STATUS OF IN-REACTOR TENSILE STRAINING OF PURE COPPER AT A CONSTANT STRAIN RATE -

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OBJECTIVE

The primary objective of this collaboration was to study the effect of in-reactor tensile straining on microstructural evolution and mechanical behavior in pure copper irradiated at 90°C in the BR-2 reactor in Mol, Belgium.

SUMMARY

Annealed tensile samples of pure copper were irradiated in the fission reactor BR-2 in Mol, Belgium at 90°C with a damage rate of 6 x 10^{-8} dpa/sec. The tensile specimens were a sheet-type specimen with a 3-mm gage width and a gage length of 7 mm. The first experiment involved two specimens, one of which was irradiated with no load to provide a comparative specimen to the other specimen, which was loaded at a constant strain rate of 1.3 x 10^{-7} s^{-1}. The uniaxial tensile load was applied 4 hrs after the irradiation rig was inserted into the reactor core. This corresponded to a total displacement damage of 8.6 x 10^{-4} dpa accumulated before the tensile test was started. The tensile test was considered complete once the load began rapidly falling and reached 100 MPa, at which level the test was stopped and the load quickly reduced to zero to leave the specimen intact. For these test conditions the specimen reached a total plastic strain of ~13% when the test was stopped, considerably less than that of unirradiated pure copper tested under the same strain rate, which was on the order of 50% total elongation. The second experiment involved two samples irradiated under identical irradiation and test conditions, but in this case the tensile load was not applied until a total dose of 1 x 10^{-2} dpa (~50 hours after insertion into the reactor core) was reached. Once the load was applied, the stress immediately climbed to ~150 MPa with little plastic strain, followed by a small yield drop and work hardening up to a maximum stress of ~200 MPa. As in the first experiment, the tensile test was stopped when the load began decreasing and reached a level of 100 MPa.

These samples were removed from the reactor and shipped to Risø, where mechanical tests were performed on the unloaded specimens and microstructural characterization performed on the all four individual conditions. The tensile specimens subjected to the in-reactor loading were found to have a necked region with a crack partially separating the two halves of the tensile specimen. Transmission electron microscopy (TEM) specimens were taken from areas as close to the cracked region as possible and also from regions further into the gage section away from the failed region. The TEM analysis revealed no evidence of substantial homogenous plastic flow in either of the loaded conditions, i.e. no cell wall formation and only minimal dislocation tangles due to forest hardening interactions. Dislocation channels were the predominant marker of plastic flow, however, these channels varied greatly from area to area in number density and width. The dislocation channels were relatively free of deformation-induced dislocations; however, a qualitative assessment indicated defects such as stacking fault tetrahedra were still present inside the channels, although at a much lower density than the surrounding regions outside the channels. These defects may have formed inside inoperative dislocation channels as the irradiation continued. This work demonstrated that the defects produced during irradiation begin to alter the deformation response of the material from very early stages even in the presence of deformation-induced dislocations, effectively inhibiting homogenous flow and the accumulation of dislocations. Future work will quantify the distribution of defects and dislocations in the microstructure.

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PROGRESS AND STATUS

Introduction

Post-irradiation tensile testing has been the primary investigative method for studying the effects of radiation hardening on metals and alloys. These experiments have typically involved irradiation of a particular material in an unstressed condition to a desired displacement level, followed by removal from reactor for testing and microstructural characterization. In effect the specimens are allowed to accumulate radiation damage in the absence of an applied stress and the deformation-induced dislocations. In-reactor pressurized tube creep tests have proven that applied stresses can produce a different microstructure than observed in post-irradiation testing and characterization of materials not loaded when in reactor, but these tests were usually not aimed at studying radiation hardening at low irradiation temperatures (<0.3-0.4T_m). While the post-irradiation tensile experiments have certainly provided unique insights into the behavior of irradiated materials, these experiments do not necessarily represent their in-service behavior.

The materials employed in the structural components of fission or fusion reactors are exposed simultaneously to external stresses and irradiation-induced defect populations produced continuously during neutron irradiation. Under these conditions both the magnitude and the spatial distribution of defect accumulation and hence the deformation behavior may be substantially different from that observed in the case of post-irradiation experiments. This raises the question of whether the results and conclusions of such post-irradiation testing can always be safely extrapolated to the behavior of structural materials in a nuclear reactor. To address this question, a series of in-reactor uniaxial tensile tests have been initiated in the BR-2 fission reactor in Mol, Belgium. The dynamic stress-strain curves have been measured for samples of pure copper loaded at a continuous strain rate while neutron irradiated at 90°C. The in-reactor tests have been completed and the samples returned to Risø, where the samples have recently been characterized by TEM. The preliminary results of microstructural characterization and their implications will be discussed in the following sections.

