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In situ Measurements of Irradiation-Induced Creep on Amorphous Micropillars

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Introduction

Cladding materials used in nuclear power plants experience irradiation-induced creep (IIC), due to combined irradiation damage and stress. Reliable design of the power plant requires a detailed understanding of the IIC. *In situ* creep measurements using high energy ion beams provide an accelerated way of characterizing IIC. In such experiments, heavy ions in the MeV energy regime are used to accurately simulate the damage conditions in a nuclear reactor environment. However, the penetration depth of MeV heavy ions is only about 1 µm; therefore, micron-sized specimens are required for direct measurements.

IIC measurements on miniaturized specimens are challenging due to the high force resolution (~1 µN) and displacement resolution (~1 nm) requirements. We have overcome this challenge by developing a micropillar compression apparatus that combines a microfabricated silicon transducer with an attocube ECS3030 nanopositioner and an FPS3010 interferometric displacement sensor [1]. We used the apparatus to measure the IIC of amorphous $Cu_{56}Ti_{38}Ag_6$ micropillars. Measurements show that the creep rate is proportional to the applied stress. The irradiation induced fluidity of the sample was measured to be 2.1 dpa^{-1}GPa^{-1} (dpa: displacement per atom).

Setup Description

Fig. 1 shows a schematic of the measurement apparatus [1]. The micropillar is mounted on the nanopositioner and the laser spot of the displacement sensor is aligned with the center of the transducer. When the nanopositioner is moved, the micropillar specimen deflects the transducer. Once the required deflection (~1 µm) is reached, the nanopositioner keeps the micropillar stationary. The micropillar under compressive stress is then bombarded with 2.1 MeV Ne$^+$ ions. As a result, the micropillar experiences IIC, and its deformation corresponds to a decrease in the transducer deflection, which is monitored by the displacement sensor.

The apparatus has a force resolution of 0.2 µN using a transducer with a spring constant of 200 N/m. The deflection measurement can resolve displacements as small as about 1 nm. Thus for micropillars of 2 µm height, the strain resolution is 0.05%.

The silicon transducer is a doubly clamped beam with dimensions of $10 \times 80 \times 2000\ \mu m^3$, and was fabricated using standard microfabrication techniques [1]. A 40 nm thick Al layer was
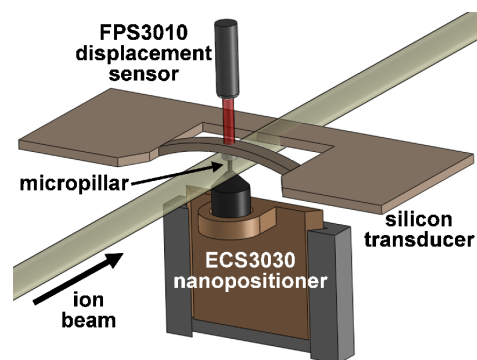

Figure 1: *Schematic of the measurement apparatus. Using the ECS3030, the micropillar is pushed against the transducer, resulting in its deflection. The micropillar is bombarded with 2.1 MeV Ne$^+$ ions, and its creep is measured through the change in the transducer deflection using an FPS3010 interferometer.*

sputtered on the sensor side of the transducer for high reflectivity. $Cu_{56}Ti_{38}Ag_6$ bulk samples were prepared by ball milling and micropillars of 1 µm diameter and 2 µm height were fabricated by focused ion beam. The amorphous structure of the sample was verified by X-ray diffraction analysis. Measurements were performed at room temperature in an irradiation chamber with vacuum level $< 1 \times 10^{-7}$ Torr. A Van de Graaff accelerator provided 2.1 MeV Ne$^+$ ions at an ion flux of ~1.7×10^{12} ions/(cm^{2}s).

Measurement Results

Difference between the nanopositioner position reading and the displacement sensor reading provides information on the deformation of the micropillar. Fig. 2 shows the deformation of a $Cu_{56}Ti_{38}Ag_6$ micropillar under irradiation as a function of time [1]. The micropillar was loaded three times, with different transducer deflections, resulting in different stress levels. The results show that IIC rate is proportional to the applied stress, indicating Newtonian flow. This observation is consistent with previous measurements on other amorphous materials under irradiation [2].

Irradiation-induced fluidity of a material is defined as the inverse of the viscosity under irradiation normalized by the displacement damage rate. For the $Cu_{56}Ti_{38}Ag_6$ specimens, the fluidity was measured to be 2.1 dpa^{-1}GPa^{-1} [1]. This value is close to results obtained using molecular dynamics simulations [3].

Summary

We have demonstrated *in situ* measurements of irradiation-induced creep (IIC) through micropillar compression. The attocube ECS3030 nanopositioner provided accurate control of micropillar position and transducer deflection, whereas the attocube FPS3010 displacement sensor has measured the deformation of the micropillar with excellent accuracy and precision. The apparatus provides a new and effective approach to IIC measurements for the accelerated evaluation of promising materials for future nuclear power plant applications.

References

[1] S. Özerinç, R. S. Averback, W. P. King, J. Nucl. Mat. **451**, 104 (2014).
[2] E. Snoeks, T. Weber, A. Cacciato, A. Polman, J. Appl. Phys. **78**, 4723 (1995).
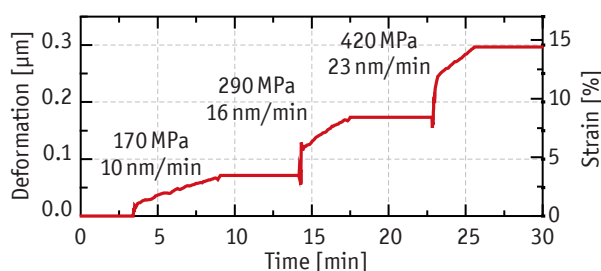[3] S. G. Mayr, Y. Ashkenazy, K. Albe, R. S. Averback, Phys. Rev. Lett. **90**, 055505 (2003).

Figure 2: *Deformation of the micropillar as a function of time under irradiation. Micropillar stresses and corresponding creep rates are indicated for each loading.*

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