Irradiation-Induced Creep in Metallic Nanolaminates Characterized by *Insitu* TEM Pillar Nanocompression

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Abstract

This work reports on irradiation-induced creep (IIC) measured on nanolaminate (Cu-W and Ni-Ag) and nanocrystalline alloys (Cu-W) at room temperature using a combination of heavy ion irradiation and nanopillar compression performed concurrently *insitu* in a transmission electron microscope. Appreciable IIC is observed in multilayers with 50 nm layer thicknesses at high stress, $\approx \frac{1}{2}$ the yield strength, but not in multilayers with only 5 nm layer thicknesses.

Background

Non-conservative fluxes of irradiation-induced point defects to non-recombinative sink sites underpins many of the mechanisms for irradiation-induced degradation of materials.[1-3] Mitigation strategies focus, accordingly, on introducing a high density of recombinative sink sites that are either kinetically stable under operating conditions or exist in a non-equilibrium steady-state under irradiation fluxes.[4, 5] A common approach for implementing this strategy exploits the high density of interfaces associated with nanostructured materials.[6, 7] Examples include oxide dispersion strengthened (ODS) alloys, self-organized nanostructured alloys, and nanolaminates.[8-11]. Nanostructured alloys have indeed been shown to suppress irradiation induced defect evolution and promote defect recombination, and in many instances they also provide exceptional mechanical properties, making them attractive for application in extreme environments. Conversely, nanostructured materials are susceptible to thermal creep, owing to grain boundary and interfacial creep processes.[12-14] Limited work, however, has focused on creep in nanostructured materials, particularly in an irradiation environment. Mechanisms considered for irradiation-induced creep (IIC) have mostly involved dislocation interactions with irradiation induced point defects.[15, 16] Since the dislocation content is inherently small in nanocrystalline metals, IIC has not been considered to be a significant issue for these materials. Recent IIC investigations on nanostructured Cu-W alloys, however, demonstrate that point defect fluxes to grain boundaries can drive creep deformation.[17] By this mechanism, fluxes of vacancies and interstitials are accommodated at the grain boundaries via atomic rearrangements similar to those in shear transformation zones.[18] The applied stress biases the direction of this atomic relaxation process, and over many such events, significant deformation takes place. In Cu-W thin films measured via *insitu* bulge testing, this mechanism was found to be active at stresses as low as

1MPa and temperatures as low as room temperature, as interstitial and vacancy mobility are still appreciable in Cu at this temperature.[17, 19] Radiation-induced stress relaxation measurements have similarly indicated that creep mechanisms are operative in several nanograin thin-film materials.[20] For grain sizes <9 nm, Mayret *al.*[20] invoked an irradiation-induced coarsening mechanism to explain irradiation-induced stress relaxation. When the diameter of the thermal spike in a collision cascade is comparable to the grain size, lattice sites are not necessarily conserved and a creep mechanism similar to that in amorphous materials is suggested to be active. This mechanism, notably, does not require point defect migration. Irradiation induced stress relaxation was also observed in Pt films with an initial grain size of 9-12 nm over the range of 25 to 200°C.[21] In this case, the large epitaxial stresses were thought to produce anisotropy in the point defect diffusivities by affecting the energy barrier for diffusional jumps, thereby biasing the fluxes of vacancies and interstitials.[21] These various experiments indeed demonstrate that IIC is active in nanograin polycrystals at low temperatures, and that the mechanisms are likely to differ significantly from those active in coarser grained materials. The range of possible creep mechanisms, however, remains to be fully explored in different regimes of grain size, temperature, particle flux, and stress.

Measuring IIC presents substantial experimental challenges.[22, 23] Experiments performed in reactors are enormously expensive, and they require excessively long experimental times to reach doses on the order of a DPA (displacement per atom). Moreover, in-reactor experiments typically offer little opportunity to systematically vary the applied stresses and temperature. These limitations can be overcome using ion irradiation, but the penetration depths of ions are quite small for mechanical testing. Light ions, e.g., protons and He, have reasonably large penetration depths (microns), but they do not simulate the damage profile of fission neutrons, and owing to their small damage cross sections, they also require long exposures to reach damage levels beyond 1 DPA.[23] Such techniques, nevertheless, have been applied to characterize irradiation creep at low doses in ODS alloys with sample size on the order of 100 μm . [24, 25] Heavy ions, on the other hand, simulate the damage profile of neutrons quite well, and they provide much higher DPA rates, but heavy ions provide uniform damage profiles only over very small length scales, 10's to 100's of nanometers.[26] This requires that sample sizes must be commensurate with the depth of relatively uniform damage. Mechanically testing samples smaller than a few hundred nanometers in diameter during ion beam irradiation introduces a number of experimental difficulties associated with beam heating, mechanical alignments, and overall achievement of precision testing. We refer the readers to reference [23] for a quantitative discussion of the practical experimental issues. In that work, creep was measured in metallic and oxide glasses for which the creep rates are quite high.[22, 23] The lower creep rates associated with polycrystalline materials are far more challenging. In this work, we use high-energy, heavy-ion irradiation combined with *insitu* TEM nanocompression to study IIC. The *insitu* characterization ensures that the samples and loading apparatus are well aligned and that deformation modes are consistent

