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In situ creep measurements on micropillar samples during heavy ion irradiation

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ABSTRACT

We report on the development of an *in situ* micropillar compression apparatus capable of measuring creep under heavy ion beam irradiation. The apparatus has a force resolution of 1 μN and a displacement resolution of 1 nm. The experimental setup consists of a nanopositioner, a laser displacement sensor, and a microfabricated doubly clamped silicon-beam transducer. The system was tested by measuring the creep rate of amorphous $Cu_{56}Ti_{38}Ag_6$ micropillars as a function of applied stress during room temperature irradiation with 2.1 MeV Ne^+. Measured values of the irradiation induced fluidity are in the range 0.5–3 dpa^{-1} GPa^{-1}, and in good agreement with values obtained by stress relaxation experiments on other metallic glasses, and with predictions of molecular dynamics simulations. The *in situ* apparatus provides a practical approach for accelerated evaluation of irradiation induced creep in promising nuclear materials.

1. Introduction

Generation IV nuclear power plants are expected to operate at temperatures as high as 1000 °C and with displacement damage reaching 150 dpa (displacements per atom) in the cladding materials [1,2]. Maintenance of the dimensional stability of the cladding and the surrounding components under these extreme conditions will require advanced structural materials that are both resistant to void swelling and irradiation induced creep (IIC) [3]. A promising means for reducing void swelling employs materials containing a high density of neutral sinks such as nano-precipitates [4], grain boundaries [5], or nanolayered structures [6]. While some work has begun to examine the creep resistance in these materials under irradiation, [7–9], these studies have been very limited and a systematic understanding of IIC in advanced alloys is still lacking. A key need for faster progress in this area is the development of a robust testing procedure that can reliably and quickly measure creep in a wide range of materials under reactor-like conditions. The current work describes a novel capability to perform *in situ* creep experiments on ion-irradiated bulk materials.

IIC has been investigated in the past through both in-reactor tests and accelerated ion beam experiments. In-reactor experiments enable the direct evaluation of neutron irradiation effects on bulk materials, typically employing expansion of pressurized tubes [10–17], stress relaxation of elastically bent beams [18–21] or loaded helical springs [22–24]. Such in-reactor tests, however, generally suffer from high costs, low damage rates, and difficulties associated with post-irradiation examination of radioactive materials. High energy ion irradiations provide an alternative and accelerated approach for simulating the neutron induced displacement damage in nuclear power plants [25]. Simulation by ion irradiation, however, has its shortcomings as well: (i) the difference in primary recoil spectra between ions and neutrons, (ii) the difficulty in correcting for the accelerated kinetics, and (iii) the small volume of sample that can be irradiated. By employing heavy ion irradiations, as opposed to e.g., protons, the first of these issues can be largely eliminated [26], and indeed, such experiments have long been utilized to study microstructural evolutions in irradiated alloys and ceramics. Measurements of mechanical behavior on ion irradiated samples pose more significant problems, since large samples are usually required for reliable analysis. Extensive efforts are presently being focused on developing new protocols for analyzing mechanical properties from tests on miniaturized samples [27]. Irradiation induced creep (IIC) measurements are particularly challenging owing to the difficulty of measuring strain *in situ* on micron-sized specimens.

Experimental methods currently employed to evaluate IIC during ion irradiation usually involve either stress relaxation or uniaxial creep measurements. Stress relaxation is attractive owing to the ease of specimen preparation and accurate measurement of small

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strains by wafer (or beam) bending methods [28,29]. These experiments, however, are limited to very low strains, and they are usually subject to highly inhomogeneous damage [30–35]. Uniaxial creep tests using ion beam irradiation have the advantages of active control of specimen stress and straightforward data interpretation, but these measurements generally require larger specimens, >50 μm [9,36–48]. For this reason, such studies have mostly been performed using high energy protons [9,37,42,45,48] or other light ions [36,38,39,41,43,44]. The recoil spectra associated with light ion irradiation, however, are shifted to low energies, in comparison to fast neutrons, and displacement rates are very low. Damage levels are typically limited to under ≈1 dpa in these experiments, even after many hours of irradiation. A few experiments have utilized GeV heavy ion irradiations [46,47], as they provide more uniform damage and high damage rates; however, the damage in this case stems mainly from the pronounced electronic stopping of swift ions, not from nuclear collisions, and hence they probe a different physics [49].

In contrast to light ions, MeV heavy ions produce very high damage rates, and the hard-sphere like interactions between ions and target atoms yield recoil spectra very comparable to fast neutrons [49]. MeV heavy ions, however, have short penetration depths in solids, and thus they have been used mostly with stress relaxation measurements [30–34,50,51]. Recently, *in situ* bulge tests have been successfully employed with heavy ions, with plastic strains over 5% being achieved [52,53]. These experiments, however, have so far required thin film specimens grown by physical vapor deposition, and therefore they are not conducive to testing bulk materials with complex microstructures, such as nano-ODS ferritic/martensitic steels and other technologically significant materials. The method described here overcomes this difficulty, and many of the others described above, by using *in situ* compression tests on micropillar specimens that can be cut from any bulk material. We apply our method to the test case of amorphous $Cu_{56}Ti_{38}Ag_6$. Creep in amorphous materials is particularly interesting since it is typically orders of magnitude greater than in crystalline materials, although the mechanisms of creep [54,55] remain uncertain. As part of the analysis of our method, we apply finite element method to evaluate the errors associated with ion beam heating and the inherent inhomogeneous displacement damage with ion irradiation.

