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A novel on chip test method to characterize the creep behavior of metallic layers under heavy ion irradiation

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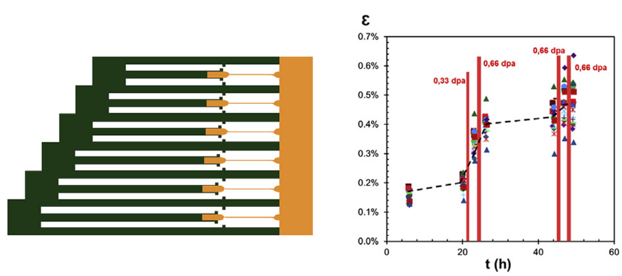
HIGHLIGHTS

- On chip method developed to test freestanding films under ion irradiation.
- Several tens of *in situ* irradiation creep tests performed simultaneously.
- Technique successfully validated on 200 and 500 nm thick Cu films.
- Fast creep rates measured in Cu films due to irradiation.

GRAPHICAL ABSTRACT



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ABSTRACT

An on chip test method has been developed to characterize the irradiation creep behavior of thin freestanding films under uniaxial tension. The method is based on the use of a long beam involving large internal stress protected from the irradiation flux that imposes a spring like deformation to a specimen beam. These elementary freestanding structures fabricated using a combination of deposition, lithography and release steps are multiplied with different dimensions in order to test different levels of stress and of initial plastic deformation. The method has been validated on 200 and 500 nm thick copper films under heavy copper ions irradiation. The irradiation creep rate is shown to be at least one order of magnitude larger than in the absence of irradiation.

1. Introduction

The creep deformation response under irradiation of metals and alloys is an important issue for structural materials used in nuclear applications. For instance, it can lead to dimensional instability of cladding tubes and to the decrease of the fastening torque of bolts. The creep behavior of stainless steels, zirconium alloys or Inconel 718 is usually assessed by conducting experiments on bulk material samples in test reactors under combined fast neutron flux and applied mechanical load [1,2]. These experiments require long duration of irradiation in order to reach significant doses, are expensive and lead to the activation of the material with all the associated complications in subsequent handling and characterization of the samples. Furthermore, these experiments do not permit *in situ* observations of the deformation mechanisms at the

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microscopic scale.

The use of ion irradiation and, in particular, heavy ion irradiation is a way to circumvent these problems. Indeed, the damage rate under heavy ion irradiation is several orders of magnitude higher than with neutrons [3,4], the materials are not activated and some facilities are compatible with *in situ* experiments such as at the JANNUS platform in France [5] coupled to Transmission Electron Microscopy (TEM). The main drawback is the limited penetration depth of the ions around a few tens of micrometers for light ions (typically 30 μm for 2 MeV protons in Zr) and usually not more than a few micrometers for heavy ions (typically 3 μm for 12 MeV Zr ions). Therefore, the mechanical tests must be adapted to probe the response of very small volumes of materials.

Nanoindentation is the first method of choice to characterize the mechanical response of micron or submicron sized volumes of irradiated material [6–11]. The limitations with nanoindentation are the difficulty to extract information about material failure as well as to identify complete viscoplastic hardening laws. Furthermore, it is still difficult to perform long term creep in an accurate way due to stability issues of the electronics or thermal drift effects. Several authors have conducted thermal creep experiments on various metals and alloys [12–14]. Nevertheless the extraction of the creep behavior from nanoindentation test remains tricky [15]. The complications to perform *in situ* nanoindentation measurements during exposure to the ion flux are also difficult to overcome. More recently, micro-pillar compression and micro-cantilever beam bending have been developed owing to the use of Focus Ion Beam (FIB) milling and of precise nanomechanical instrumentation [14,16–23].

However, these techniques have been essentially applied to assess the post-irradiation mechanical behavior of materials.

Very recently, Tai et al. [24] proposed a version of bulge test in which a pressure is applied, with a gas flow, on one side of a thin copper membrane while an ion beam irradiates the other side. The strain rate ($\dot{\varepsilon}$) in the copper film normalized by the damage rate (in dpa. s^{-1}) under ion irradiation is equal to 0.5×10^{-3} dpa^{-1} under a stress of a few MPa.

