

# In-beam mechanical testing of CuCrZr

P. Marmy \*

*Fusion Technology-Materials, CRPP, EPFL, Association EURATOM-Confédération Suisse, 5232 Villigen PSI, Switzerland*

## Abstract

In the ITER design, CuCrZr has been selected as the heat sink material for components of the first wall and the divertor. The objective of this work is to check the material fatigue performance when the CuCrZr alloy is cyclically deformed concurrently with irradiation, using an in situ testing machine placed in a 590 MeV proton accelerator. Three fatigue experiments have been conducted at 100 °C, under strain control, at a total strain range of 0.8%. The in-beam specimen reached the longest life. The post-irradiation tested specimen had the shortest life. The total plastic strain measured in the in-beam specimen was larger than the plastic strain measured in the statically irradiated specimen or in the unirradiated specimen.

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## 1. Introduction

In the ITER design, a high thermal conductivity material, a copper alloy, is interposed between the materials facing the plasma and the cooling water channel structure. The main role of the copper interlayer is to transport the heat deposited in the armour materials to the cooling water channel structure. Due to the pulsed operation of the tokamak, the stresses imposed to the material will be cyclic in nature. They will act on the copper alloy, which is at the same time being irradiated with a flux of high energy neutrons.

Due to a good combination of physical and mechanical properties, among them better fracture toughness and relatively good irradiation resistance, CuCrZr seems to be the most adequate material for the copper interlayer in ITER [1].

The low cycle fatigue performance has been studied extensively in the past for the copper alloys candidate materials (CuCrZr, CuAl25, CuNiBe). The fatigue behaviour has been measured before and after irradiation with fission neutrons, at temperatures between 22 and 350 °C.

For the CuCrZr alloy in particular, the results obtained seem to indicate a fatigue behaviour close to that

observed in OFHC Cu, from the point of view of the mechanical properties and the dislocation structures developed after deformation [2]. When compared to CuNiBe or CuAl25 (Glidcop) the fatigue performance is slightly inferior.

Taking into account recent results from tensile and fatigue studies [3–5], it was decided to conduct the first in situ testing of CuCrZr around 100 °C, where the material seems to be the most sensitive to irradiation effects.

The test program includes three basic tests, all performed at 100 °C and at a strain amplitude of 0.8%; an unirradiated fatigue test performed with the in situ device, the *in-beam* test and the *post-irradiation* comparison fatigue test.

## 2. Experimental procedure

### 2.1. Material

The chemical composition of the CuCrZr alloy is as follows: Cr 0.78 wt%, Zr 0.13%, Si 0.003% and the balance Cu. The material came from the Otokumpu company in Finland, in the form of a plate of 46 mm thickness. The plate had been previously cold rolled about 60% after solution anneal at 960 °C for 1 h and subsequently precipitation heat treated at 460 °C for 2 h.

\* Tel.: +41-56 310 2932; fax: +41-56 310 4529.

E-mail address: [pierre.marmy@psi.ch](mailto:pierre.marmy@psi.ch) (P. Marmy).

The following heat treatment consisting of a solution anneal and precipitation heating, was given to the specimens:

30 min at 975 °C, water quench,

30 min at 475 °C, water quench.

After the above heat treatment, a bimodal distribution of mesoscopic and microscopic precipitates is observed in the material. The mesoscopic precipitates have sizes between 0.1 and 1  $\mu\text{m}$  and spacing of 5–10  $\mu\text{m}$ . The microscopic fine precipitates have a size of 2–10 nm (mean size around 3 nm) and spacings of 30–40 nm. The fine precipitation controls the mechanical properties of the alloy. Details on the microstructure are given in [5,6].

## 2.2. In situ device

In beam fatigue tests are conducted with the in situ device, a specially dedicated irradiation head of the PIREX facility [7], at the PSI accelerator. The irradiating particle used in the PIREX facility are 590 MeV protons. They deposit a large amount of heat in the materials, which has to be removed by forced cooling. A flow of pressurized helium (30 bars, 100  $\text{Nm}^3/\text{h}$ ) is used for cooling the irradiated specimen.

The in situ device consists of three subsystems: the vertical drive which moves the machine *in* and *out* of *beam*, the helium temperature controlling system consisting of an heat recuperator and a heater and finally the testing machine on the bottom of the system. The in situ device is shown in Fig. 1, in the extended configuration.

The specimen is mounted by means of grips, at the bottom of the system.

The in situ device is installed in a container and the irradiation is performed under vacuum. After the experiment, the device is transported to the hot cells in a shielded transport cask. More details on the machine are given in [8].

## 2.3. Test specimen

The test specimen is a tubular specimen with an inside diameter of 2.5 and 0.45 mm wall thickness (see [8]). The geometrical gauge length is 5 mm. Two thermocouples are glued on the copper material, in the center and at the end of the gauge length. The thermocouples are needed for adjusting the irradiation temperature and help for centering the beam. The specimen is sealed with two metallic O-rings. The failure under fatigue is indicated when the pressure in the surrounding vacuum increases.

The displacement is measured with two ceramic bars and transformed into an electrical output with a classical strain gage transducer.

