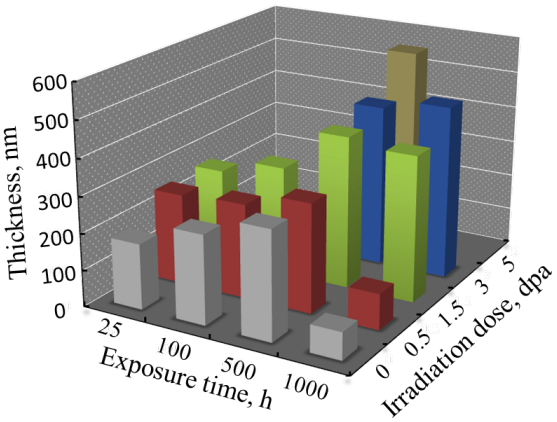
In recent years, environmental degradation of materials in nuclear power plants has been investigated extensively in China. However, due to the capacity limitation of experimental facilities, there is still a lack of understanding of the environmental degradation of core structural materials under the synergic effect among irradiation, high temperature, water corrosion, and stress. By utilizing the advanced ion beam research facilities of the Michigan Ion Beam Laboratory, this program investigates the irradiation assisted corrosion and stress corrosion cracking of domestically-fabricated nuclear grade stainless steels, with a main focus on: 1) Irradiation assisted corrosion of 304 stainless steel in simulated primary water; 2) irradiation assisted stress corrosion cracking of 304 stainless steel in simulated primary water; and 3) effect of annealing on irradiated microstructure and property of 308 stainless steel. Through these research projects, the validity of domestically-fabricated nuclear grade stainless steels for the fabrication of core structures will be determined.

Proton irradiation on samples for corrosion and SCC experiments were conducted to doses of 1, 3 and 5 dpa at 360°C at MIBL. Preliminary results of the experiment revealed the enhancing effect of irradiation on corrosion, shown in the figure. Increasing the irradiation dose increased the thickness of oxide scale formed on 304 SS at each of the exposure times. At a longer exposure time of >500 hr, a reduction of the oxide scale thickness was observed for non-irradiated and 0.5 dpa irradiated samples. This was likely due to the dissolution of the oxide particles in the external layer of the oxide scale. The reduction of the oxide scale thickness, however, was not observed for samples irradiated at higher doses of 1.5 and 3 dpa, suggesting an enhanced growth of the oxide particles in the external layer of the oxide scale by irradiation. The corrosion and SCC experiments are being continued at the Institute of Metal Research in China

This work is supported by the International S&T Cooperation Program of China under grant 2014DFA50800.



Thickness of the oxide scale formed on 304SS as a function of the irradiation dose and exposure time following exposures in simulated PWR primary water.

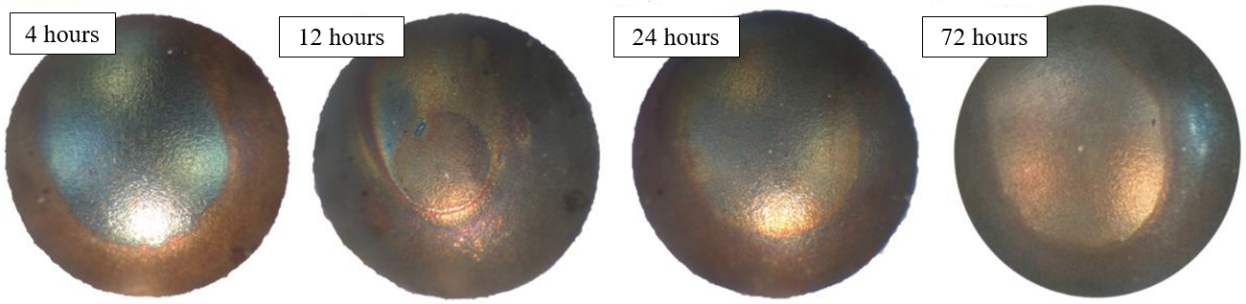
IRRADIATION ACCELERATED CORROSION OF 316L STAINLESS STEEL

S. S. Raiman, G. S. Was, University of Michigan

Corrosion and stress corrosion cracking (SCC) are areas of continued concern for nuclear reactor core structural components. Among the many factors which influence corrosion and SCC, radiation is among the least understood. To better understand how irradiation affects corrosion of stainless steel in pressurized water reactor (PWR) primary water conditions, a dedicated beamline with a controlled chemistry water loop was used to facilitate proton irradiation of samples in high temperature, high pressure water.

A 3.2-MeV proton beam was used to irradiate 316L stainless steel samples while they were simultaneously exposed to simulated PWR primary water. The proton beam was transmitted through the 37 μm thick sample which served as a "window" into a corrosion cell containing flowing 320˚C water with 3 wppm H_2. Irradiation was found to increase the corrosion potential at the surface of the samples through the creation of oxidizing radiolysis products. The images below show samples irradiated for 4, 12, 24, and 72 hrs. The irradiated areas at the center of each sample are discolored, and examination revealed that the areas affected by radiation had thinner and more porous oxides that were deficient in chromium.

This work was supported by the DOE-NEUP, grant number DE-AC07-05ID14517 and EDF Contract No. 8610-BVW-4300243004.



Post-exposure optical images of samples irradiated for 4, 12 24, and 72 hrs at damage rate of 7 x 10^{-6} or 7 x 10^{-7} dpa/s in 320˚C water with 3 wppm hydrogen.

MECHANICAL BEHAVIOR OF 410 MARTENSITIC STEEL AFTER HIGH TEMPERATURE PROTON IRRADIATION

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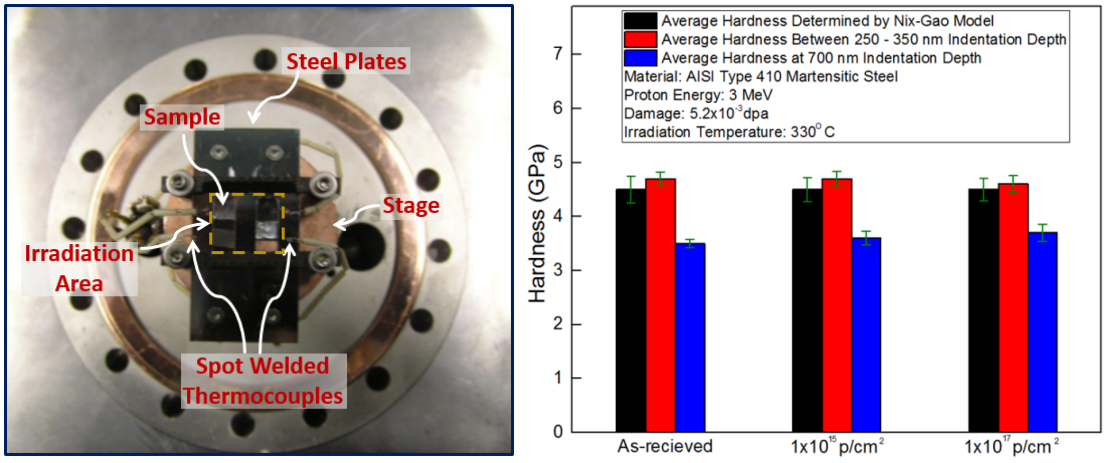
[1]Department of Nuclear and Quantum Engineering, Korea Advanced Institute of Science and Technology,
[2]Department of Materials Science and Engineering, Graduate School of Green Energy Technology, Chungnam National University, Korea
[3]Department of Materials Science and Engineering, Korea Advanced Institute of Science and Technology
[4]New Power Development Project, KEPCO E&C, Republic of Korea
[5]Mechanical System Engineering Department, KEPCO E&C, Republic of Korea

Some ferritic martensitic (F/M) steels such as Eurofer97, T91 and EM10 etc. show significant irradiation hardening, increase in DBTT levels and reduction in ductility due to defects clusters and irradiation loops which restrict the movement of dislocation. This size and density of defects increase with an increase in the irradiation dose and temperature. These microstructural changes can degrade the mechanical properties and reduce the usefulness of F/M steel in nuclear applications.