At a larger scale, irradiation creep experiments have also been designed using light ions such as protons or helium ions [25,26] or even high energy xenon ions [27]. The irradiation depth may reach several tenth micrometers, allowing to test nearly a bulk material. However, in that case the issue of the deposited power must be addressed with care, the experiment is often limited to low doses and foreign species may also be implanted in the material. Furthermore, it is difficult to adapt this type of experiment inside a TEM.

In this work, an original extension of an on-chip tensile test method [28] has been developed, relying on thin film specimens to characterize the mechanical response under irradiation by heavy ions (self-ion irradiation) with a specific focus on irradiation creep. The objective of this paper is to describe the test method and to assess it in an application on copper films. Even though the most challenging and novel aspect is the creep under irradiation, the method can be used to address irradiation hardening but for which nanoindentation or other methods are also available.

2. Experimental methods

2.1. Description of the elementary on-chip deformation method

The test specimens are produced in the following way starting with a silicon substrate: (i) a sacrificial layer of silica, intended to be etched away, is deposited first; (ii) second, an amorphous silicon nitride (Si_3N_4) film is deposited with large internal stress in tension and patterned by photolithography in the form of a very long beam; (iii) third, the specimen layer (here a copper film) is deposited and patterned with a dog bone shape. An overlap between the copper beam and the actuator beam ensures a sufficiently wide contact zone between the two beams [29]. The final geometry of the elementary test configuration is shown in Fig. 1. Small cursors are also introduced in the overlap region.

When the sacrificial layer is removed, the internal stress in the actuator relaxes leading to the contraction of the actuator and to the deformation of the specimen beam until force equilibrium is attained, see Fig. 1 a. The displacement, after reaching the equilibrium position, of the overlap between the copper beam and the actuator is measured at high magnification using a Field Emission Gun (FEG) Scanning Electron Microscope (SEM). The deformation (ε) and the applied stress (σ) of the specimen can be extracted based on the measured displacement (u) and mismatch strain in the actuator prior to release (ε_a^{mis}) with the following relationship:

$$\begin{cases} \varepsilon = \dfrac{u}{L} \\ \sigma = \dfrac{S_a}{S} E_a \left(-\dfrac{u}{L_a} - \varepsilon_a^{mis} \right) \end{cases} \quad (1)$$

where L and S are the specimen beam length and cross-section area respectively, L_a and S_a the actuator beam length and cross-section area respectively, E_a the actuator Young's modulus. The mismatch strain, also called misfit strain or transformation strain [30] represents the change of dimensions of the film independent of the stress such as due to thermal expansion, microstructure evolution or epitaxial strain. Due to the constraint imposed by the substrate, this mismatch strain is compensated by an equal and opposite elastic strain which leads to the internal stress.

The actuator acts thus also as a stress sensor. As a consequence, one structure gives one point in the specimen stress-strain response. The longer is the actuator beam, the larger is the deformation imposed to the specimen. By designing several test structures with various actuator lengths, several equilibrium points can be obtained and therefore several points of the stress-strain curve, see Fig. 1 b. The further stress relaxation of the specimen beams will still be following the linear behavior of the actuator, see grey points in Fig. 1 c. Hundreds to thousands of tests are performed at the same time “on a chip”. Further information on the method can be found in Refs. [29,31,32]. This method was, for instance, already used with success to assess the stress relaxation of palladium films at room temperature [33–35]. SEM micrographs showing one array of test structures and a zoom on one specimen are provided in Fig. 2.

In order to determine accurate stress and strain values in the test specimen, the results were analyzed by taking into account some correcting factors in the data reduction scheme as explained in Ref. [32]. In this approach, the system is decomposed into smaller sections to take into account the deformation of the irradiated parts and the elastic deformation of the junction regions. As explained in Refs. [33,36], the technique can be extended to creep analysis by letting the structure deform with time while measuring the displacements at different time intervals.

2.2. Extension to irradiation conditions

In the present study, the on-chip method is particularized to the following set of parameters and of configurations. Each tensile test structure is a member of a series of 40 structures. In each series, every specimen beam has the same length and the same width. Two arrays of structures with 6 μm-wide specimen beams are addressed: one with 50 μm-long and the other one with 100 μm-long specimen beams. The choice was made to irradiate the 50 μm-