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In-reactor uniaxial tensile testing of pure copper at a constant strain rate at 90 °C

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Abstract

Recently, annealed specimens of pure copper have been tensile tested in a fission reactor at a damage rate of 6×10^{-8} dpa/s with a constant strain rate of 1.3×10^{-7} s^{-1}. The specimen temperature during the test was about 90 °C. The stress response was continuously recorded as a function of irradiation time (i.e. displacement dose and strain). The experiment lasted for 308 h. During the dynamic in-reactor test, the specimen deformed and hardened homogeneously without showing any sign of yield drop and plastic instability. However, the specimen yielded a uniform elongation of only about 12%. The preliminary results are briefly described and discussed in the present note.

1. Introduction

The fact that neutron irradiation causes a substantial amount of hardening and alters significantly the deformation behaviour of irradiated metals and alloys at temperatures below the recovery stage V (i.e. <0.3–0.4T_m where T_m is the melting temperature in K) has been a subject of investigations for more than 40 years [1,2]. A critical review of this topic has been recently published by Singh et al. [3]. The post-irradiation deformation experiments have demonstrated consistently that neutron irradiation not only causes hardening but leads also to a drastic reduction in the uniform elongation (i.e. ductility) of the irradiated materials. This has been a matter of concern from the point of view of mechanical performance and lifetime of materials used in structural components of a fission or fusion reactor.

While recognizing the seriousness of this technological concern, it has to be acknowledged that the conclusions regarding the adverse effects of neutron irradiation on mechanical properties of metals and alloys are based exclusively on the results of post-irradiation deformation experiments. Traditionally, in these experiments the samples are first irradiated in unstressed condition to a certain displacement dose level. This means that during irradiation the material does not experience plastic deformation. Consequently, the accumulation of irradiation induced defects and their clusters (which are responsible for hardening and loss of ductility) takes place in the absence of deformation-induced mobile dislocations. The samples are then taken out of the reactor and mechanically tested but in the absence of irradiation, i.e. the specimens without experiencing the effect of continuous production of defects and their clusters.

The materials employed in the structural components of a fission or fusion reactor, on the other hand, will be exposed simultaneously to external stresses and irradiation induced defect population produced continuously during irradiation. Under these conditions, both the magnitude and the spatial distribution of defect accumulation and hence the deformation behaviour may be substantially different from that in the case of post-irradiation experiments (see later for further details). This raises a serious question as to whether or not the

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results and the conclusions of the post-irradiation deformation experiments can be taken to represent the behaviour of materials used in the structural components of a nuclear or thermonuclear reactor. In our view, this question can be answered properly and reliably only by determining experimentally the deformation behaviour of materials subjected simultaneously to plastic deformation and neutron irradiation. In an endeavour to address this problem, we have carried out in-reactor deformation experiments and have determined the dynamic stress–strain curves for pure copper subjected simultaneously to plastic deformation and neutron irradiation at ≃90 °C. In the following we describe the main experiment (Section 2) and present some preliminary results (Section 3). The implications of the present results are briefly discussed in Section 4 and the main conclusions are summarized in Section 5.

2. Material and experimental procedure

2.1. Material

The material used in the present investigation was thin (0.3 mm) sheet of oxygen-free high conductivity (OFHC) copper containing 10, 3, <1 ppm and <1 ppm of Ag, Si, Fe and Mg, respectively. The oxygen content of this copper was found to be 34 appm. Tensile samples of polycrystalline OFHC-copper (see Fig. 1 for size and geometry) were annealed at 550 °C for 2 h in a vacuum of 10^{-9} bar. The resulting grain size and dislocation density were about 30 μm and $\leqslant 10^{12}$ m^{-2}, respectively. These annealed specimens were used in the uniaxial tensile testing experiments performed inside the BR-2 reactor at Mol.

2.2. Test module and irradiation rig

In order to carry out these tensile experiments, a special test facility consisting of a pneumatic tensile test module and a servo-controlled pressure adjusting loop was designed and constructed.