In addition to high doses, the degradation of the mechanical properties of martensitic steels at doses as low as $3.4x10^{-3}$ dpa also revealed irradiation hardening. However, this research was carried out at temperatures ranging from 60ºC to 100ºC. Therefore, the analysis of F/M 410 steel irradiated at system operation temperature of an in-vessel control rod drive mechanism of compact reactors i.e. ~300ºC and relatively lower irradiation damage due to lower power rating and fluence in compact reactors is vital.

For the above-mentioned reason, the proton irradiation of AISI Type 410 martensitic steel samples was carried out by exposing the ~35 µm thick samples to 3 MeV protons up to $1.0x10^{17}$ p/cm^2 fluence at 330ºC. The assessment of the possible deleterious effects of low-dose irradiation on the mechanical behavior of AISI Type 410 martensitic steel samples was carried out via cross-sectional nano-indentation. No significant difference in mechanical behavior of as received and irradiated samples was observed. This study ensured the integrity of the structural and magnetic components of nuclear reactors made of AISI Type 410 martensitic steel at high temperature irradiation damage level of approximately $5.2x10^{-3}$ dpa.

This work was supported by Nuclear Power Core Technology Development Program of the Korea Institute of Energy Technology Evaluation and Planning (KETEP), Ministry of Trade, Industry & Energy of the Republic of Korea under grant 20131510101680.



Sample loaded on stage of 3 MV Tandem accelerator (left), comparison of average hardness of as-received and irradiated sample (right).

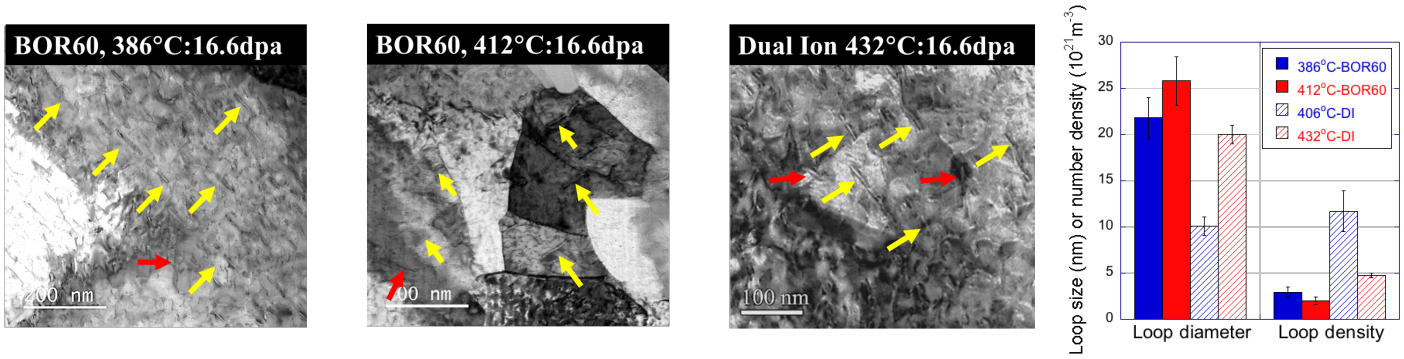
HIGH FIDELITY ION BEAM SIMULATION OF HIGH DOSE NEUTRON IRRADIATION

S.Taller[1], Z. Jiao[1], K. G. Field[2], G.S. Was[1]
[1]Department of Nuclear Engineering & Radiological Sciences, University of Michigan
[2]Materials Science and Technology Division, Oak Ridge National Laboratory

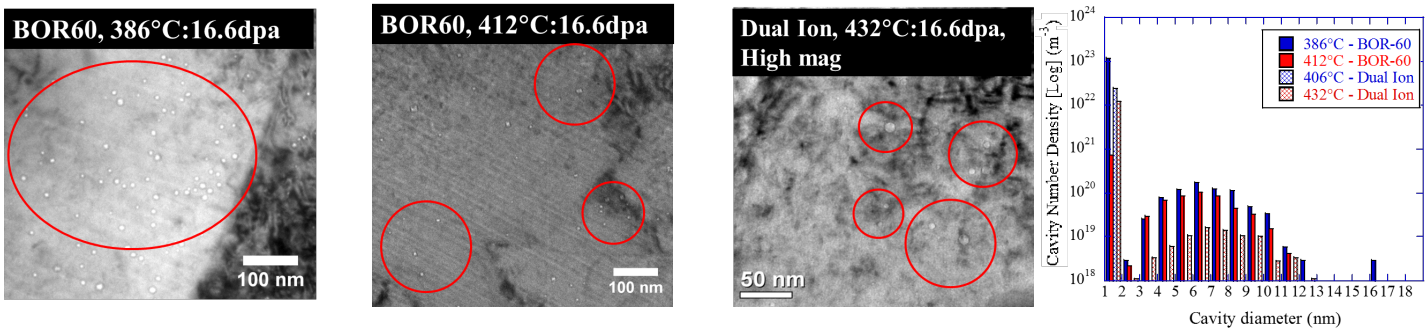
Traditional research efforts to understand radiation-induced processes in materials requires years of comprehensive post-irradiation characterization effort of test reactor produced neutron irradiated material. The same levels of radiation damage can be achieved using heavy ion irradiation under tightly controlled conditions in days or weeks instead of years in a nuclear reactor, albeit with several challenges. The purpose of this work is to address these challenges in using ion irradiation experiments as a surrogate for neutron irradiation.

Several dual ion irradiations were performed using 5.0 MeV defocused Fe^{++} ions to damage the material while simultaneously injecting He^{++} ions in a fixed He/dpa ratio to emulate gas buildup from nuclear transmutation reactions. Bars of T91 were dual ion irradiated to 16.6 dpa with 0.22 appm helium per dpa at 406°C, 432°C, 445°C, and 460°C to compare with multiple conditions from the BOR-60 fast reactor to the same level of damage. These specimens are being examined with transmission electron microscopy and atom probe tomography to determine the effects of simultaneous helium injection and radiation damage on the irradiated microstructure of these materials.

This work is supported by the U.S. Department of Energy under award DE-NE0000639.



A comparison of STEM Bright Field (BF) images in T91 30176 irradiated with neutrons in BOR-60 or dual ions at MIBL (above) under [100] or [110] zone axis conditions to distinguish between a<100> (yellow arrows) and a/2<111> (red arrows) Burgers vector dislocation loops.



Cavities were imaged in STEM BF and High Angle Annular Dark Field (HAADF). Cavities examined up to 1000 nm from the free surface of the ion-irradiated material in 100 nm increments. Underfocused TEM BF images are shown here for clarity of cavity observation. Cavities are visible as white spots with a darker fringe around it.

SHADOW CORROSION OF ZIRCALOY-2

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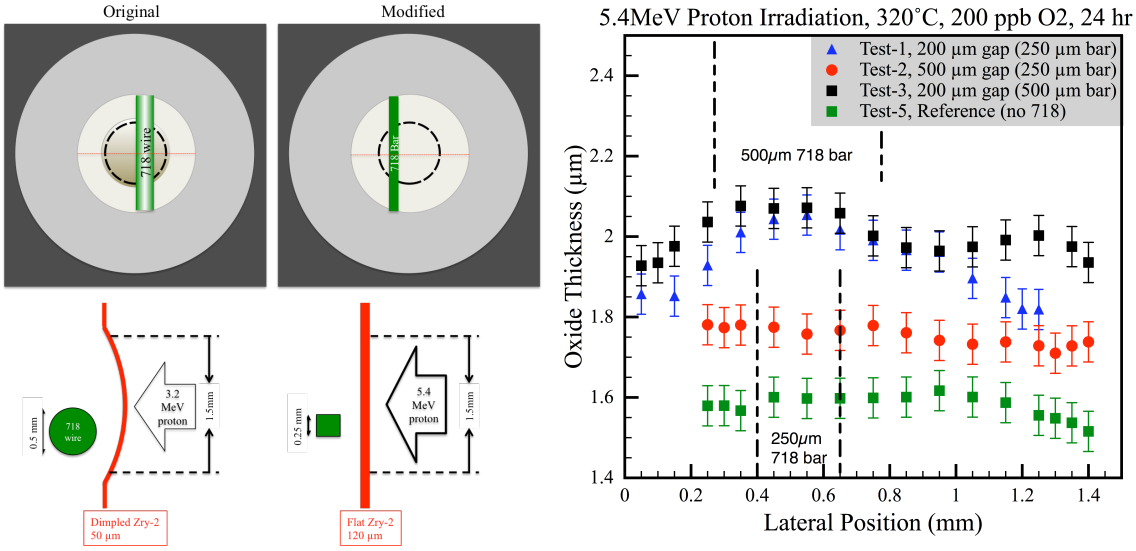
This project was aimed to refine the 2015 initial project results and optimize the experiments to maximize the shadow corrosion effect through modification of the sample design, the separation distance between Zry-2 and Inconel 718 and surface area of the Inconel 718 bar.

