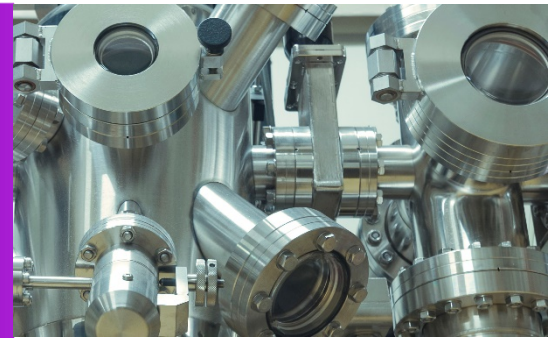


Nanoindentation Study of Property Changes in Irradiated Nuclear Materials



Introduction

The global increase in the use of nuclear technology contributes to nuclear electric power as one of the main growth energy sectors in the U.S, as shown in Figure 1. As a result, there is a strong demand for new engineering materials that can withstand both high temperatures and high doses of nuclear radiation. Damage caused by irradiation in nuclear power plants results in material changes over time. Finding correlation between microstructural and mechanical property changes due to radiation is of critical interest to scientists in developing materials suited for extreme environments and to avoid potential catastrophic failures^{1,2}.

U.S. PRIMARY ENERGY PRODUCTION BY MAJOR SOURCES, MARCH 2019

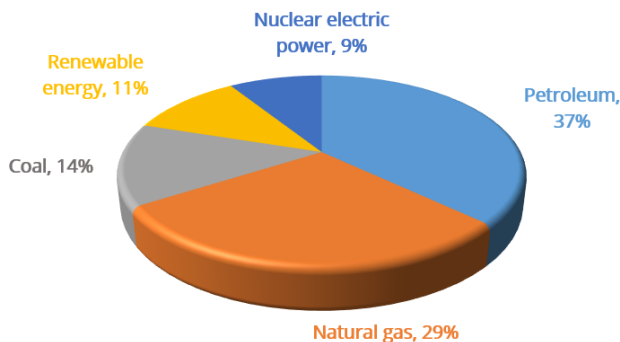


Figure 1. U.S. primary energy production by major resources, 2019, with 9% associated with nuclear electric power.

Nanoindentation has been shown to be a unique technology capable of measuring properties at small scales². Irradiation effects into a surface as function of dose, temperature and material has been vastly studied in the literature using nanoindentation techniques³. In this application note, we studied the extent of layer damage by charged iron particles and protons (Figure 2) into the bulk of irradiated steel by measuring mechanical properties as a function of penetration depth. The objective of this study was to show the capability,

reliability and accuracy of the data collected by the KLA Nano Indenter® G200 testing on irradiated materials.

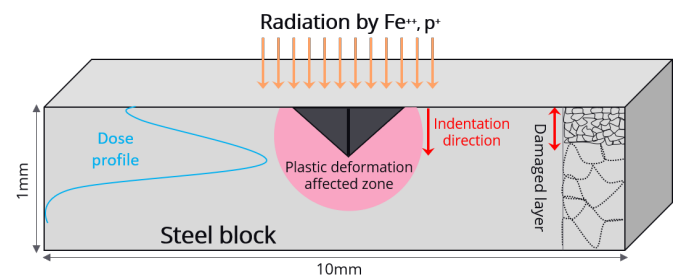


Figure 2. Schematic of irradiated steel with high energy ions/protons penetrating into the bulk and the affected damaged layer. The blue graph at the left shows the radiation dose profile. Indentation tests were performed at different depths in the same direction as the radiation to measure hardness change as a function of depth.

Experimental Method

HCM12A is highly engineered steel grade used in nuclear reactors. A rectangular block of this material was irradiated by Fe^{++} ions and protons, as shown in Figure 2, to emulate neutron irradiation in actual nuclear power plant environments. Samples were irradiated with a dose of 3dpa of each particle type at a temperature of 500°C.

Nanoindentation was performed in quasi-static cyclic load-controlled mode in the direction shown in the Figure 2 schematic, and hardness was measured as a function of depth range. The tests were also repeated on as-received reference steel samples to evaluate the effects arising from size or surface roughness and to find the final delta hardness values caused only by radiation-induced hardening. A sharp Berkovich indenter tip was used and the tests were performed using cyclic loading for 10 cycles and a maximum load of 20mN.