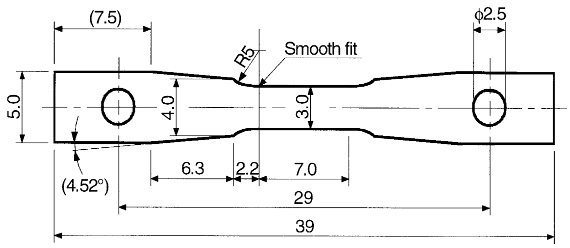


Fig. 1. Geometry and dimensions of the tensile specimen used in the in-reactor experiment. The specimen thickness was 0.3 mm. The dimensions are given in mm.

The basic principle of the tensile test module is based on the use of a pneumatic bellow to introduce stress and a linear variable differential transformer (LVDT) sensor to measure the resulting displacement produced in the tensile specimen. The schematic design of the tensile test module is shown in Fig. 2. The outside diameter of the module is 25 mm and the total length of the module together with the LVDT is 150 mm. A special calibration device was used to calibrate the applied gas pressure in the bellow with the actual load acting on the tensile specimen. A two-step calibration procedure was implemented where in the first step the characteristic stiffness of the bellow together with friction forces of the moving parts of the module were determined and in the second step the load induced on the tensile specimen by the applied gas pressure was measured directly by a load cell. The accuracy of the load calibration is approximately ±1% of the actual value of the stress resulting from the applied pressure causing the displacement in the specimen up to 1.3 mm. The calibration curves are shown in Fig. 3.

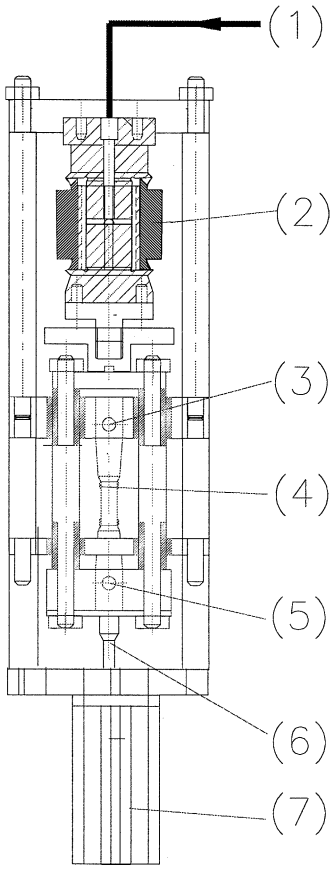


Fig. 2. Schematic design of the tensile test module: (1) gas line, (2) pneumatic loading unit, (3) firm specimen fixing point, (4) specimen, (5) movable specimen fixing point, (6) LVDT plunger and (7) LVDT holder. Details are given in [4].

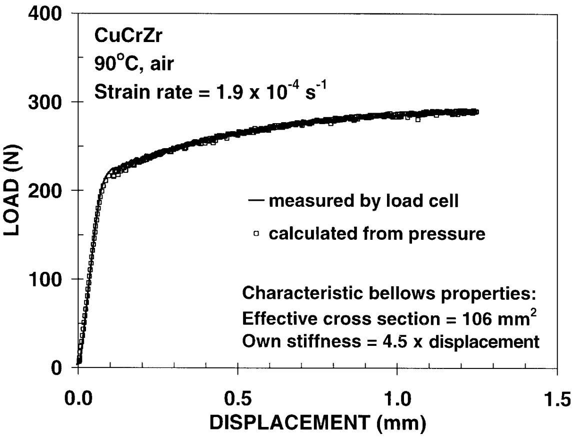


Fig. 3. Stress–strain curves measured with a load cell and calculated from the bellow pressure of the tensile test module. The strain was measured with a LVDT sensor [4].

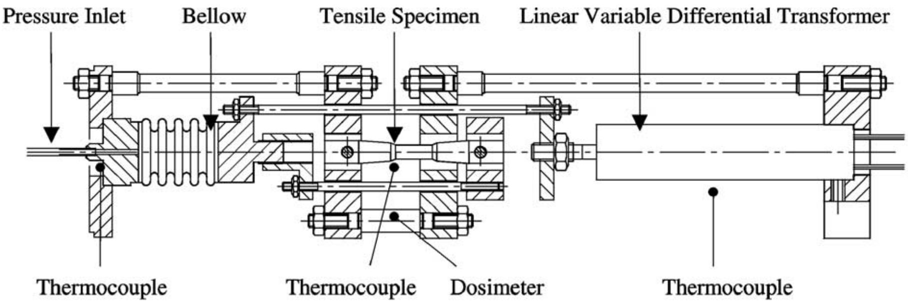
The pneumatic servo-controlled pressure adjusting loop is based on continuous helium gas flow through the electrically controlled servo valve. The movement of the bellows is controlled by LVDT sensor which also gives the feedback signal for the servo controller. The tensile test was performed under a constant displacement rate where the servo controller compares the actual LVDT signal to the set value and close/open servo valve to induce the movement of the bellow by increase/decrease of the bellow pressure.