A flat sample design was implemented in this study to reduce the uncertainty due to the curved sample surface in the previous study. Hence, a higher energy proton beam was used to compensate for the loss of proton penetration depth caused by the increased sample thickness.

Several mechanisms have been proposed to explain the appearance of shadow corrosion, and the majority are related to the electrochemical nature of the Zircaloys. However, to date, shadow corrosion has only been observed on samples exposed in reactor. This implies the possible mechanisms by which radiation assists the shadow corrosion process; (1) by increasing the conductivity of the oxide film on Zr alloy, (2) by increasing the conductivity of the water by creating radiolysis products.

In this study, several aspects which could influence shadow corrosion had been evaluated: First, effects of anode to cathode separation distance were verified. The resulting oxide thickness was inversely proportional to the separation distance. Second, cathode-to-anode (C/A) area ratio had negligible effect on the galvanic current density and hence, its influence on the corrosion rate was negligible. Finally, the shadow corrosion was more pronounced on tests conducted at higher temperature.

This research was supported by the AREVA Germany, Contract No.GF01/1016033557.



Geometry of the new and original sample designs and positioning of the Inconel 718 for the shadow corrosion experiment (left), oxide thickness measurement across the irradiated area (right).

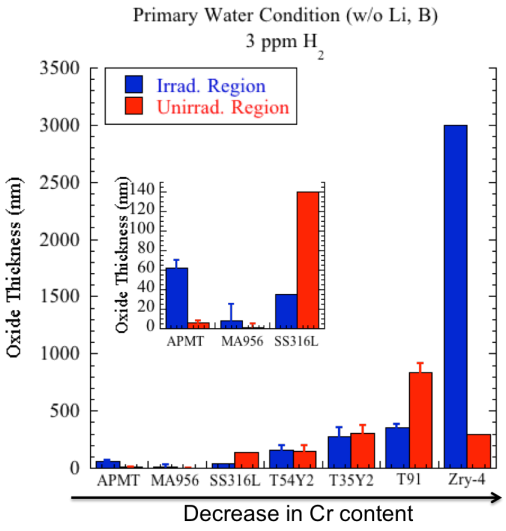
OXIDATION OF Fe-Cr-Al ALLOYS IN SIMULATED PWR PRIMARY WATER DURING IN-SITU PROTON IRRADIATION

P. Wang[1], D. Bartels[2], G. S. Was[1]
[1]Department of Nuclear Engineering & Radiological Sciences, University of Michigan
[2]Department of Chemistry and Biochemistry, University of Notre Dame

Following the high oxidation rate of fuel rods during the Fukushima Daiichi nuclear accident in 2011, the emphasis for nuclear fuel R&D activities has shifted to the development of accident-tolerance of LWR fuel. FeCrAl alloys were selected as one of the candidate materials systems for Accident-Tolerant Fuel application due to their excellent high temperature (1200˚C) corrosion resistance in steam, greater strength, and resistance to stress corrosion cracking. However, their general corrosion resistance is borderline and more importantly, no data exist on whether the iron-base alloys being considered for fuel cladding for ATF fuel are susceptible to irradiation-accelerated corrosion. There is also lack of understanding of the mechanisms by which corrosion rates are accelerated to be of any predictive value to the ATF program. The objective of this work is to assess the corrosion behavior of ATF candidate iron-based alloys under normal LWR operating conditions consisting of high temperature, relevant water chemistry and irradiation.

In-situ irradiation-corrosion experiments were performed on several ATF candidate materials: ferritic-martensitic alloy (T91), and FeCrAl alloys (MA956, APMT, T54Y2 and T35Y2) in high purity water at 320˚C containing 3 appm H_2 using a 5.4 MeV protons beam. The thin sample (80 µm) acts as a "window" to allow protons to fully penetrate and deposit their energy in water to induce radiolysis while creating displacement damage in the metal. Results were compared with SS316 and Zircaloy-4 which were also tested previously under the same conditions. The corrosion rate increased with decreasing alloy Cr content, indicating the importance of Cr as an alloying element for corrosion resistance. The thinner oxide observed in the irradiated region indicated that oxide dissolution may have occurred under irradiation due to the more corrosive environment created by radiolysis.

This research was supported by the DOE-NEUP, Contract No. DE-NE0008272.



Oxide thickness measurement comparison of the ATF candidate alloys against SS316 and Zircaloy-4

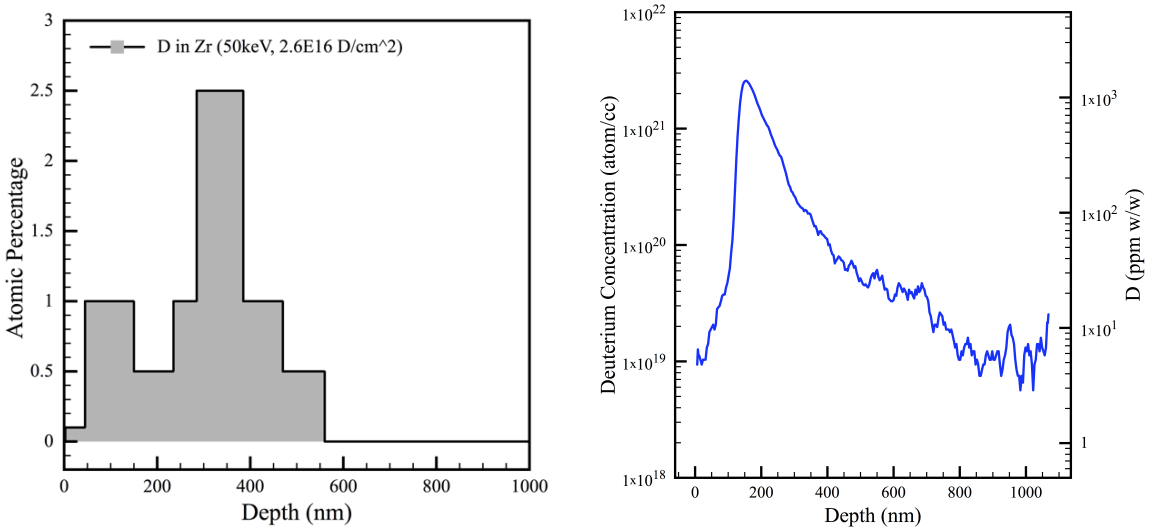
DOSE RATE EFFECT ON CORROSION AND HYDROGEN PICKUP FACTION OF ZIRCOLOY-4 IN DEUTERATED PWR PRIMARY WATER USING IN-SITU PROTON IRRADIATION-CORROSION EXPERIMENTS

P. Wang, G. S. Was
Department of Nuclear Engineering and Radiological Sciences, University of Michigan

This project aims to understand how irradiation dose rate affects corrosion and hydrogen pickup behavior of zirconium alloys in PWR primary water. Previous study on in-situ proton irradiation-corrosion had indicated that corrosion rate increased with dose rate. Hence, a detailed study of corrosion rate as a function of dose rate was proposed. Furthermore, the hydrogen loss through the thin zirconium sample to the beamline vacuum was addressed by applying a hydrogen permeation barrier layer (e.g. ~100nm amorphous-Al_2O_3) to the vacuum side of the sample. Deuterium oxide (D_2O) will be used as corrosion medium, and Secondary Ion Mass Spectroscopy (SIMS) will be used to determine the deuterium pickup and distribution.