with expectations. Such *in situ* TEM observation, also, enables direct verification of the strain values measured by the instrumented nanoindentation holder. Here we demonstrate the utility of this experimental approach for acquiring IIC rates from nanostructured alloys.

Immiscible alloy nanolaminates have been intensively studied for possible application in extreme particle irradiation environments because they are thermally stable and promote defect recombination at their interfaces.[11, 27, 28] The creep response of such materials in radiation environments, however, is largely unknown. Specifically, it is unclear whether the accommodation mechanisms for point defects at heterophase interfaces in these materials are similar to those at grain boundaries. If nanolaminate materials are anticipated to be load bearing, then understanding their irradiation creep response will be vital. This work compares room temperature irradiation creep in Cu-W nanolaminates and Ni-Ag nanolaminates, as well as to dilute Cu-W alloys. Interstitials are highly mobile in all of these metals. Vacancies, though, are immobile in W and Ni at room temperature, quite sluggish in Cu, but reasonably mobile in Ag (vacancy diffusivity in Ag, $\approx 10 \text{ nm}^2 \text{ s}^{-1}$, is about a factor of 5 higher than in Cu at 300 K).[29] Comparing irradiation creep in the two systems and two morphologies thus enables us to assess the underlying creep mechanisms.

Experimental Methods

Multilayer nanolaminate samples of Cu-W and Ni-Ag were grown on pre-tilted Si pillars following a method described in detail elsewhere.[30] Pre-tilt angles near 45° were used such that if defect fluxes to the interface were to drive creep, the maximum resolved shear stress would be applied at these interfaces. Cu(99.99% Lesker) - W(99.95% Lesker) multilayers with individual layer thicknesses, h , of nominally 50 nm and 5 nm were grown via magnetron sputtering in Ar in a system with a base pressure of $\approx 10^{-8}$ torr. Ag (99.99% Lesker) - Ni (99.99% Lesker) multilayers with $h = 50 \text{ nm}$ and Cu_{99}W_1 alloy films were also grown by magnetron sputtering. Pillars for nanocompression were fashioned into their final geometry using annular milling in a focused ion beam (FIB, FEI DB238). The 250 to 550 nm pillar diameters far exceed the grain size (around 50 nm) of the Cu and W and therefore surfaces should not be a significant defect sink. The literature suggests that the bulk yield strengths of Cu-W and Ni-Ag multilayers of comparable thickness are $\approx 5.5 \text{ GPa}$ and $\approx 1.17 \text{ GPa}$, respectively.[31, 32] Their interfacial shear strengths are reported to be $\approx 0.55 \text{ GPa}$ and 0.53 GPa , respectively.[33] *In situ* nanocompression tests were performed using a Hysitron PI-95 Picoindenter in a 200 kV JEOL 2100 I³TEM.[34] Values of strain reported by the instrumentation were verified through imaging. Results obtained under a constant loading rate were much more reliable than those under constant load, as the latter was more prone to sample drift. The load control experiments were performed with a loading rate of 25 MPa s^{-1} up to $\sim 750 \text{ MPa}$, after which the loading rate was varied between 0.63 and 33 MPa s^{-1} . The attached 6 MV EN Tandem Van de Graaff-Pelletron accelerator was used as the source of 3 MeV Cu^{3+} ; it delivered between 30 and 170 nA within a spot size of approximately 4 mm in diameter, resulting in average damage rates in Cu

between 2×10^{-3} to 1×10^{-2} DPA s^{-1} . Ni-Ag samples were irradiated at 30 nA producing a damage rate in Ag of 2×10^{-3} DPA s^{-1} .

