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342 – Stainless Steel 304 Micro-Pillar Mechanical Response to Ion Irradiation and Helium Implantation Under Transmission Electron Microscopy Observation

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Austenitic stainless steels make up various components inside light water nuclear reactors due to their desirable corrosion resistance, adequate mechanical properties, and radiation tolerance. Components like the bolts that are used to hold core internals are made from 304 Stainless Steels (SS) are subjected to the extreme environment near a nuclear core which includes elevated temperatures up to 350 °C, mechanical stress, and neutron irradiation [1]. Additionally, helium originating from alpha decay and transmutation can become trapped in the material and alter its mechanical properties [2]. To better understand the role of irradiation damage and helium implantation on the mechanical response of 304SS, in situ micropillar compression tests were conducted. Other studies [3-5] perform some versions of hardness tests after irradiation and helium implantation and lose the ability to view the mechanical response of the irradiated material. In situ micropillar compression tests give the ability to view the evolution of the irradiated and helium implanted microstructure under stress under Transmission Electron Microscopy (TEM) observation. The use of micropillar compression also allows for the use of ions to simulate neutron irradiated with little ion implantation [6]. Micropillars were fabricated using focused ion beam techniques. To determine the geometry to use for micropillar compression testing, two kinds of pillars were created. One pillar geometry had a square cross section geometry with a width and thickness of 300 nm and a height of 500 nm (Thick Pillar). The other pillar was fabricated with a rectangular cross section geometry which was also had a width of 300 nm and a height of 500 nm but had a thickness of approximately 100 nm (Thin Pillar). The resultant stress-strain curves are evident in Figure 1. The thin pillar has many load drops compared to the thick pillar. The thin pillar was found to bend instead of compressed which can alter the results of the stress-strain curves. Although the thinner pillar is better for TEM imaging, it cannot provide reliable mechanical data. The large grain size of about 30-40 µm meant that each pillar was a single crystal and that groups of pillars had the same orientation.

In Situ Ion Irradiation under TEM Observation

Various experimental conditions were used to probe the mechanical properties of irradiated materials. Three pillars served as the control group and only underwent a 300 °C heat treatment for the duration of time the other pillars were irradiated at the same temperature. Two pillars were only irradiated with 1 MeV Krypton ions at 300 °C until a dose of 5 dpa was reached at the Intermediate Voltage Electron Microscope (IVEM) facility at Argonne National Laboratory. Finally, other pillars were preimplanted with 25 keV helium ions to a fluence of either 1×10^{17} He ion/cm2, 5×10^{17} He ion/cm2, or 1×10^{18} He ion/cm^2. The Kr ion irradiated only pillars showed the formation and disappearance of irradiation induced defects. The number density of defect clusters decreased while the size of the defect clusters increased with increasing dose. Before Kr irradiation, only the pillars implanted with 1×10^{18} He ion/cm^2 showed signs of cavities. After Kr irradiation, cavities could be seen in all pillars implanted with helium. The largest of cavities was seen in the pillars with the highest fluence of helium.

In Situ Micropillar Compression under TEM Observation

Pillar compression tests were performed using a PI 95 Picoindenter from Hysitron© which can accurately measure the force as a function of displacement. All pillar tests were conducted at a displacement rate of 7 nm/s. Load as a function of displacement curves were collected and converted to stress strain curves to extract mechanical properties. Due to the ambiguity of determining the yield stress from micropillar compression tests, the flow stress at 5% strain was used. The flow stress of the Kr irradiated only pillar was larger than the flow stress of the control pillars at 5% strain. All the pillars implanted with helium followed by Kr irradiation had larger flow stresses than the Kr irradiated only pillars. Moreover, the flow stress decreased with increasing amounts of helium. It should be noted that the orientation of the different groups were slightly different with the Kr irradiated only pillars having a compression axis about 11° from the [111] direction while the helium implanted pillars were about 21° from the [111] direction. To quantify the amount of hardening that could be coming from the helium bubbles, the Friedel-Kroupa-Hirsch (FHK) hardening model was used [7]. The model under predicted the amount of hardening that should be seen when compared to the Kr ion irradiated only pillars. Specifically, the model predicts a hardening of 23.1 GPa, 19.4 GPa, and 10.6 GPa for the 1×10^{17} He ion/cm^2, 5×10^{17} He ion/cm^2 , and 1×10^{18} He ion/cm^2 pillars, respectively. Possible explanations for the deviation include that helium might not just be located inside the bubbles but might also be inside the crystalline lattice. Furthermore, the 25 keV helium ions also lead to a considerable amount of damage in the first 150 nm of the pillars. These factors could have led to the additional hardening that the model does not predict.

Load drops in the stress strain curves were only evident in the pillars that were irradiated with Kr Ions, especially the pillars that were pre-implanted with helium. The presence of load drops in irradiated stress strain curves of micropillars has been found in other papers [8] and they are speculated to be associated with defect free channels. Snap shots of the 1×10^{17} He ion/cm^2 pillar over various strains with its accompanying stress strain curve is shown in Figure 2.