Experimental Procedure

A more thorough description of the experimental setup for the in-reactor tests have been described elsewhere by Singh et al. [1], so only a brief description will be provided here. The material used for this experiment was oxygen-free high conductivity (OFHC) copper containing 10 and 3 ppm of Ag and Si, respectively, and <1 ppm of Fe and Mg each. Tensile samples were fabricated from polycrystalline copper annealed for 550°C for 2 hrs to yield a well annealed microstructure with a dislocation density $<10^{12}$ m^{-2} and a grain size of ~30 µm. The specimens were loaded into a specially designed test assembly constructed to apply a continuous load at a constant strain rate inside the reactor core. Additional specimens were included near the loaded samples, but these specimens were not loaded during the experiment in order to provide a direct comparison.

The basic principle of the tensile test module inserted into the reactor core involved using a pneumatic bellow (pressurized by He gas) to apply the stress and a linear variable displacement transducer to measure the resulting displacement. The entire test assembly and specimens remained submerged in stagnant, demineralized water for the duration of the experiment. The temperature was monitored using three thermocouples located in the assembly near the specimen, and three dosimeters were placed in the assembly to measure the neutron flux. The test assembly was inserted into the core in open tube G60 of BR-2 during steady state operation of the reactor. The neutron flux at this position was 3×10^{17} n/m^2 (E>1 MeV) corresponding to a displacement damage rate of ~6×10^{-8} dpa/s. The water near the specimens reached 90°C in ~15 minutes, and the bellow was monitored constantly to ensure no load was applied to the specimen. The strain rate chosen for this experiment was 1.3×10^{-7} s^{-1} to ensure an appreciable displacement level was reached before the specimen failed. The tensile test was stopped

once the stress decreased to 100 MPa, at which point the load was completely removed to preserve the specimen.

For the first set of experiments involving copper (other metals and alloys will be tested in future experiments), two test conditions were employed. The first condition involved loading the specimen at four hours after insertion of the test assembly into the reactor core. The total dose when the load was applied was ~8.6 x 10^{-4} dpa. The second condition allowed the specimen to sit in the reactor for 50 hours before the load was applied, letting the specimen accumulate a total dose of 1 x 10^{-2} dpa. The resulting load versus time curves for these two test conditions are compared in Figure 1. The 1st condition ended when the specimen began failing after ~300 hours (the total strain when the load was removed was ~13% elongation), whereas the second test condition began failing after roughly a 160 hours in reactor under load (load was removed at ~7.5% total elongation). These specimens were removed from the reactor and shipped to Risø. Optical metallography and SEM revealed a necked region in the gage section that had begun separating by cracking. TEM disks were punched from regions close to the cracked region and further away to characterize the microstructure that may have experienced different levels of strain than the crack region. A preliminary assessment of the microstructural results will be presented in the following section.

Results

The TEM characterization revealed that neither of the two samples tested in reactor exhibited cell walls or a significant dislocation network. Examples of the microstructures of the two tested conditions (13% elongation and 7.5% elongation) are shown in Figures 2 and 3, respectively. It was anticipated based on the tensile curves in Figure 1 that each of the two conditions would exhibit some dislocation networks and