Results and Discussion

Figure 3 shows the results of the nanoindentation testing on three different samples: as-received, irradiated by charged iron particles (Fe^{++}) and charged protons (p^+). The graph shows, as

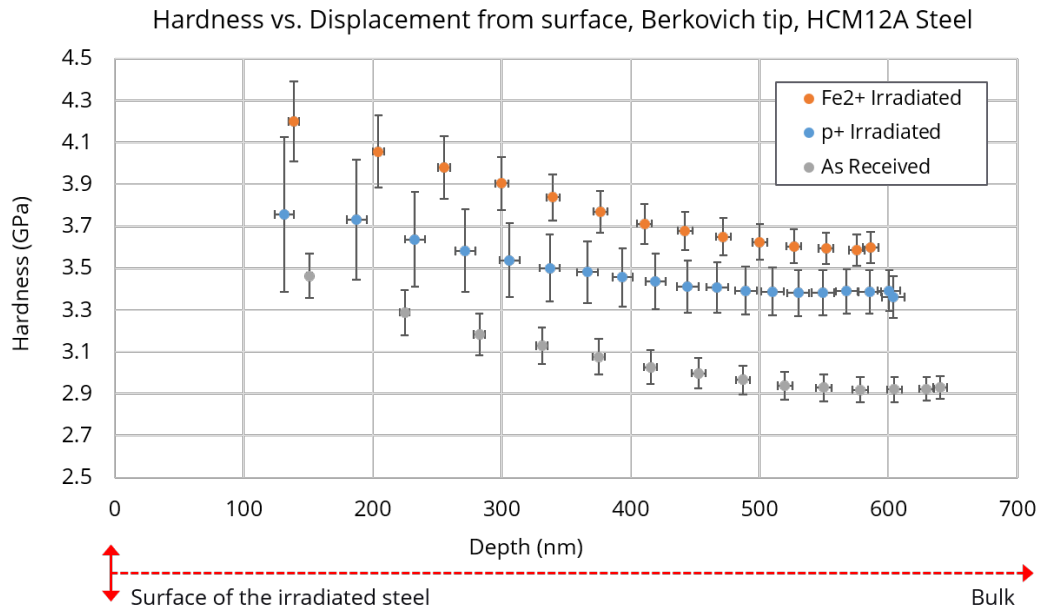


Figure 3. Hardness measurements of the damaged layer caused by irradiation as a function of depth. Hardness was measured for as-received sample (gray) and samples irradiated with Fe^{2+} ions (orange) and protons (blue) with a 3dpa dose at 500°C.

expected, a decrease in hardness with increasing distance from the surface into the bulk.

The higher hardness values at the surface are partly due to surface roughness/size effects, which are also reflected by the as-received sample. Also, as the indenter presses deeper into the bulk, it samples some unirradiated material. Evidence from transmission electron microscopy (TEM) and atom probe tomography (APT) showed a heavily damaged microstructure at shallow irradiation depths^{4,5}. The high energy of radiating particles causes atomic scale defects in the microstructure such as nano-voids, dislocation loops, nanoclusters, etc., that are captured by high resolution microscopy and are reflected by changes in measured nanomechanical properties⁴. Results from nanoindentation use hardness to verify mathematical models that correlate micro-structural damage to irradiated dose.

Conclusions

Nanoindentation was used as a powerful and fast technique to study the properties of shallow damaged irradiated layers at elevated temperatures. Results revealed the extent of the damaged layer into the bulk of the nuclear material due to the heavy radiation. Microstructure characterization has shown evidence that verifies the high hardness measured at the surface layer of the irradiated materials.

Acknowledgments

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References

1. P. Hosemann, D. Kiener, Y. Wang and S.A. Maloy, "Issues to consider using nano indentation on shallow ion beam irradiated materials," *Journal of Nuclear Materials*, volume 425 (2012) 136-139.
2. A. Lupinacci, K. Chen, Y. Li, M. Kunz, Z. Jiao, G.S. Was, M.D. Abad, A.M. Minor and P. Hosemann, "Characterization of ion beam irradiated 304 stainless steel utilizing nanoindentation and Laue microdiffraction," *Journal of Nuclear Materials*, volume 458 (2015) 70-76.
3. A. Kareer, A. Prasitthipayong, D. Krumwiede, D.M. Collins, P. Hosemann and S.G. Roberts, "An analytical method to extract irradiation hardening from nanoindentation hardness-depth curves," *Journal of Nuclear Materials*, volume 498 (2018) 274-281.
4. C.K. Dolph, D.J. da Silva, M.J. Swenson and J.P. Wharry, "Plastic zone size for nanoindentation of irradiated Fe-9%Cr ODS," *Journal of Nuclear Materials*, volume 481 (2016) 33-45.
5. M.J. Swenson and J.P. Wharry, "Nanocluster irradiation evolution in Fe-9%Cr ODS and ferritic-martensitic alloys," *Journal of Nuclear Materials*, volume 496 (2017) 24-40.

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