To accurately determine and quantify the deuterium concentration depth profile, a standard Zr sample with known deuterium concentration was created using the 400 keV ion implanter. Energy Recoil Detection (ERD) measurement was conducted using a 3 MeV He beam in the Ion Beam Analysis chamber to confirm the implanted fluence and distribution of deuterium within the implanted sample. This standard sample of deuterium implanted zirconium will be used as a reference sample to determine the sensitivities of each ion that was measured by SIMS, and produce a relative sensitivity factor that can be used later for further analysis of the real D_2O corroded samples to yield quantitative data.

This research was supported by the Consortium for Advanced Simulation of Light Water Reactors (CASL) under U.S. Department of Energy Contract No. DE-AC05-00OR22725.



Modeled deuterium concentration from ERD measurement (left), and corrected deuterium concentration profile from SIMS measurement on the standard sample (right).

TEM *IN SITU* MECHANICAL TESTS OF ION IRRADIATED OXIDE DISPERSION STRENGTHENED ALLOY

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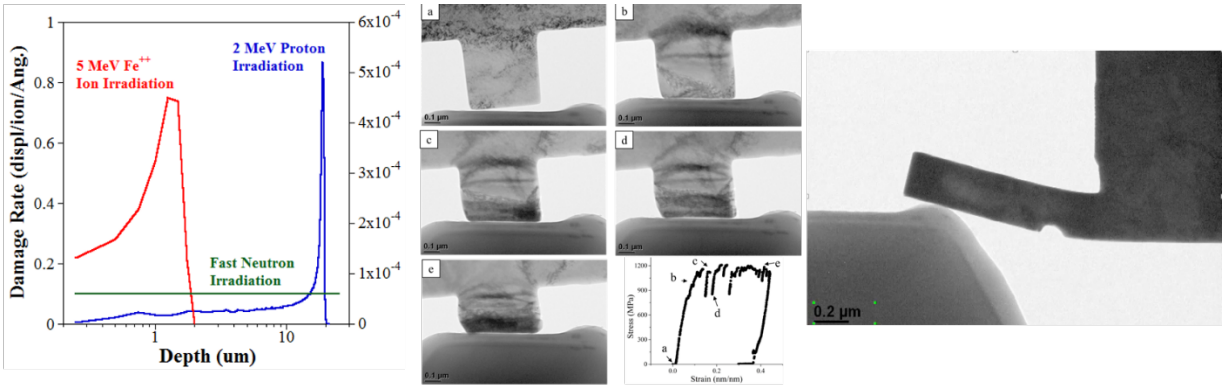
[1]Micron School of Materials Science and Engineering, Boise State University
[2] School of Nuclear Engineering, Purdue University

Increasing use of charged particle irradiation in the evaluation of nuclear reactor candidate materials requires novel techniques to determine the mechanical properties in the near-surface, few-μm thick irradiation damage layers. *In situ* transmission electron microscopic (TEM) mechanical testing is one such promising method because they are able to isolate mechanical performance in the thin, irradiated layer, from the unirradiated bulk. In this project, we conducted 2 MeV proton and 5 MeV Fe^{2+} ion irradiations of a model Fe-9% Cr oxide dispersion strengthened (ODS) alloy at MIBL. Subsequently, we tested the irradiated and as-received materials in both micro-pillar compression and micro-cantilever bend geometries.

The goal of the micropillar compression testing was to understand the feasibility of collecting basic mechanical properties at this size scale. Yield strengths were measured directly from the TEM *in situ* pillar compression tests and are consistent with Orowan calculations based on the irradiated microstructure. Measured elastic modulus values required adjustment for the deformation and deflection in the base of the pillars, but were also within the expected range. A size effect was observed in pillars with a minimum dimension ≤ 100 nm due to the low inter-obstacle spacing.

The goal of the cantilever beam testing was to ascertain whether there is a change in the grain boundary cohesion of the material after irradiation, due to grain boundary chemical segregation. The cantilevers were designed based on the expected fracture toughness of the material, and with a grain boundary of interest centered below the notch. However, even after testing multiple cantilevers, we have been unable to attain fracture, suggesting that the effect of radiation induced segregation on grain boundary cohesion may be so small that it is below the resolution of this testing approach. Ongoing work will further refine the design of the cantilevers to attempt to induce fracture.

This research was sponsored in part by the US Nuclear Regulatory Commission NRC-HQ-84-14-G-0056.



[Left] Comparison of damage profiles for self-ion, proton and neutron irradiation. [Middle] In situ compression test of as received ODS 400 nm x 400 nm x 100 nm pillar. (a-e) show pillar condition at each labeled point in the stress-strain curve. Dislocation bursts are observed at (c) and (d), representing load drops observed on the stress-strain curve. [Right] Screen shot from in situ cantilever testing.

DEFECT DRIVEN METALLIC OXIDES FOR RECHARGABLE LITHIUM ION BATTERIES

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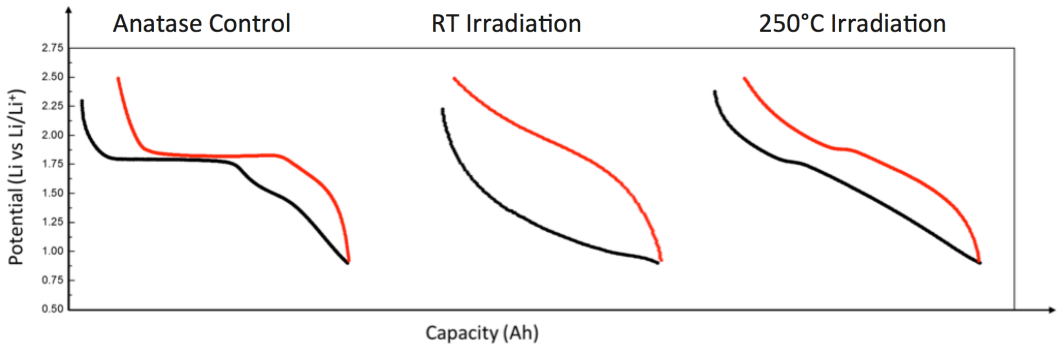
[1]Micron School of Materials Science & Engineering, Boise State University
[2]School of Nuclear Engineering, Purdue University
[3]College of Mines & Earth Sciences, University of Utah

Rechargeable lithium-ion batteries have transformed the portable electronics industry and are considered amongst the most promising electrochemical energy storage methods for electric vehicles and renewable energy systems. As society shifts further towards clean and renewable energy sources, energy storage becomes evermore critical. Nanostructured titanium dioxide (TiO_2) is a promising electrode material for rechargeable Li-ion batteries because of its improved electrochemical reactivity with lithium compared to bulk TiO_2. However, researchers have yet to achieve the theoretical charge storage capacity of 1.0 Li/Ti at room temperature. Previous work has demonstrated that TiO_2 nanotubes with cation vacancies exhibit desirable short- and long-range order to promote lithium ion intercalation and charge storage. In addition, synthetic methods of doping and ion implantation can induce defects, which enhance the charge storage capacity of metal oxides. This project hypothesizes that irradiation can produce intentional defects in nanostructured crystalline TiO_2 electrode materials, thereby enhancing the electrochemical performance of the TiO_2 electrode in a Li-ion battery.

For this project, we grew anatase TiO_2 nanotubes on titanium foil. The nanotubes were irradiated at MIBL with 400 keV protons to doses of ~0.4 displacements per atom (dpa) at room temperature and at 250°C. The irradiated foils were then placed into lithium ion coin cell batteries, and their electrochemical behavior was recorded (see figure). In the anatase unirradiated control specimen, a three-step reaction was observed. First, lithium insertion led to the formation of a solid solution, as indicated by the potential drop from the open-circuit potential to 1.7 V in the leftmost image in the figure. This was followed by a two-phase storage reaction, indicated by the presence of a long, distinct plateau at 1.7 V. Finally, interfacial storage was indicated by monotonic curves beyond 1.7 V. However, the irradiated specimens behave markedly different than the unirradiated control. The change in voltage profile, seen in the middle and rightmost images in the figure, indicated a change in charge storage mechanism from two-phase storage in the unirradiated control, to a solid solution charge storage mechanism. Future work involves irradiating single crystal TiO_2 using protons as well as heavier ions (e.g. Ni^+, Nb^+) to understand the types of defects produced by irradiation, to help us fundamentally explain the change in charge storage mechanism.