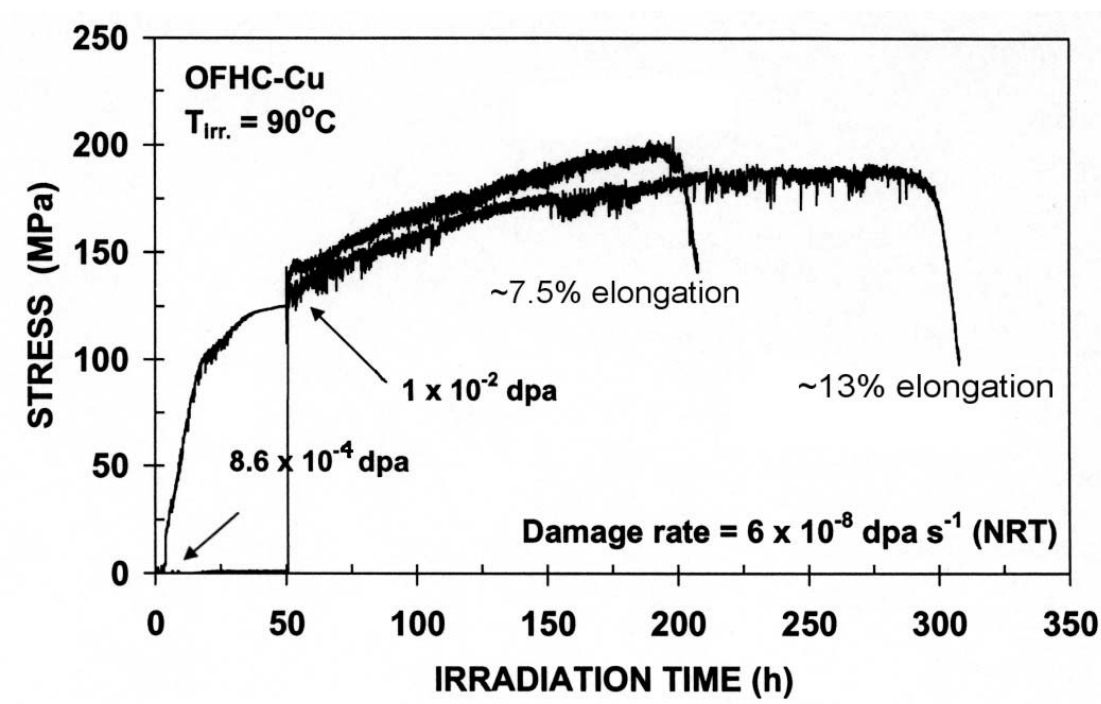

Figure 1. Uniaxial loading curves versus time are shown for the two different samples loaded while in reactor. The first sample loading was started 4 hours the test assembly was inserted into the reactor core, whereas in second experiment the test was started 50 hours after insertion. The first test was stopped at ~13% elongation, whereas the second test was stopped at ~7.5% elongation. The fluctuation in the tensile stress was determined to be noise in the electronics and test assembly.