Results

We first performed stress-strain measurements on 50 nm Cu-W multilayers in the absence of irradiation in load controlled and displacement controlled modes and after irradiation to 100 DPA in load controlled mode. Figure 1a shows the response of these samples. Interfacial shearing introduces a discontinuity in the curve, indicated by the red arrow, and is the primary source of plastic deformation. Pre-irradiation clearly enhances the interfacial shear strength of Cu-W, similar to previous reports for Cu-Nb.[30] These load control tests were performed at $0.63 \text{ MPa } s^{-1}$ and the noise in the curve is associated primarily with drift in the instrumentation over the time scale of the experiment (10^3 s). The *insitu* imaging is thus particularly valuable for distinguishing between noise associated with long loading times and actual strain in the sample and observing critical events such as interfacial sliding. Low rates of loading ($0.63 \text{ MPa } s^{-1}$) were used in this experiment in order to be commensurate with the lowest loading rates utilized for the IIC experiments to follow. The displacement controlled experiment was performed at $1 \text{ nm } s^{-1}$, and it yields similar results. Figure 1b depicts the stress-strain curves of 50 nm Cu-W multilayers, first irradiated *insitu* at approximately 1.4×10^{-2} DPA s^{-1} , and then tested under load control at $0.63 \text{ MPa } s^{-1}$. The pristine sample and the sample pre-irradiated to 90 DPA exhibit similar behavior. The main differences between these curves regard the discontinuity introduced by an interfacial shearing event in the pristine samples, which as described above has a lower interfacial shear strength. Videos of these tests are available in *supplemental videos S1-S2*.

Comparison of the stress-strain results for the conventionally loaded samples just described to those for samples tested *insitu*, during irradiation, shows a clear difference, as seen in Fig. 2. The *insitu* irradiated samples undergo significant bulk plastic strain, which is absent in the conventionally tested samples. This is clear evidence for creep in these Cu-W multilayers during room temperature irradiation. Pre-irradiation, in contrast, appears to have limited impact on IIC, at least under the conditions tested here. Both *insitu* and post-testing *exsitu* imaging show that deformation occurs primarily in the Cu layers. Tests were also performed using loading rates of 1.5, 6.25 and $33 \text{ MPa } s^{-1}$ under different levels of pre-irradiation. With the exception of interfacial shear events that occurred at different levels of stress in each sample, the stress-strain curves in the various cases are quite similar in shape (see figure 2). When plotted on a log-log scale the time dependent strain response of each sample is non-linear, but they all exhibit stress exponents of $n < 2$. For the load controlled samples, the loading rate affects the irradiation creep strain-rate, which averaged between 800 and 1500 MPa and varies approximately linearly; 1.8×10^{-2} , 2.8×10^{-2} , 1.6×10^{-1} , 3.5×10^{-1} DPA $^{-1}$ for loading rates of 0.63, 1.5, 6.3, and $33 \text{ MPa } s^{-1}$, respectively. This dependence on loading rate suggests that under the dynamic loading conditions employed, the samples do not reach steady-state and are in a transient creep regime. This is supported by the fact that steady-state creep rates at constant load, discussed below, are much lower in all cases.

To explore steady-state creep, we tested samples under conditions of constant load. Figure 3 shows the creep response of a 50 nm multilayer sample under a constant load of 600 and 800 MPa, which produces irradiation creep rates of 3.5×10^{-3} and $4.6 \times 10^{-2} \pm 0.1 \times 10^{-2} \text{ DPA}^{-1}$, respectively. These data were obtained by measuring the distance between reference points on the TEM images. The two data from the 800 MPa tests are quite reproducible, aside from the transient in one sample associated with shearing of the interface. The tested samples can be seen in the before and after images in the figures 4 and 5. The evolution of the material during creep apparently facilitates the interfacial sliding event that occurs later in the constant load experiment. However, it is clear that the steady-state creep response results primarily from deformation in the bulk, rather than shearing at the heterophase interfaces. This is apparent from measuring the change in relative thicknesses of the Cu and W layers before and after creep. Tai *et al.* [17] performed measurements of the dependence of IIC on applied stress in the low-stress regime of 1 to 10 MPa at 473 K and reported a stress exponent of unity. Linear extrapolation of their results at 473 K to 600 MPa (see figure 6), however, results in a normalized creep rate that is a factor of ~ 3 lower than the present results on the Cu-W nanolaminate. Further extrapolation of their results to room temperature using their reported temperature dependence, moreover, yields a creep rate a factor of ~ 10 lower than in the nanolaminate. For further comparison with the work of Tai *et al.*, [17] we tested Cu_{99}W_1 alloy nanopillar samples (see figure 6) at high stresses, 400 and 600 MPa, and at these stresses we measured creep rates slightly *larger* than those measured in the multilayers. It remains unclear why the bulge test measurements and those in the present experiments produce differing creep compliances, $\frac{\dot{\epsilon}}{\sigma \dot{D}}$, where $\dot{D}$ =dpa rate, $\dot{\epsilon}$ =strain rate, and σ =stress. However, it is perhaps not too surprising that the mechanism for creep will change as the applied stress is increased a factor of ~ 100 and approaches the yield strength.