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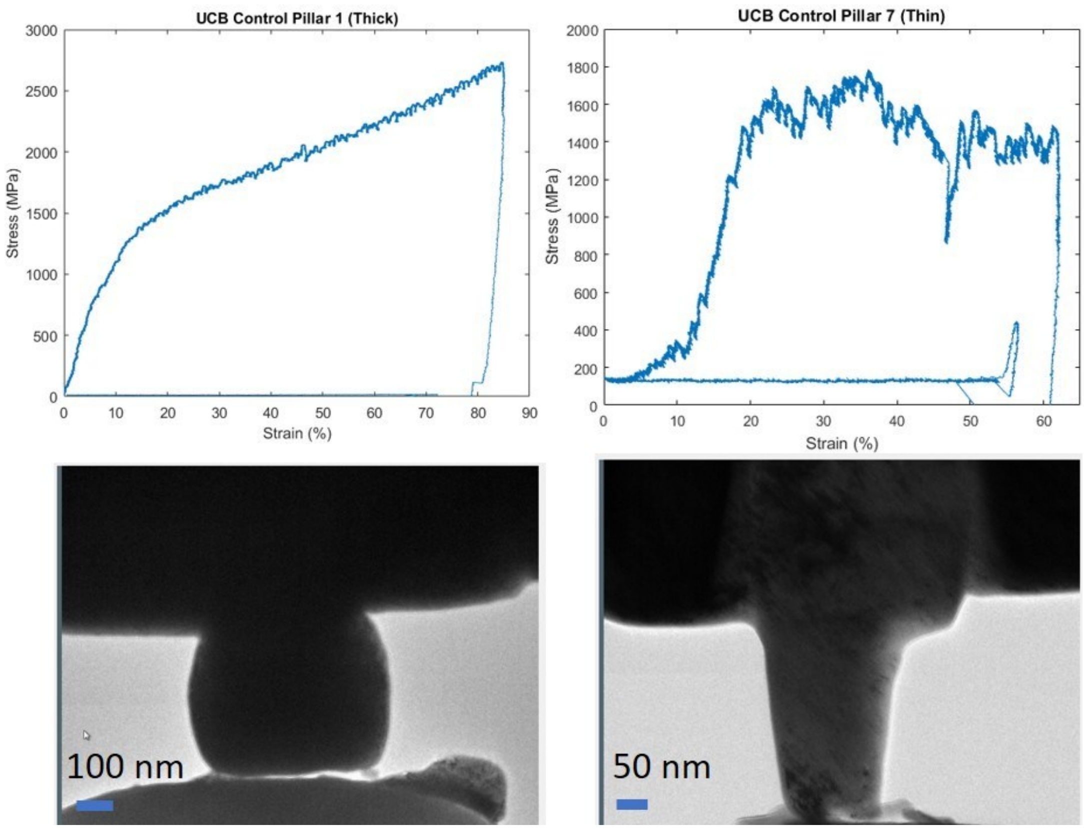

Figure 1. Figure 1: Stress-Strain Curves from a Thick (left) and Thin (Right) Pillar

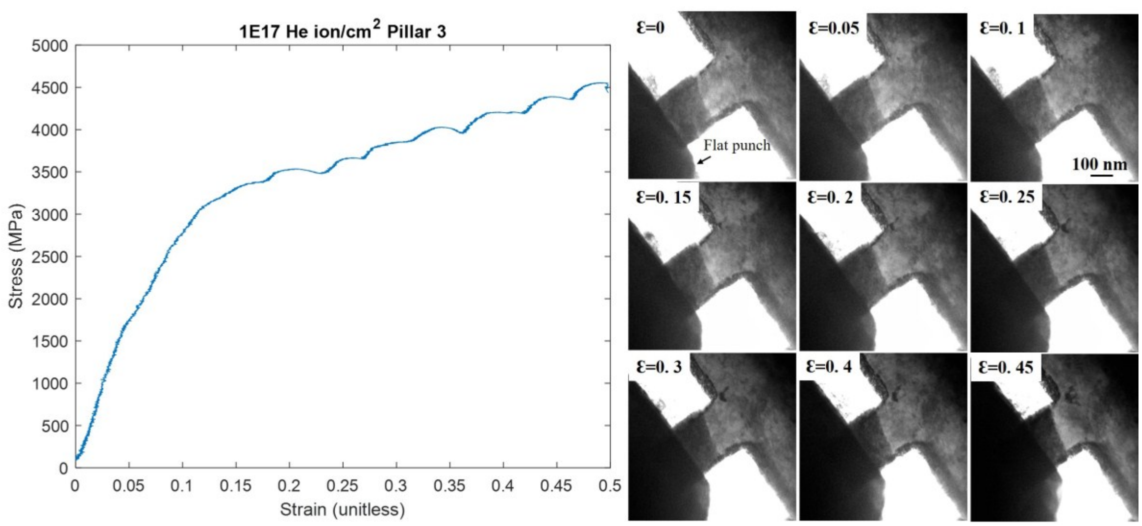

Figure 2. Figure 2: Snap shots of the 1×1017 He ion/cm2 pillar over Various Strains and the Stress Strain Curve

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