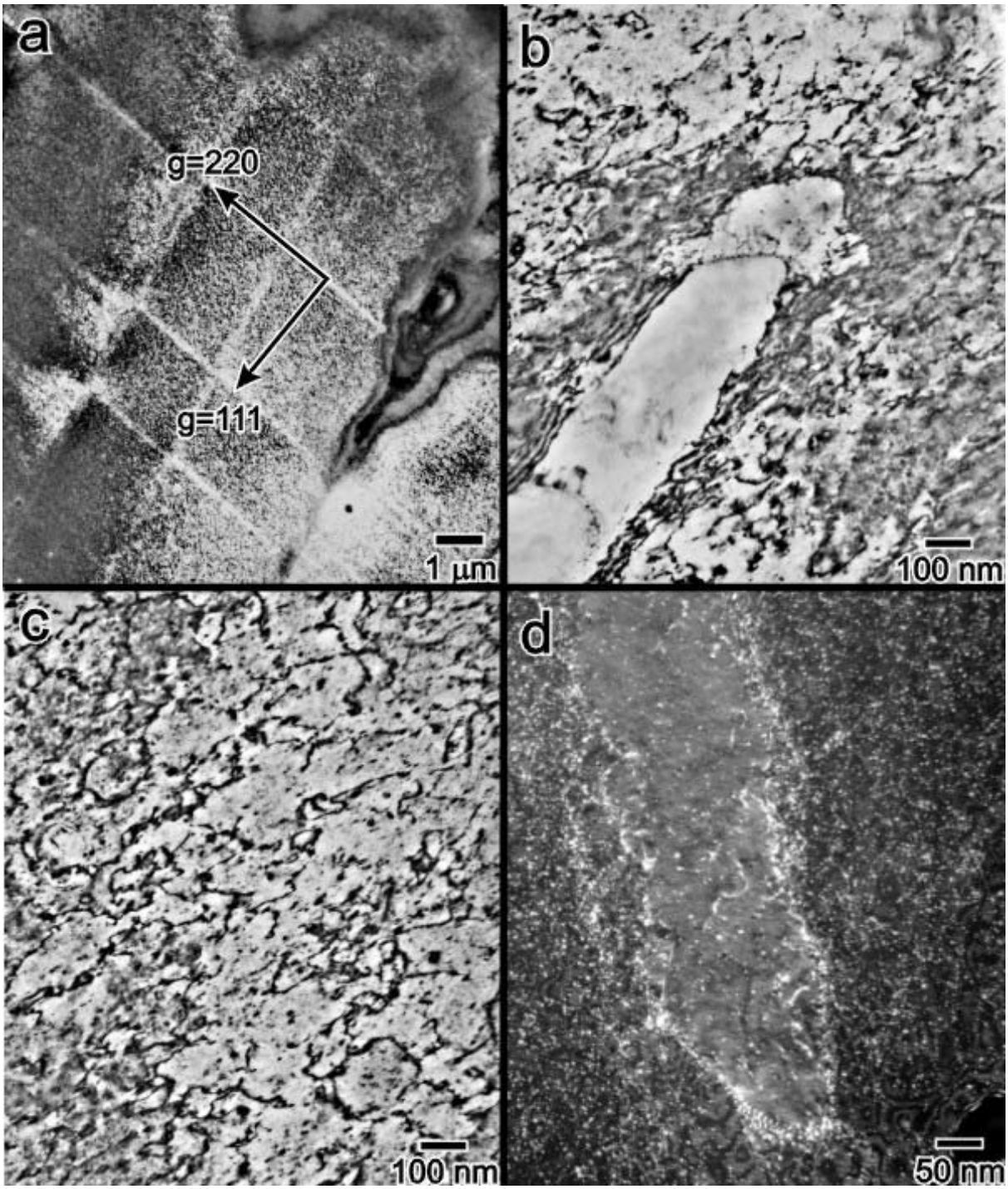

Figure 2. Dislocation channels in the sample that failed after 13% are shown in (a) oriented on a g=111 2-beam condition between a <112> and a <011> zone axis. A cleared channel similar to a shear band in heavily deformed, unirradiated copper is shown in Figure (b) and (d). The dislocation microstructure shown in (c) indicates some minor movement or "percolation" of the dislocations through the defects in the matrix, but in general no large scale clearing of the defects is visible.