This work is supported by the National Science Foundation through award number DMR-1408949.



Effect of 400 keV proton irradiation on Li^+ charge capacity of TiO_2 nanotubes.

IRRADIATION EFFECTS IN FERRITIC-MARTENSITIC STEELS AT VERY HIGH DAMAGE LEVELS

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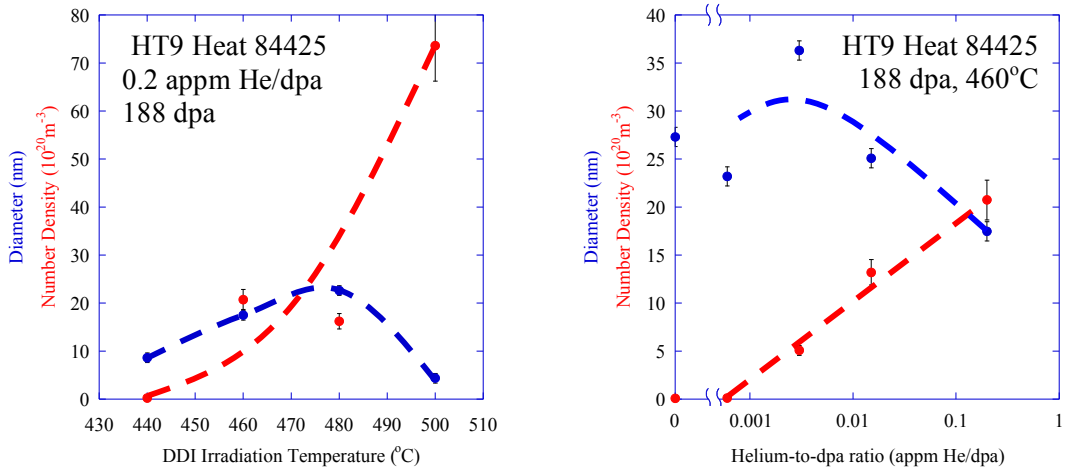
Understanding the microstructural evolution in ferritic-martensitic steels is important for predicting the safety and reliability of future reactors. Dual ion irradiation experiments have been performed on a heat of HT9 ferritic-martensitic steel to examine the effects of temperature and co-injected helium on dislocation loops, secondary phases and cavities. Irradiations were performed at the Michigan Ion Beam Laboratory with 5 MeV Fe^{++} ions from a 3 MV Pelletron accelerator and ~2 MeV degraded He^{++} ions from a 1.7 MeV Tandetron accelerator to simulate damage and transmutation gas, respectively. Experiments were performed to 188 dpa for temperatures from 440°C to 500°C at a constant helium-to-dpa ratio of 0.2 appm He/dpa and helium-to-dpa ratios of 0 to 0.2 appm He/dpa at a constant temperature of 460°C. The microstructural behavior was examined using scanning transmission electron microcopy. The dislocation microstructure, precipitate formation and cavity behavior were examined for each condition to map out the effect of temperature and helium on microstructure evolution.

Dislocation and precipitate evolution followed similar trends with temperature at a constant helium-to-dpa ratio. The loop and G-phase precursor diameters increased with increasing temperature while the densities decreased with increasing temperature. M_2X precipitates were observed only at 440°C. Cavity formation was observed under almost all conditions. For a constant helium-to-dpa ratio of 0.2 appm He/dpa, cavity size exhibited a peaked behavior with a maximum at 480°C. Cavity density tended to increase with increasing temperature. This resulted in a peak swelling temperature of 480°C for a helium-to-dpa ratio of 0.2 appm He/dpa.

For a constant temperature of 460°C, dislocation loops and G-phase precursors were insensitive to changes in helium-to-dpa ratio. M_2X precipitates were observed in various amounts for all helium-to-dpa ratios less than 0.2 appm He/dpa. In contrast, cavity evolution exhibited a strong dependence on helium-to-dpa ratio. A decreasing ratio led to a decreasing density and a decreasing diameter. At low enough helium levels, the diameter saturated while the density dropped almost to 0. The competing effects of lower cavity nucleation with higher cavity growth with a decreasing ratio led to a peak in swelling at 0.003 appm He/dpa.

This work is supported by the TerraPower, LLC.



Cavity diameter and density in HT9 heat 84425 as a function of (a) temperature at 188 dpa with a helium-to-dpa ratio of 0.2 appm He/dpa and (b) helium-to-dpa ratio at 188 dpa and 460°C.

IRRADIATION DAMAGE IN NI-BASED ALLOYS

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Alloy 690 is widely used in existing LWR plants due to its superior stress corrosion cracking (SCC) resistance compared to Alloy 600. Alloy 625 is used in more limited applications but offers the benefits of both high strength (in the aged condition) and corrosion/SCC resistance. Research has shown that both alloys can undergo phase changes due to thermal or irradiation exposure. In the precipitation-hardened condition, Alloy 625 "softens" during neutron irradiation as the strengthening precipitates decompose and metastable precipitates form. However, the nature and rates of these transformations as a function of exposure conditions are not well understood.

The objective of this research is to address thermal and irradiation-induced transformations mechanisms of commercial Alloys 625, 625 plus, 690 and model binary Ni-Cr alloys. Here, alloys 625, 625 plus, 690 and Ni-33 at%Cr were irradiated to 1.5 dpa at 300 °C using 2 MeV protons. An indium thin foil was compressed between the irradiated samples and the stage as a heat transfer medium. During the irradiation, four thermocouples were welded on the guide bars to calibrate the temperature. From the infrared thermal image, the temperature was controlled well (the average temperature of all eighteen areas of interest was 300.8 ± 6.0 °C) throughout the irradiation process, indicating a successful irradiation experiment. The resulting microstructures will be characterized using transmission electron microscopy, atom probe tomography and nano-indentation.

This work is funded by the Department of energy Nuclear Energy University Program.

VOID SWELLING IN ION-IRRADIATED MOLYBDENUM

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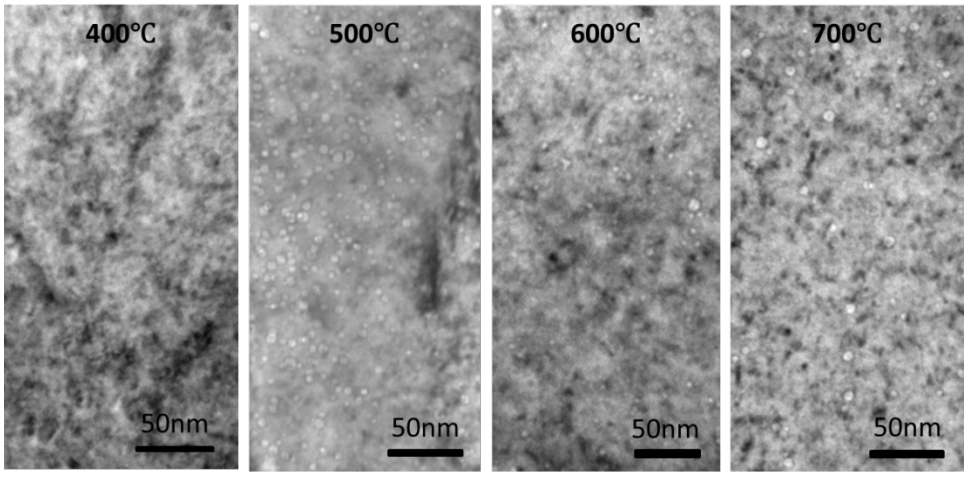
Molybdenum is a refractory metal that has been considered for applications at high temperatures where an inert or reducing atmosphere can be maintained, such as structural components and advanced nuclear applications. The change in the properties of molybdenum and molybdenum alloys after irradiation has received sporadic attention over the last 40 years. The changes in microstructure and resulting changes in the mechanical properties of molybdenum and its alloys are strongly dependent on the irradiation temperature. Swelling is dependent on both the size and number density (ND) of voids, and serves as an indirect measure of defect density.