2. Experimental

Fig. 1(a) shows a schematic of the experiment. The measurement apparatus consists of an Attocube ECS3030 closed-loop nanopositioner with 1 nm positioning accuracy, an Attocube FPS1010 interferometric laser displacement sensor, also with 1 nm reproducibility, and a microfabricated doubly clamped silicon beam (the transducer). Micropositioning stages (not shown) facilitate the alignment between the micropillar, transducer center, and laser spot. The nanopositioner controls the position of the micropillar, and the laser sensor measures the height of the transducer as illustrated in Fig. 1(b). The micropillar is moved by the nanopositioner, resulting in a deflection of the transducer, and a uniaxial compressive stress in the micropillar (Fig. 1(c)). During the test, the nanopositioner holds the base of the micropillar stationary after loading; this results in a nearly constant stress in the micropillar, even for large strains. Deformation of the micropillar is obtained from the change in the deflection of the center of the transducer, measured by the laser sensor (Fig. 1(d)). The stress in the micropillar is determined from the measured transducer deflection and the transducer spring constant.

Fig. 2 shows the fabrication steps of the single crystal silicon transducer. Doubly clamped beams, 2 mm in length, 80 μm in

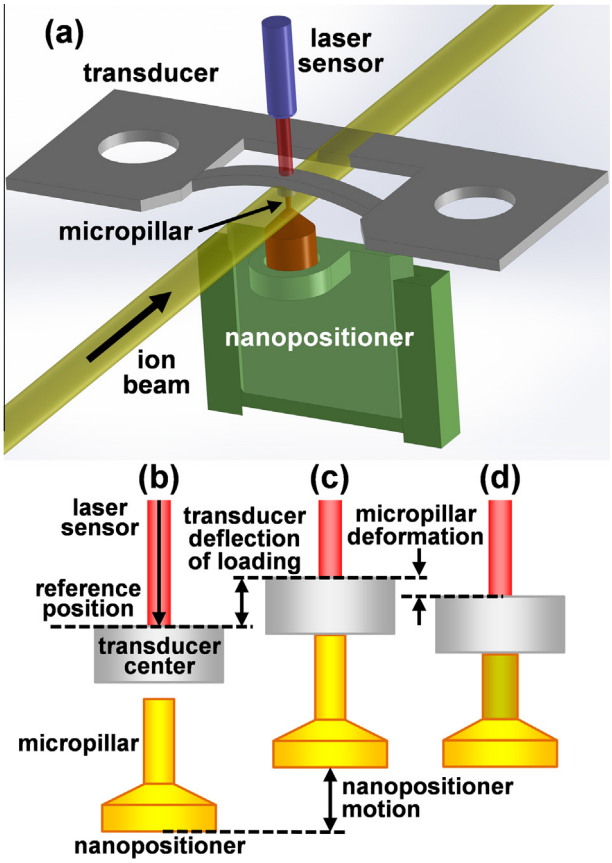

Fig. 1. (a) Schematic view of the main components of the experiment. (b) Micropillar is disengaged from the transducer. (c) Transducer is deflected and the micropillar is under load. (d) Micropillar deforms, resulting transducer deflection to change.

effective width, and 10 μm in thickness were microfabricated from silicon-on-insulator wafers with a 30 μm device layer, a 2 μm silicon dioxide layer, and a 400 μm handle layer. A first photolithography step followed by deep-reactive ion etching (DRIE) for 10 μm defined the silicon beam structure (Fig. 2(a)). A second step following the same procedure formed the 20 μm diameter, 20 μm tall cylindrical punch at the center of the transducer (Fig. 2(b)). This punch facilitates the alignment of the micropillar with the transducer center. Backside patterning and subsequent DRIE for 400 μm (Fig. 2(c)), followed by hydrofluoric acid etching of the exposed silicon dioxide layer released the transducers (Fig. 2(d)). The final step was the sputter-deposition of a 40 nm thick Al layer on the laser side of the transducer for high reflectivity.

Fig. 3(a) shows an SEM image of a fabricated transducer. The inset shows a close-up view of the cylindrical punch region. The double-leg design of the transducer improves the torsional stiffness and facilitates the alignment of the laser spot with the center of the transducer. An Asylum MFP-3D atomic force microscope was utilized to measure the transducer spring constants (≈200 N/m) using a calibrated commercial microcantilever.

Amorphous $Cu_{56}Ti_{38}Ag_6$ specimens were prepared by high-energy ball milling of elemental Cu, Ti, and Ag powders using a SPEX 8000 mill in a purified argon glove box. Compaction of the powders at 0.85 GPa at 150 °C produced cylinders of 1 cm diameter and ≈1 cm height. Electrical discharge machining (EDM) was used to cut out 1.6 mm diameter pins from the cylinder. This was followed by mechanical polishing to reduce the diameter of the pins to 20 μm. The final shape of the specimen, 1 μm diameter and 2 μm height, was then obtained using an FEI Helios Nanolab 600i fo-