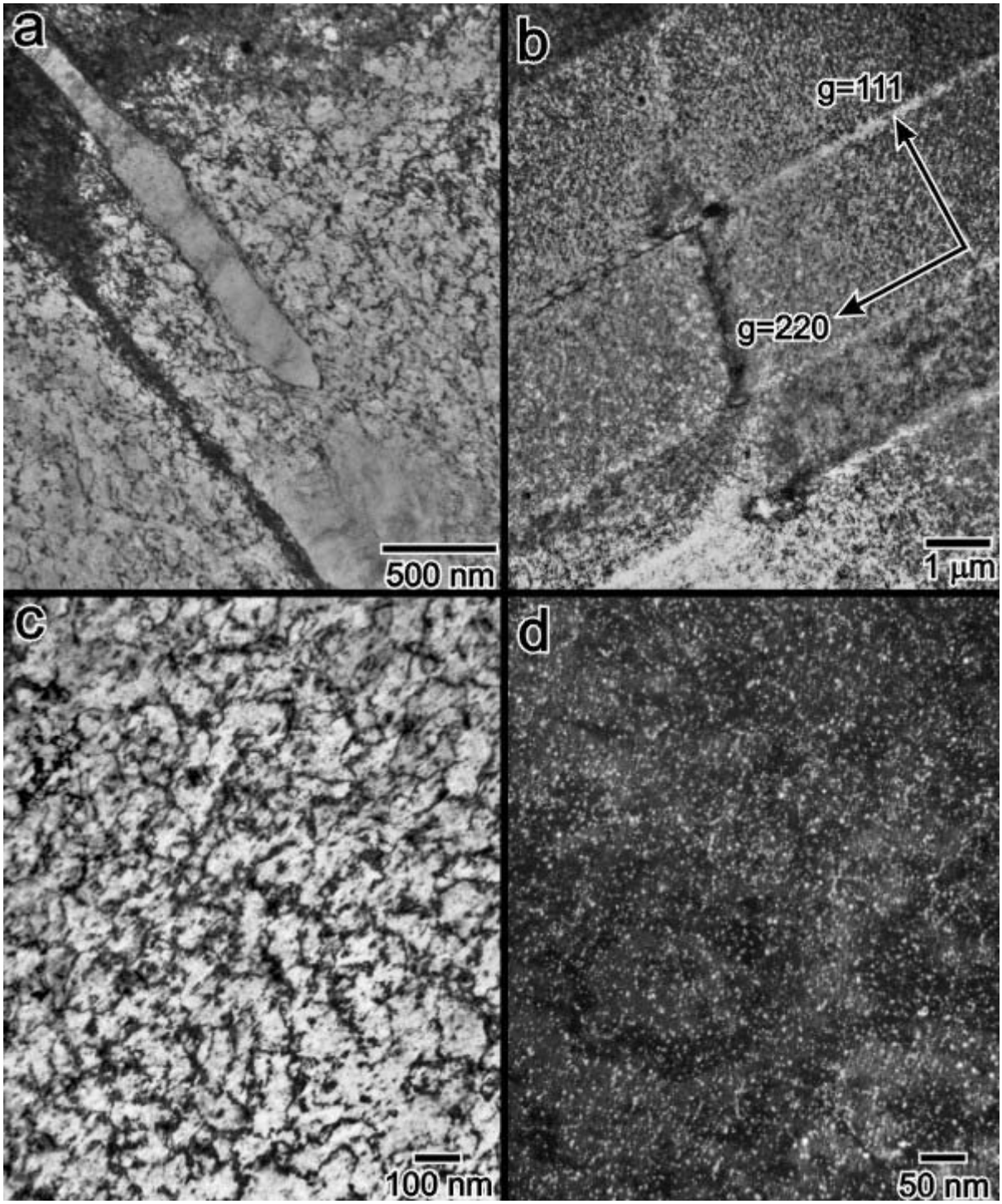

Figure 3. Examples of dislocation channels in the sample that began failing around 7.5% elongation. The channel in (a) resembles a shear band commonly observed in heavily cold rolled copper. The dislocation segments shown in (c) don't appear to have participated in the overall deformation, perhaps pinned by the high density of small defects shown in (d).

perhaps a cell wall structure given the large degree of plastic strain and work hardening observed. However, the most noticeable features of the deformed microstructures were the presence of dislocation channels and the lack of homogenous deformation, not unlike the situation observed in tensile samples irradiated and then tested out of reactor. The degree of channeling varied considerably from grain to grain, and the TEM samples taken closer to the failure region contained grains that exhibited a greater degree of channeling. Because of the variable microstructure and limited sampling volume it is difficult to make quantitative comparisons on channeling between the two different test conditions. Overall there does not appear to be a definitive difference between the two cases, but further analysis of the dislocation and defect densities is needed to confirm this statement. The defect densities in each sample are very high in the regions between the channels, and with few exceptions no general clearing of defects outside channeled regions occurred. Examples of grain and twin boundaries intersected by channels were quite common, and these examples often demonstrated that large strains had occurred judging from the shear offset along the boundaries.

The absence of cell wall formation and no visible reduction in defect density outside the channels (based strictly on qualitative estimates at this point), indicates that the defects produced during irradiation were very effective at hindering the motion of dislocations generated during the early stages of deformation. Further support for this can be extracted from the behavior of the sample tested in the second experiment where the load was not started until the sample had accumulated a dose of 10^{-2} dpa. The loading curve shown in Figure 1 indicates that not only did the extra dose result in a higher yield stress before plastic deformation began, but that a visible yield drop occurred. One explanation for the lower elongation in this case is that the extra dose accumulated forced the material into a more severe localized flow mode than the sample where the load was applied at a lower dose (8.6×10^{-4} dpa). The experimental data suggests that for this combination of strain rate and radiation damage rate, the defects produced during irradiation can strongly affect both the generation and mobility of the dislocations. A higher strain rate might prove more effective at removing the defects and prevent a buildup of high density, but further experimentation is needed. A higher strain rate would, of course, limit the dose accumulated before the sample failed, making direct comparisons very difficult.

Future Work

A more detailed analysis of microstructures for each of the different test conditions will be performed in the next two quarters. A comparison of defect and dislocation densities outside and inside the dislocation channels will be made, as well as comparisons made of the defect and dislocation distributions produced by the two loading conditions. These results will also be compared to the unloaded reference samples irradiated along side each of the in-reactor tested samples to provide a baseline for the microstructural characterizations. Additional comparisons will be made with the microstructures of unirradiated samples of pure copper tested under the same strain rate and test temperatures to further evaluate the effect of in-reactor loading. Future irradiation experiments are planned that will include in-reactor testing of CuCrZr, pure Fe and a ferritic steel.

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References

[1] B. N. Singh, S. Tähtinen, P. Moilanen, P. Jacquet and J. Dèkeyser, Journal of Nuclear Materials, **320**, 299-304 (2003).