Heavy-ion irradiation experiments have been performed on heats of molybdenum alloys to determine void swelling behavior at 400°C to 700°C with 20 displacements per atom (dpa). Irradiations were performed with 10 MeV Ag^{3+} ions on samples using a raster beam with a 3 MV Pelletron accelerator at the Michigan Ion Beam Laboratory.

Swelling was observed in samples irradiated at 500°C, 600°C, and 700°C. Void density decreased while average void size is increased slightly with temperature. The swelling data of the molybdenum alloys are consistent with literature results.

This work is supported by Shanghai Nuclear Engineering Research and Design Institute.



TEM images (under focus) of 10MeV Ag^{3+} ions irradiated molybdenum alloys at 400°C, 500°C, 600°C, and 700°C.

IRRADIATION RESPONSE AND IASCC BEHAVIOR OF ADDITIVELY MANUFACTURES 316L STAINLESS STEEL

G. S. Was[1], M. Wang[1], M. Song[1], X. Lou[2]
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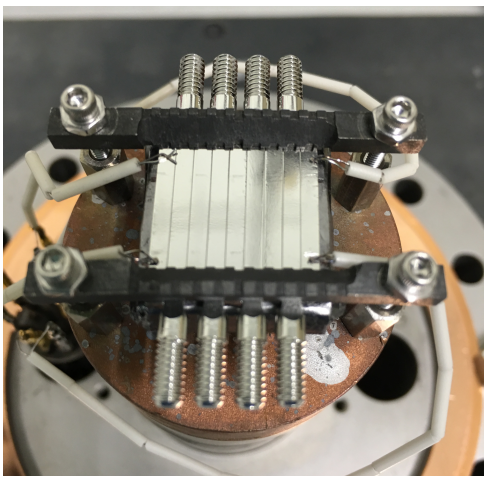
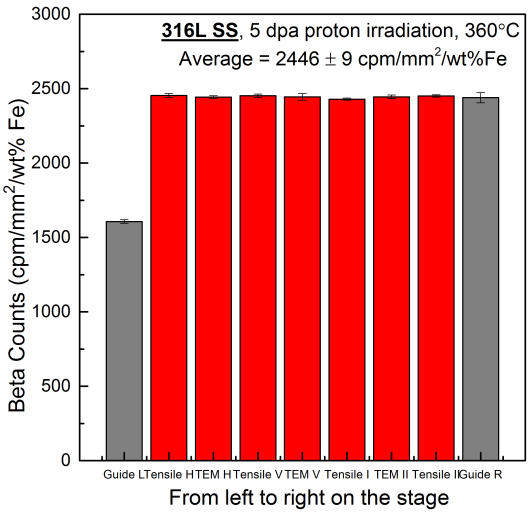
[2]General Electric Global Research

The major objective of the proposed program is to develop a novel SCC and irradiation resistant 316L SS with high strength and good ductility using the direct laser mantling (DMLM) process to save on both deployment schedules and overall life cycle costs with improved plant reliability, and to demonstrate the feasibility and benefits of this process in nuclear applications.

Among the behaviors of interest are the microstructure changes during irradiation, and stress corrosion cracking behavior in the irradiated condition. As such, samples of both wrought and additively manufactured (AM) 316L were irradiated with 2 MeV protons at 360°C to 5 dpa. Both tensile and TEM bars were irradiated. The wrought tensile bars were in two orientations relative to the forming direction; horizontal (H) and vertical (V). Another set of samples were irradiated with 5 MeV Fe^{2+} ions at 400°C to 100 dpa. These irradiation conditions represent a lower dose (5 dpa) condition that is sufficient to assess IASCC resistance, and a high dose (100 dpa) condition that will be used to assess IASCC but also dimensional stability (void swelling resistance).

Samples were then tested in constant extension rate tensile (CERT) mode in simulated boiling water reactor normal water chemistry (BWR-NWC) at 288°C, in a flowing system. Water chemistry was controlled to keep the conductivity below 2 μS/cm and the oxygen content at 200 ppm. Samples were strained at a rate of 3×10^{-7} s^{-1} to a plastic strain of 4%. The cracking behavior will be assessed in both the irradiated and unirradiated sections of the samples to determine the role of irradiation on the IASCC susceptibility.





Sample arrangement on the irradiation stage (left) and resultant beta activity following 5 dpa irradiation with 2 MeV protons (right) showing the sample-to-sample uniformity of the irradiation.

MECHANISTIC STUDY OF NANOCRYSTAL PLASMA POLYMERIZATION OF COLLOIDAL NANOCRYSTAL ASSEMBLIES

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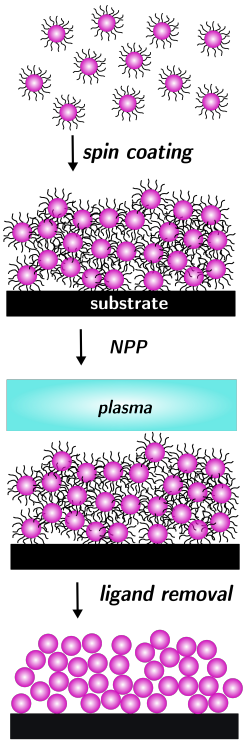
Colloidal nanocrystal assemblies could serve as unique precursors for the fabrication of materials by design. One of the key obstacles is the complete removal of the ligands that functionalize the surface of the nanocrystals. Plasma etching of CNAs (termed as Nanocrystal Plasma Polymerization, NPP) can efficiently remove ligands from CNAs, while preserving the size and composition of the nanoparticles. However, it is not clear how the plasma effectively removes organics from CNAs that are several hundreds of nanometers thick. Specifically, it is not clear what is the role of plasma-generated UV-light, or radicals generated in the plasma, as well as inside the material. To answer these questions we are characterizing the removal of the ligands from the CNAs as a function of the particle size, type of ligands, superlattice structure, and plasma parameters (feed gas, pressure, power, processing time, geometry). Characterizing the change in the concentration of carbon as a function of processing time will allow us to construct a model of the structural evolution of the CNA and understand the kinetics of etching.

We are close to completing most of the study of the effect of the plasma parameters on ligand etching from ZrO_2 CNAs and in the process of deducing the plasma etching mechanism.

This work is supported by the Member-Specific-Research-Intel program of Semiconductor Research Corporation under Award No. 2015-IN-2582.



Schematic of nanocrystal plasma polymerization process.

CONTROL OF BORON TO TITANIUM RATIO DURING MAGNETRON SPUTTER DEPOSITION OF TiB_X THIN FILMS

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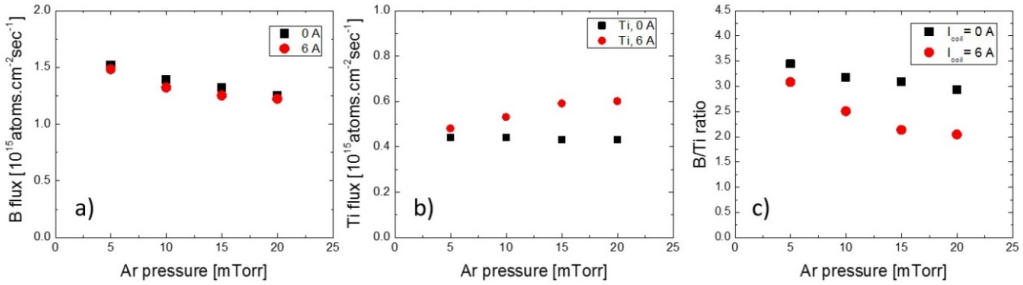
Titanium diboride (TiB_2) hard coatings have been extensively investigated for enhanced functional properties and materials lifetime. They have many desirable physical and mechanical properties such as a high melting point, good thermal and electrical conductivity, high hardness, high wear- and corrosion resistance, as well as excellent chemical stability at elevated temperatures. A common problem in sputter-deposited TiB_x layers is that the film contains excess boron with x ranging from 2.4 to 3.5. It was shown that the excess B can form interesting nanostructures that enhance hardness; however, it is important to control the B/Ti ratio during film growth as a first step to synthesizing epitaxial single crystal films to investigate their fundamental properties. Here, we undertake a study aimed at establishing control of the B/Ti ratio in magnetron sputter-deposited TiB_x thin film and to synthesize stoichiometric TiB_2 layers.