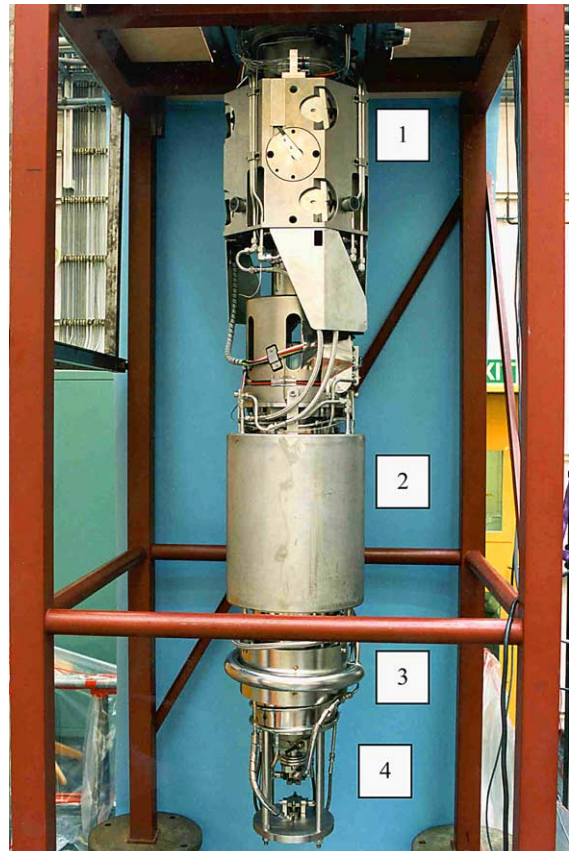


Fig. 1. Photograph of the in situ device with its main components, the vertical drive which moves the system up and down 200 mm (1), the heat recuperator (2), the heater which heats the helium up to 400 °C (3), the grips of the testing machine with helium manifolds (4). The testing machine itself is located behind the recuperator and heater and is protected from the heat by a water cooled shield.

## 2.4. Beam

The proton beam has a gaussian distribution. It was adjusted to a size of  $4\sigma_y = 3$  mm and  $4\sigma_x = 6$  mm. The beam was then wobbled with an amplitude of 2.5 mm along the gauge length, with a frequency of about 3 Hz, to achieve a constant dose distribution. The beam intensity was 11  $\mu\text{A}$ . Under these conditions, the mean current density was 50  $\mu\text{A}/\text{cm}^2$  and the mean proton flux was  $3 \times 10^{14}$   $\text{p}/\text{cm}^2/\text{s}$ . The resulting dose rate was  $1.5 \times 10^{-6}$   $\text{dpa}/\text{s}$ .

## 2.5. Fatigue parameters

The fatigue was conducted under total strain control, following a  $R = -1$  symmetrical signal. During all mechanical tests, a test temperature of 100 °C was adjusted at the specimen, taking into account beam

heating. The total strain amplitude was 0.8% (half strain amplitude 0.4%). The test frequency was chosen at 0.01 Hz ( $T = 100$  s,  $\dot{\epsilon} = 1.6 \times 10^{-4}$  [s<sup>-1</sup>]) for the *in-beam* test. At 100 °C, the test frequency should have no influence on the mechanical behaviour, therefore, for practical reasons, the test frequency for the comparison tests was increased to 0.028 Hz ( $T = 36$  s).

The end of life criteria used to compare the lives was when the main crack penetrated through the specimen wall ( $N = N_a$ ). This point was clearly indicated in the *in-beam* experiment by a failure of the vacuum produced by the out flowing helium. To this point corresponded an inflexion point in the bottom part of the strain–stress hysteresis, which is also clearly marked in the classical fatigue specimen, although no helium pressure is present in the specimen. After the test was interrupted by the vacuum failure, the test was continued without helium, in a vacuum, at an equilibrium temperature of about 40 °C.  $N_a$  is considered a better parameter for comparison since at cycles higher than  $N_a$ , the mechanical conditions are modified. Depending on the orientation of the main crack, the number of cycles to specimen separation is also affected by a larger dispersion.

The damage ratio of the *in-beam* test was  $\dot{D}/\dot{\epsilon} = 9.4 \times 10^{-3}$  [dpa]. The damage ratio is the ratio of the displacement damage rate and the fatigue strain rate. This basic parameter is used to compare different in situ experiments [8].