A number of modules were extensively tested for their functional performance and reliability before finalizing the design. The detailed design and geometry of the module was then adjusted to make it compatible with the irradiation conditions in the BR-2 reactor (i.e. the geometrical, neutronic, thermal and cooling environment).

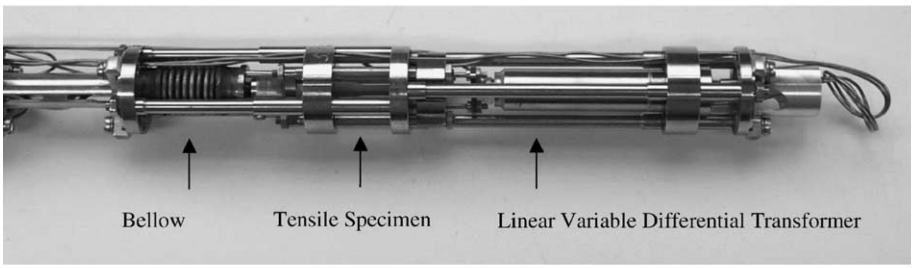
To accommodate the test module and the necessary instrumentation to perform the uniaxial tensile test in the reactor, special irradiation rigs were designed and constructed at Mol. Fig. 4(a) shows the simplified layout and operational features of the test module including the instrumentation. The photograph in Fig. 4(b) shows the final assembly of the test module, specimens and the necessary instrumentation. The whole assembly is loaded in the irradiation rig which is hanged into a thimble tube. During irradiation the whole test assembly including the module and the specimens remained submerged in stagnant demineralised water. The temperature profile in each module was measured by three thermocouples placed at different levels (LVDT, specimen and bellow) in the rig (see Fig. 4(a)). Three dosimeters were placed at the specimen level to measure the neutron flux.

2.3. In-reactor uniaxial tensile test

The irradiation rig was manually inserted into the open tube position G60 of the BR-2 reactor core during steady state operation of the reactor. The tensile



(a)



(b)

Fig. 4. The figure shows (a) the simplified layout and operational features including the necessary instrumentation of the test module and (b) the final assembly of the complete test module in the irradiation rig.

modules together with the tensile test specimen were irradiated with neutron flux of 3×10^{17} n/m^2 s ($E > 1$ MeV) corresponding to a displacement damage rate of $\simeq 6 \times 10^{-8}$ dpa/s. The temperature of the test module increased rapidly due to a gamma heating power of 4.4 W g^{-1} and the stagnant reactor pool water close to test specimen reached an equilibrium temperature of about 90 °C within about 10–15 min. It should be noted that the test specimen was not loaded during this temperature excursion of the module and that the movement of the bellow was continuously measured with the LVDT sensor. The uniaxial tensile test using a constant strain rate of 1.3×10^{-7} s^{-1} was started about 4 h after the rig was inserted in the reactor core. In other words, the displacement damage accumulated for 4 h corresponding to 8.6×10^{-4} dpa while the tensile specimen was still without any load. It should be pointed out that this low stain rate was chosen to ensure that the specimen should survive the in-reactor deformation for long enough time to accumulate a displacement dose level of about 0.1 dpa. This was necessary in order to assess the dynamic effects of irradiation and the applied stress on the deformation behaviour of the specimen.

The tensile test was discontinued soon after the specimen showed a rapid decrease in the applied stress indicating the initiation of fracture (i.e. before the fracture was complete). The test rig was then taken out of the reactor.

