Depth-Dependent Hardness of Irradiated Steel

Introduction

Commercial nanoindentation was conceived and developed in Oak Ridge, Tennessee, precisely for its unique ability to characterize the mechanical strength of ion-irradiated surfaces. By continuously measuring load and penetration throughout the entire indentation cycle, Young's modulus and an equivalent Vickers hardness may be determined at the sub-micron scale[1,2]. Furthermore, these same properties can also be measured as a continuous function of surface penetration by dynamic nanoindentation, which superimposes a small oscillation on the semi-static loading[3,4]. In this work, the KLA iMicro nanoindenter is used to measure the near-surface hardness of an irradiated steel, $Fe_{14}Cr$.

Sample Preparation

After fabrication, the high-purity binary alloy $Fe_{14}Cr$ was heated at 850°C for one hour. The surfaces of two samples, one control and one irradiated, were mechanically polished with silica paper to 4000 grit, followed by a diamond suspension polish with decreasing particle size down to 0.05µm. Ion irradiation was conducted at the Michigan Ion Beam Laboratory at the University of Michigan, Ann Arbor. Exposure was 8MeV Fe^{2+} at 450°C with a dose rate of 10^{-4}dpa/s, where dpa is the displacement per atom. The Stopping and Range of Ions in Matter (SRIM) simulation predicted an irradiated layer of ~2µm, with an integrated damage of 3.5dpa. No further polishing was performed after irradiation.

Nanoindentation

The KLA iMicro nanoindenter was used to perform 25 indentations on both the irradiated and control $Fe_{14}Cr$ to a peak depth of 1000nm using the iMicro test method "Advanced Dynamic E and H." Indentations were performed on the same surface and in the same direction as the irradiation. The indentation strain rate was 0.05/sec, and the superposed oscillation was 2nm at 45Hz. Each indentation gives a continuous measurement of both Young's modulus and Vickers hardness throughout the loading process. Properties were calculated as described elsewhere for nanoindentation[1,2] with one exception: contact depth was calculated as equal to the total depth, not less than the total depth by the amount of the surface deflection. This is a common assumption for metals that do not manifest sink-in of the surface around the indentation.

Results and Discussion

The Young's modulus and Vickers hardness for both samples are plotted in Figure 1. Each trace represents the average measurement for the parameter (Young's modulus or Vickers hardness) over all 25 tests, with the error bars spanning one standard deviation.

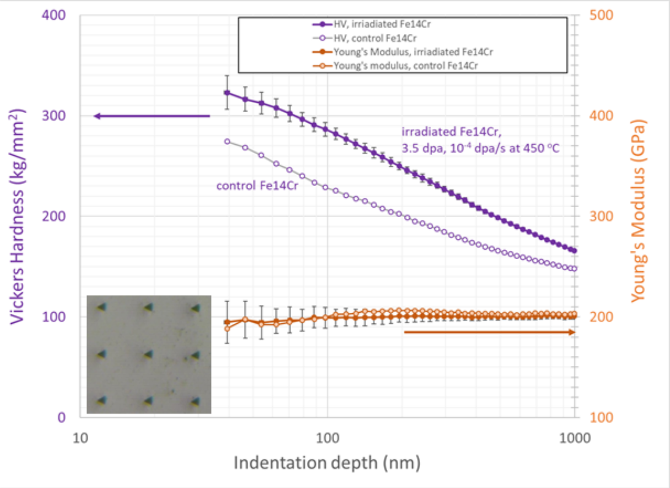

Figure 1. Vickers hardness (purple) and Young's modulus (orange) as a function of indentation depth for both control irradiated $Fe_{14}Cr$. Inset: indentations in irradiated $Fe_{14}Cr$, with 1µm depth and 40µm spacing.

The measurement of Young's modulus is reassuring. First, it is constant and measurable for indentation as small as 40nm. Second, the value of 200GPa is reasonable for steel. Third, radiation exposure has no significant effect on the Young's modulus, as evidenced by the similarity between the irradiated and control samples.

Vickers hardness is highest near the surface, gradually decreasing with depth for both the irradiated and the control sample. The decreasing hardness of the control is due to a convolution of hardening due to polishing and indentation size effect. The term “indentation size effect” describes the reality that many metals are stronger in small volumes, due to the lack of local dislocations necessary for plasticity[5]. The difference in hardness between the irradiated sample and the control is due to the radiation exposure, and this difference is largest at a penetration depth of about 100nm.

Conclusion

Dynamic nanoindentation is the key to characterizing irradiated materials, because it gives both Young’s modulus and hardness as a continuous function of depth. In this work, radiation had no effect on the elasticity of $Fe_{14}Cr$, as quantified by the Young’s modulus. However, radiation increased the near-surface hardness by as much as 25%, with the greatest change occurring at a penetration depth of 100nm. Further work aims to explain this increased hardness in light of microstructural changes.

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KLA Corporation
One Technology Drive
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