We also compared the response of the Cu-W multilayers to that of the Ni-Ag multilayers (See figure 7). The Ni-Ag multilayer exhibits an even higher creep compliance, a factor of ~ 10 larger than in Cu-W. Notably, this creep compliance is still a factor of 2 lower than those reported from thin film stress relaxation experiments on $\text{Ag}_{50}\text{Co}_{50}$ alloys with domain sizes $< 9 \text{ nm}$. [20] In the $\text{Ag}_{50}\text{Co}_{50}$ alloys the initial (epitaxial) stresses were comparable to those applied in the present experiments. Overall, these results clearly indicate that IIC in multilayer nanolaminates can indeed be quite high, approaching values associated with amorphous samples, but with the exact value depending on the compositions of the component layers.

To examine the effect of length scale on the creep mechanism active in our samples, we tested 5 nm-thick Cu-W multilayer samples. Due to their higher strength, these samples could be tested up to loads of $\sim 4 \text{ GPa}$, at loading rates between 5 and 25 MPa s^{-1} . Figure 8 shows example stress-strain results for samples tested with the ion beam on and the ion beam off. In this case, the irradiation has little effect, indicating that IIC is negligible in these samples. The primary mode of plastic deformation in

all of these samples at stresses below ≈ 3 GPa is interfacial shearing. Notably, the measured interfacial shear strength is larger in the 5 nm multilayers than in the 50 nm multilayers, even absent irradiation. The absence of appreciable irradiation creep deformation in the 5 nm Cu-W multilayers is somewhat surprising in view of experiments by Mayret *et al.*[20] cited above, which showed stress relaxation in irradiated two-phase Co-Ag specimens with domain sizes of ≈ 9 nm. They also seem at odds with reference,[18] which predicted that ICC should increase inversely with grain size. The results thus suggest an important difference in the mechanisms operating in three dimensional nanostructures and two-dimensional nanolaminates. The work also suggests that an appropriate nanolaminate design could be exploited to dramatically suppress irradiation creep.

Discussion

Three mechanisms have been proposed previously to rationalize IIC in nanocrystalline materials: they relate to; i) coarsening when the grain size is comparable to the thermal spike radius,[20] ii) stress biased relaxation of excess grain boundary volume introduced by non-conservative point defect fluxes,[18] iii) anisotropic point defect fluxes resulting from stress induced anisotropic diffusivities.[21] We exclude mechanism (i) as the Cu grain size in our samples is around 50 nm, which is already large, and it does not appear to increase during *in situ* TEM irradiation observations. Presumably the grain size is constrained by the nanolaminate structure. Mechanism (ii) should operate in the high stress regime, but since the steady-state creep rates in Cu-W measured at 600 MPa are 10 times larger than those extrapolated from 10 MPa measured by Tai *et al.*,[17] we conclude that it cannot be the dominant creep mechanism in the high stress, low-temperature regime. Mechanism (iii) operates specifically in the regimes of low temperature and high stress, and therefore it should be relevant here. We note that this mechanism could also explain why IIC is negligible in the 5 nm nanolaminate sample. The model relies on anisotropic diffusion: in compression vacancies preferentially migrate to Cu/W interfaces and interstitials to the Cu grain boundaries normal to the hetero-interface. As the layer thicknesses become small, relative to the lateral grain size, more and more point defects, including interstitials, will annihilate at the hetero-interface, owing to its proximity. A high aspect ratio will thus reduce the net point defect fluxes to boundaries and hence the creep rate. Assuming that FCC Cu, Ni, and Ag exhibit similar susceptibility to stress induced anisotropy in diffusivity as calculated for FCC Pt, we predict an upper bound for the IIC compliance to be on the order of 0.4 dpa^{-1} . In Cu the production efficiency of freely migrating defects has been measured to be in the range of 1-5%.[28, 35] Thus the mechanism proposed by Chan *et al.*,[21] should enable a maximum creep compliance on the order of $4 \times 10^{-3} \text{ dpa}^{-1}$ to $2 \times 10^{-2} \text{ dpa}^{-1}$. This possibly explains the results in the Cu based systems, but it significantly underestimates the ICC compliance observed in Ni-Ag.