3. Experimental results

Before presenting the actual results of the in-reactor tensile testing experiment it is only appropriate to clarify the experimental conditions under which the materials response has been determined. Furthermore, this clarification is crucially important in order to understand and appreciate fundamental differences between the traditional post-irradiation tensile tests and the present in-reactor tensile test. The post-irradiation tensile test is performed, for instance, on a specimen which has been already irradiated (in the absence of any applied stress) at a certain temperature and to a certain displacement dose level. In other words, the specimen, prior to the commencement of the tensile test has a given defect microstructure which has evolved in response to the irradiation temperature and the dose level. The stress–strain response measured during a post-irradiation test must reflect, therefore, the deformation behaviour of the frozen-in irradiation-induced microstructure.

In the case of the in-reactor tensile test, on the other hand, the situation is entirely different. Here, the tensile specimen is loaded in the fully annealed condition and begins to deform when it has only a relatively low density of irradiation-induced defect clusters. Hence, the material will begin to deform in a manner similar to the unirradiated virgin material with a characteristic low yield stress. However, as test continues with a constant damage rate and strain rate, the density of both the irradiation-induced clusters (interstitial loops and clusters and vacancy SFTs) and deformation-induced dislocations will increase with increasing displacement dose.

Thus, the increase in the stress level at a given plastic strain value is due not only to the increase in dislocation density and dislocation–dislocation interactions (i.e. work hardening) but also includes the effect of increase in cluster density as well as of the cluster–dislocation interactions (i.e. radiation hardening). It is, therefore, rather complicated to make a direct comparison between the stress–strain curves obtained during the post-irradiation tensile tests and those determined during the in-reactor dynamic tensile experiments.

Fig. 5 shows the result of the in-reactor uniaxial tensile test performed at $\simeq$90 °C as a function of irradiation time. First, Fig. 5(a) illustrates the magnitude of the accumulated strain in the specimen for the applied strain rate of 1.3×10^{-7} s^{-1} as a function of irradiation time. The corresponding stress response of the specimen is shown in Fig. 5(b). It should be noted here that the stress response of the specimens includes the contribution of increasing displacement damage level as a function of irradiation time (Fig. 5(c)). As indicated in the previous section, it took about four hours (after the specimen was brought into the reactor core) of adjustment of the displacement of the bellow to ensure that the specimen was under stress. In other words, the actual tensile test with a constant strain rate of 1.3×10^{-7} s^{-1} started only after four hours of irradiation (i.e. 8.6×10^{-4} dpa). This is illustrated in the insert in Fig. 5(b).

In Fig. 5(b), the 'effective' elastic line (based on out-of-pile tests of the material in the test module) is shown to indicate that the specimen seems to deform elastically up to a dose level of about 3.6×10^{-3} dpa. At this dose level, the stress acting on the specimen reaches a value of about 90 MPa. Beyond this dose level, the specimen begins to deform plastically and becomes progressively harder with increasing dose and strain level. As indicated earlier, this effective hardening must be a combined effect of the conventional work hardening due to increasing strain and the radiation hardening due to an increasing amount of irradiation induced defect clusters. As can be seen in Fig. 5(b), the increase in hardening decreases with increasing strain and irradiation dose level and finally saturates at a stress level of about 180 MPa after about 225 h of irradiation (corresponding to $\simeq$10% strain and a displacement dose level of 4.9×10^{-2} dpa). Soon after the saturation, the stress acting on the specimen begins to decrease indicating the onset of failure. Before the onset of failure, the specimen had achieved a uniform strain level of about 12%. At a total strain level of 13.7% (i.e. after 308 h of irradiation) the test was discontinued.