TiB_x layers, ~0.4 μm thick, are grown on electrically-floating Si(001) substrates in a load-locked UHV system [ref] from a 99.99% pure TiB_2 target in Ar (99.999%) discharges at constant a DC power of 100 W for 40 min at T_s = 700 °C with a target-to-substrate separation of 65 mm. An external pair of Helmholtz coils with Fe pole pieces was unitized to create a uniform axial magnetic field B_{ext} aiding the field of the outer magnetron pole in order to controllably focus the discharge plasma near the substrate and thus vary the ion flux impinging at the film growth surface. Varying the coil current from 0 A to 6 A resulted in a change of B_{ext} from 20 to 200 G. TiB_2 layer compositions are determined from Rutherford Backscattering Spectroscopy (RBS) analyses. The probe beam consists of 2 MeV He ions incident normal to the sample surface with the detector set at 170 ° scattering angle. Back scattering spectra are analyzed using the SIMNRA program to obtain B and Ti deposition fluxes as well as B/Ti ratios.

The B deposition flux, shown in Fig.1a, exhibits an expected decrease with pressure due to gas-phase scattering. The Ti deposition flux without B_{ext} similarly shows a limited decrease with p_{Ar}. The novel observation here is that as the pressure is increased, the total Ti deposition flux with B_{ext} increases by about 40%. As a result we controllably reduce the B/Ti ratio from 3.5 to 2, i.e. we reach the stoichiometric composition. The results open the way to synthesizing stoichiometric single-crystal transition metal diborides and determining their fundamental properties. We are currently investigating the effects of substrate temperature and sputtering gas mixture. We intend to continue to use the MIBL facility to provide critical data for our research.

Funded by Swedish Research Council (VR) grant #642-2013-8020 and Air Force Office of Scientific Research.



RBS data for the deposition flux of B (a) and Ti (b) and the B/Ti ratio (c) of TiB_x films deposited by magnetron sputtering at DC power of 300 W min at T_s = 700 °C.

MICROSTRUCTURE RESPOJNSE OF WELDING MATERIALS FOR SA508 RPV STEELS IRRADIATED WITH PROTONS

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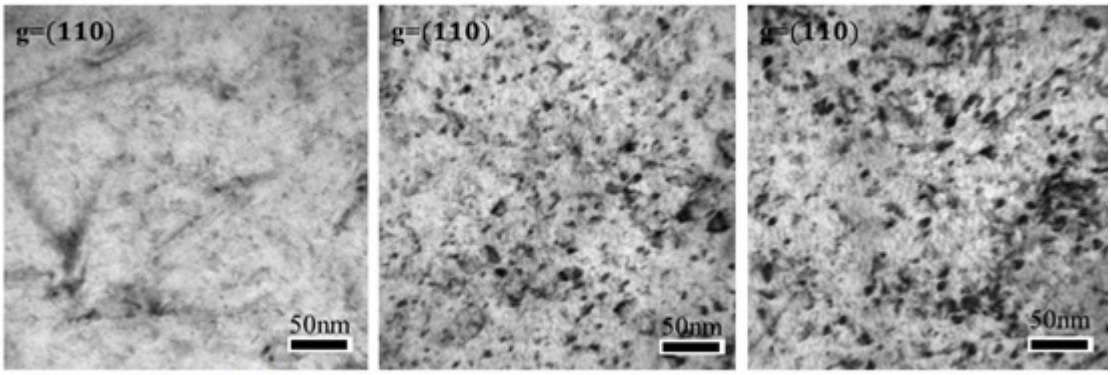
SA508 steels are typically used in nuclear reactors for critical components such as the reactor pressure vessel. Nuclear components are commonly joined using arc welding processes, but with design lives for prospective new building projects exceeding 60 years, new welding materials and technologies are being sought.

In this project, newly designed welding materials were used on the welding joint of SA508 steel. Proton irradiations up to 1 dpa were performed on to study the microstructure evolutions of these new materials at 350°C. Irradiations were performed with a 3 MV Pelletron accelerator at the Michigan Ion Beam Laboratory.

The figure shows microstructures of welding materials with different proton irradiation fluences. The dislocation loops were observed after proton irradiations, which showing as black dots damage in TEM images. Further characterization work will be done in future.

This work is supported by Shanghai Nuclear Engineering Research and Design Institute.



Microstructures of welding materials with different proton irradiation fluences.

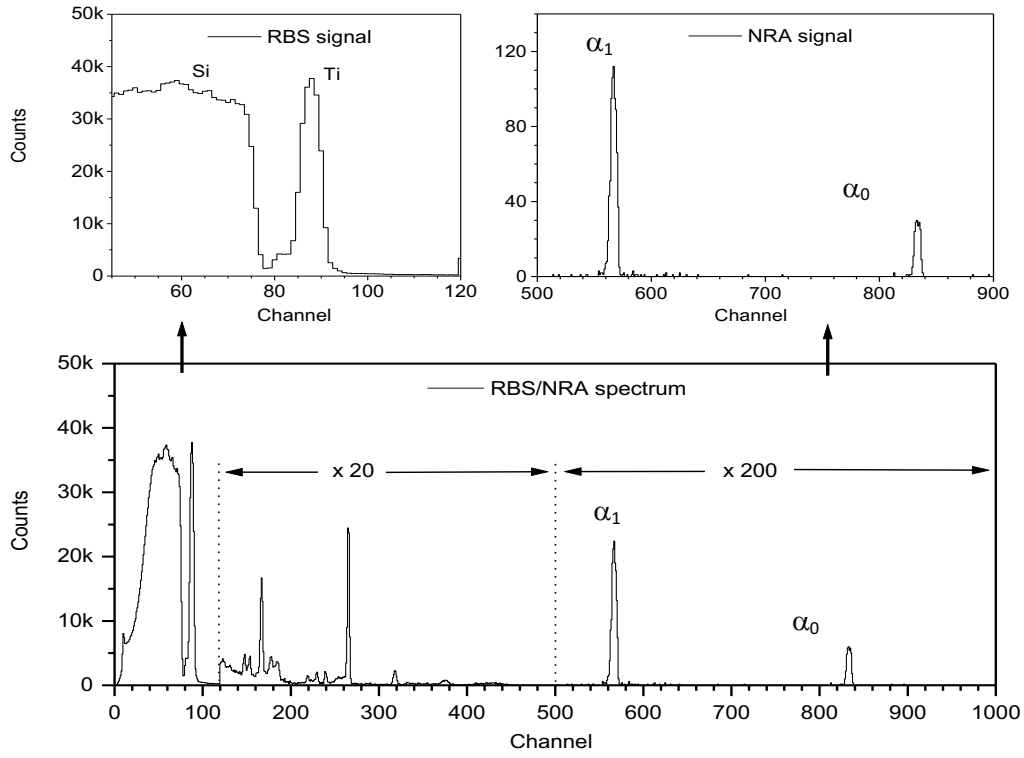
Teaching

NERS 425 LABORTORY ON NUCLEAR REACTION ANALYSIS

M. Atzmon, F. Naab and O. Toader
Department of Nuclear Engineering and Radiological Sciences, University of Michigan

For one of the modules in the NERS 425 course, students conducted an experiment to determine the stoichiometry of a Ti_xN_y sample using the reaction between a deuterium particle and a nitrogen nucleus: $N^{14}(d,\alpha)C^{12}$. Nuclear reaction analysis (NRA) is a well-established surface analysis technique. In this method, an energetic particle (deuterium – produced by the Tandem accelerator at MIBL) interacts with the nucleus of an N atom in the target to give a reaction product (α particle) that can be measured. The students also use the backscattered yield from an RBS experiment to determine the amount of Ti in the sample by implementing simulation codes like RUMP or SIMNRA with the given experimental spectrum.