Irradiation induced defects will also transport as clusters and dislocation loops. Stress-induced preferred nucleation (SIPN) of loops occurs when vacancy and interstitial loops orient preferentially to relax the applied stress during loop nucleation. SIPN has been invoked as a potential IIC mechanism in coarse grained

materials and the phenomenon has been demonstrated to occur experimentally.[36, 37] However, the mechanism underestimates creep in coarse grained material and will only produce a transient response absent migration to neutral sinks. In nanograined samples, loops nucleate close to interface sinks, where image forces, elastic interactions, and/or random walk thermal diffusion can drive loops to annihilate at interfaces.[38, 39] Nanograined materials could then exhibit steady-state IIC by a SIPN mechanism. This mechanism could also explain the suppression of creep in 5 nm thick multilayers, where both the average thermal spike radius and layer thickness are comparable. In this case, most defects would recombine at the interfaces inhibiting loop nucleation. Constitutive equations for SIPN have been derived assuming Boltzmann statistics, but the validity of this approach has been questioned,[36, 37] making quantitative analysis of a SIPN based mechanism in our system beyond the scope of this work. Nevertheless, a combination of the mechanism proposed by Chan *et al.*, [21] which accounts for IIC from freely migrating defects, and the SIPN mechanism that captures contributions to IIC from loops could account for the large creep compliance in Ni-Ag multilayers.

Lastly, we cannot discount altogether the possibility that dislocation plasticity is responsible for the observed trends. Such a mechanism would require that irradiation facilitates dislocation nucleation and/or annihilation at interfaces. Molecular dynamics simulations of Cu grain boundaries, for example, indicate that the accumulation of excess free volume at the grain boundaries enhances dislocation emission.[40] Thermal spikes intersecting a grain boundary could then possibly give rise to such a situation, but presently we are not aware of any direct evidence for such behavior. We would also expect that this mechanism would become ineffective in samples with larger grain sizes as only a small fraction of thermal spikes would intersect the boundaries. This remains to be tested.

Conclusions

In situ TEM based irradiation creep measurements were utilized to demonstrate irradiation creep in nanolaminate alloys occurring at less than half of their yield strength. At constant load steady-state creep is observed, but under constant loading rates in excess of 0.63 MPa s⁻¹ transient effects dominate. Nanolaminate thickness dependent measurements, along with *in situ* imaging, suggest that irradiation creep plasticity occurs primarily in the bulk of the Cu films rather than at the Cu-W interface. Similar results were obtained for Ag-Ni nanolaminates, where both materials deformed by irradiation creep, but at much higher rates. We attribute the irradiation-induced creep observed at these high stresses and low temperatures to stress-induced anisotropic diffusion of point defects or to a SIPN-like mechanism.

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Figure 1: (a) Stress-strain curves associated with 50 nm Cu-W multilayers obtained with the ion beam off and tested under displacement controlled and load controlled modes. (b) Stress strain curves obtained during in-situ ion irradiation. In both data the discontinuities in strain are associated with interfacial sliding events highlighted by the red arrows.

Figure 2: Stress-strain irradiation creep results for samples loaded the constant load rates indicated, along with the same data replotted as a function of time and normalized time.

Figure 3: (a) *In situ* testing of samples at constant loads of 600 MPa and 800 MPa. Images are shown at the initial condition, half way through the test, and at the end of the experiment. (b) Strain response of samples tested under *in situ* irradiation under constant loads of 600 and 800 MPa. Note that the change in slope associated with the blue data points occurs at the onset of interfacial sliding.

Figure 4: As-grown Cu-W 50 nm multilayer before nanocompression testing.

Figure 5: Bright field TEM image, annular dark field image, and EDS map of a thin section of a pillar after testing. This is specifically the sample in Figure 2 tested at a constant loading rate of 0.63 MPa s^{-1} . The top portion of the multilayer sheared off and only a single Cu and W layer remain. Note that the sample was surrounded by a Cu thick film to aid in sample preparation.

Figure 6: Comparison of current results with reported irradiation creep data from the literature (solid lines) and extrapolated values (dotted lines). Literature data included and extrapolated from references [17, 20, 41]

Figure 7: Steady-state creep response measured from Ni-Ag 50 nm multilayer samples at different loads.

Figure 8: Stress-strain results for 5 nm multilayer samples loaded with the ion beam on (*insitu*) and off (*exsitu*). Loading rates for the in situ experiments were 5 MPa s^{-1} and 25 MPa s^{-1} .