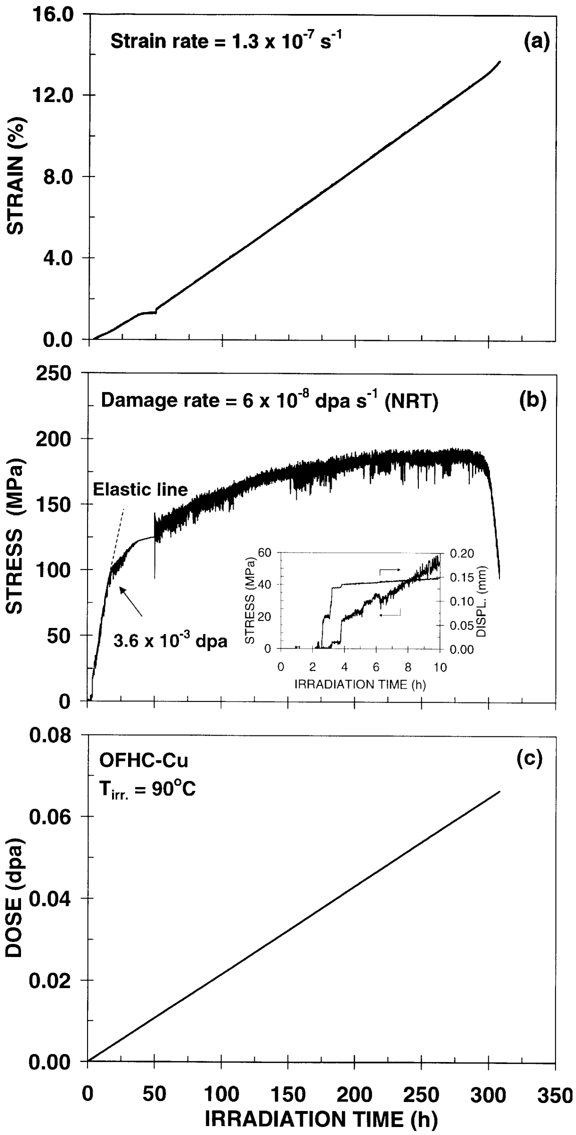


Fig. 5. The dynamic response of the tensile specimen during in-reactor tensile test showing (a) build-up of strain, (b) the corresponding stress acting on the specimen and (c) accumulation of displacement damage (in dpa) as a function of irradiation time. During the test the whole test module, including the tensile specimen, remained submerged in stagnant water at about 90 °C. The water temperature is measured at three different places (see Fig. 4(a)) and is recorded continuously throughout the whole experiment.

4. Discussion

As can be seen in Fig. 5(b), the stress response of copper (under irradiation) as a function of irradiation time (i.e. strain and displacement dose) has a similar form to that of the stress–strain curve obtained for copper in the unirradiated condition [5]. In other words, the transition from the elastic to the plastic regime occurs very smoothly and without any transient such as a yield drop. Furthermore, the hardening in the plastic regime takes place also very smoothly in a manner very similar to the one exhibited by the unirradiated material. This behaviour would suggest that the plastic deformation in the dynamic tensile test under irradiation occurs in a homogeneous fashion, at least up to the strain level at which the increase in hardening saturates (i.e. ≃12%).

It is interesting to note in Fig. 5(b) that in the present experiment copper exhibits a yield strength of about 90–100 MPa. This is considerably higher than the yield strength of unirradiated copper at 100 °C (i.e. 30 MPa) [5]. The most likely explanation of this difference may lie in the fact that the specimen in the present experiment accumulates displacement damage for a period of about 17 h (i.e. 3×10^{-3} dpa) before it begins to deform plastically. In other words, the increase in the yield strength is most probably due to the resistance provided by the accumulated number of defect clusters at the damage level of 3×10^{-3} dpa to the production and motion of dislocations. This would be fully consistent with the experimental results on the effect of irradiation dose level on the yield strength in the post-irradiation deformation experiments [5].

Finally, it should be pointed out that at present no clear and realistic explanation can be advanced as to why the material looses its ability to deform plastically already at a strain level of about 12%. May be a detailed microstructural analysis of the specimen would help answering this question.

5. Conclusions

The present work has demonstrated that it is technically feasible to carry out well defined and controlled in-reactor dynamic tensile tests. This makes it possible to investigate the intrinsic role of the applied stress and the displacement damage acting concurrently in determining the global deformation behaviour of the material under dynamic irradiation conditions.

The most significant feature of the present results is that during the dynamic in-reactor test, the material deforms uniformly and in a homogeneous fashion and does not show any sign of yield drop and plastic instability (i.e. low-temperature embrittlement) as commonly demonstrated by the post-irradiation experiments.

However, the fact that the material under the present irradiation and test conditions achieves a uniform elongation of only about 12% is still a matter of concern. This is particularly since it remains unknown as to what causes this reduction in the uniform elongation. Further investigations are necessary to determine and understand the factors responsible for the reduction in the uniform elongation due to irradiation. The same pure copper tensile tested at 100 °C in the unirradiated condition yields, for example, a uniform elongation of about 56% [5]. New experiments are being designed to address this issue.

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