In the first meeting, prior to the experiment, a short tutorial was given to the students on the accelerator, electronics, detectors, software, and vacuum components. After that, they worked independently with just the basic support from the MIBL staff (required in the setup of the ion beam and the collection of the spectra). The students decided on a few parameters of the experiment (beam energy, time for spectrum acquisition, etc.), and obtained spectra similar to the ones in the figure.



Typical RBS/NRA spectrum for the TiN film obtained during class.
Conditions: beam energy: 1.4 MeV D^+, solid angle 5 msr., detector angle 150°.

MSE 465 CLASS DEMONSTRATION AT MIBL

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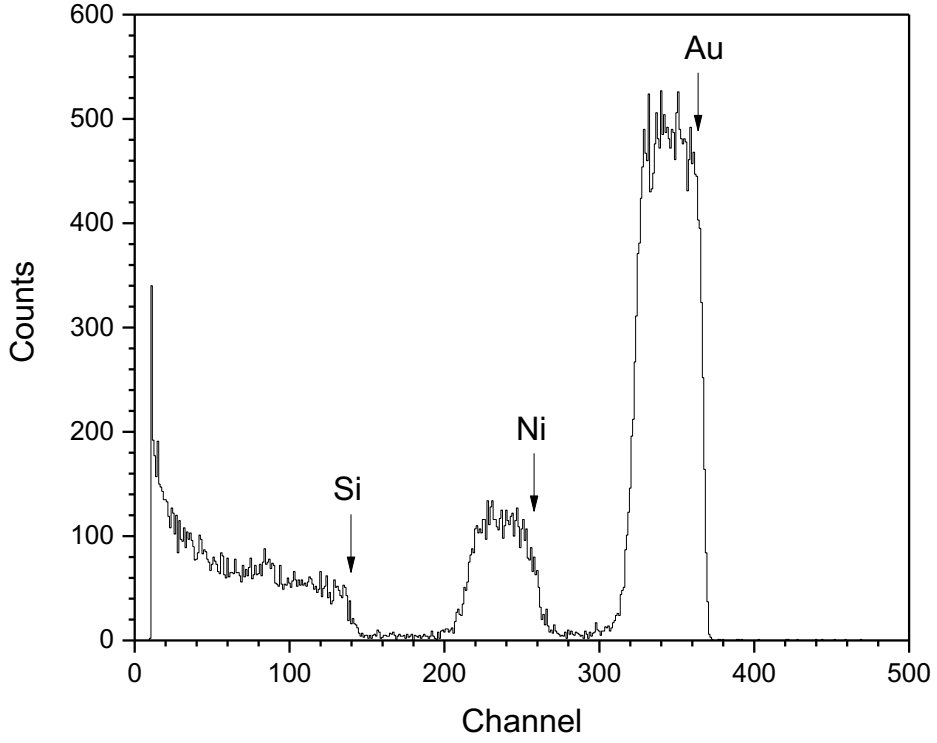
Department of Materials Science and Engineering, University of Michigan

MIBL hosted the MSE 465 class (Structural and Chemical Characterization of Materials) for a session to tour the facility and to demonstrate Rutherford Backscattering Spectrometry, a topic covered in the class. A short tutorial was given on the accelerator, electronics, detectors, software, and vacuum components. As homework, the students were given a spectrum obtained at MIBL and asked to fit the data using SIMNRA, a simulation package commonly used in these experiments. The data for a gold film on a silicon substrate, along with the simulated spectrum, is plotted below.



RBS spectrum of Au film and Ni film on a Si substrate. The spectrum was taken using a He^{++} ion beam at 2 MeV. The scattering angle was 160°.

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G. S. Was, "Accelerated Irradiation for Emulation of in-Reactor Radiation Damage," Structural Materials for Innovative Nuclear Systems (SMINS-4), Manchester, UK, July 2016. **INVITED**

G. S. Was, "Use of Ion Irradiation for Development of Accident Tolerant Fuels," Accident Tolerant Fuels Workshop, China Nuclear Power Technology Research Institute, Shenzhen, China, June 2016. **INVITED**

G. S. Was, B. D. Wirth, A. Motta, "Accelerated Ion Irradiation for Emulation of In-Reactor Radiation Damage," Nuclear Fuel and Structural Materials Symposium, ANS Annual Meeting, New Orleans, June 2016. **INVITED**

K. J. Stephenson, D. Johnson, G. S. Was, "The Role of Dislocation Channeling in IASCC of Neutron Irradiated Stainless Steel," 2016 meeting of the International Cooperative Group on Environmentally Assisted Cracking, Qingdao, China, May 2016.

G. S. Was, J. Gan, "Ion Irradiation for Studying Multiscale Radiation Effects in Actinides," MRS Spring Meeting, Phoenix, April 2016. **INVITED**

G. S. Was, "Ion Irradiation Capabilities and Needs at the Michigan Ion Beam Laboratory, a NSUF Partner Facility," Nuclear Science User Facilities Ion Beam Investment Options Workshop, Idaho National Laboratory, Idaho Falls, March 2016. **INVITED**

G. S. Was, A. Motta, B. D. Wirth, "Accelerated Irradiation for Emulation of in-Reactor Radiation Damage," Accelerated Materials Evaluation for Nuclear Application, 2016 TMS Annual Meeting, Nashville, TN, February 2016. **INVITED**

A. M. Monterrosa, Z. Jiao, E. Getto, G. S. Was, "The Effect of Pre-Implanted Helium on Void Nucleation and Growth in Ferritic-Martensitic Steels," Accelerated Materials Evaluation for Nuclear Application, 2016 TMS Annual Meeting, Nashville, TN, February 2016.

G. VanCoevering, G. S. Was, "Cluster Dynamics Modeling of Void Nucleation and Growth in Ferritic Steels," Accelerated Materials Evaluation for Nuclear Application, 2016 TMS Annual Meeting, Nashville, TN, February 2016.

E. Getto, Z. Jiao, K. Sun, A. M. Monterrosa, G. S. Was, "Microstructural Features in Self-Ion Irradiated HT9 at Very High Dose," Accelerated Materials Evaluation for Nuclear Application, 2016 TMS Annual Meeting, Nashville, TN, February 2016.

M. Song, M. Wang, D. Woodley, Z. Jiao G.S. Was, "Formation of Long Range Order Phase in Commercial Age Hardened Nickel-base Alloys Subjected to Proton Irradiation" 2016 TMS Annual Meeting, Nashville, TN, February 2016.

M. Song, M. Wang, D. Woodley, Z. Jiao, G. S. Was, "Nano-sized γ/γ' Precipitates and Their Strengthening Effect in Commercial Age Hardened Nickle-base Alloys," 2016 TMS Annual Meeting, Nashville, TN, February 2016.

P. Wang, G. S. Was, "Oxidation of FeCrAl Alloys in Simulated LWR Environment During In-situ Corrosion-Irradiation Experiment," 2016 TMS Annual Meeting, Nashville, TN, February 2016.

S. S. Raiman, G. S. Was, "Accelerated Oxide Dissolution During Irradiation-Corrosion of 316L Stainless Steel in Simulated Primary Water," 2016 TMS Annual Meeting, Nashville, TN, February 2016.

S. Taller, Z. Jiao, G. S. Was, "Effect of Helium Implantation Mode on Void Formation in Ion-Irradiated T91 Steel," Accelerated Materials Evaluation for Nuclear Application, 2016 TMS Annual Meeting, Nashville, TN, February 2016.

G. S. Was, M. McMurtrey, D. Johnson, I. Robertson, D. Farkas, "The Importance of Radiation and Deformation in Environmentally Assisted Fracture," Plasticity 2016, Kona HI, January 2016. **INVITED**

O. Toader, F. Naab, T. Kubley and E. Uberseder, “Beamlines Alignment at a Triple Ion Beam Facility”, International Beam Instrumentation Conference (IBIC), September 2016, Barcelona, Spain.

O. Toader, F. Naab, T. Kubley, E. Uberseder and G. Was, “Technical Aspects of Delivering Dual and Triple Ion Beams to a Target”, 24th International Conference on the Application of Accelerator in Research and Industry, Oct 30- Nov 04, 2016 Fort Worth Tx, USA.