### 3. Results

The total stress  $\Delta\sigma$  as a function of the number of cycles is represented in Fig. 2, in a linear scale, for the three different experiments. The number of cycles to failure  $N_a$  is indicated by an arrow. The *in-beam* experiment I32I01 was interrupted before failure at  $N = 2246$  cycles, because the deadline of the shutdown

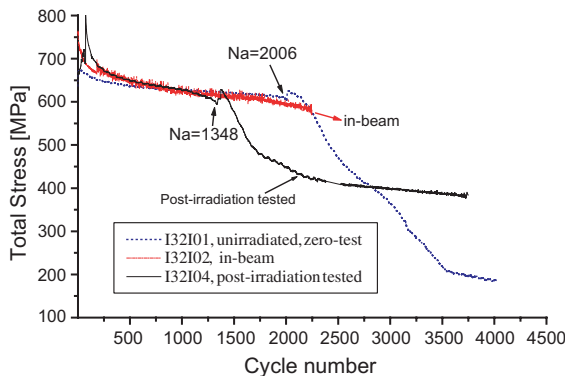


Fig. 2. The total plastic strain as a function of the number of cycles for experiments I32I01, I32I02 and I32I04.

forced the system to be stopped. During the shutdown, a repair of the vertical drive produced unknown loading of the specimen and the experiment was definitely stopped. The integrated beam current dose at the interruption was 2.453 A s. Nevertheless specimen I32I01 has the longest life as compared to the zero-test I32I01 ( $N_a = 2006$ ) and the post-irradiated test I32I04 ( $N_a = 1348$ ). The post-irradiation tested specimen has the lowest life. The softening rate is the highest in the *post-irradiation* tested specimen and the lowest in the unirradiated test.

Fig. 3 shows the total plastic strain as a function of the number of cycles. I32I04 is irradiated before start of test to 1.521 A s. The post-irradiated specimen has the lowest value in accordance with the irradiation hardening. The plastic strain in the *in-beam* test is higher than in zero-test.

Fig. 4 compares the first hysteresis loops of experiments I32I01, I32I02 and the post-irradiation

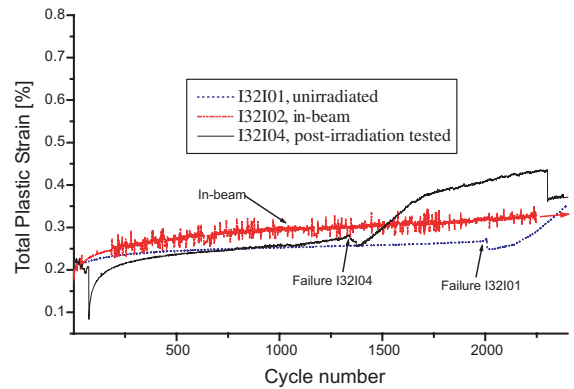


Fig. 3. The total plastic strain as a function of the number of cycles for experiments I32I01, I32I02 and I32I04.

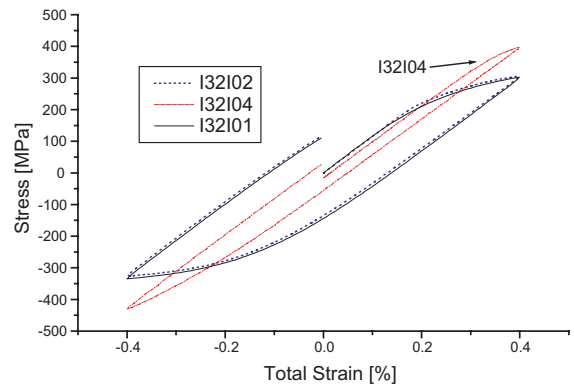


Fig. 4. First hysteresis loops of experiments I32I02, I32I01 and I32I04. I32I04 received a dose of 1.521 A s. I32I02 was irradiated to 0.0033 A s for beam adjusting and I32I01 is the unirradiated specimen.

experiment I32I04. I32I02 has been irradiated statically to 0.0033 A s for beam adjusting. The static irradiation in I32I04 introduces a big difference in terms of plasticity but not in terms of stress. The reduction of plastic strain is more significant than the increase of the stress at 0.4% total strain. It is also interesting to note that no yield point phenomena occurs after irradiation, at a strain amplitude of 0.4%, at a test temperature of 100 °C.

The dosimetry was based on the Mn54 and Co57 isotopes generated by the protons. I32I02 and I32I04 received respectively a dose of 0.23 dpa ( $4.5 \times 10^{19}$  p/cm<sup>2</sup>) and 0.11 dpa ( $2.2 \times 10^{19}$  p/cm<sup>2</sup>).

#### 4. Discussion

The different conditions imposed on the specimens, unirradiated, *in-beam* and *post-irradiation* tested have a clear effect on the fatigue endurance of the material. The *in-beam* specimen has the longest life. It lasts longer than the unirradiated specimen and significantly longer than the *post-irradiation* tested specimen. In fact, the response of the copper alloy is strange because despite the fact that the stress level in situ was slightly higher, in particular at the beginning of the test (see Fig. 2), the crack growth rate is lower than in the unirradiated material. Also interesting to note is the higher softening rate in the case of the *in-beam* specimen (see Fig. 2). The stronger softening rate is also clearly visible in Fig. 3. The plastic component of the strain as well as the rate of increase of the plastic strain are larger in the *in-beam* specimen. The *post-irradiation* tested specimen also has a very high rate of plastic strain increase but the amount of plasticity is smaller. Only at test end, the plasticity in the *post-irradiation* tested specimen is larger than in the *in situ* specimen.

One critical aspect of this study is the fact that only one experiment is conducted per condition and knowing the scatter which normally affects fatigue results, the results presented here could be different with a larger number of specimens. This objection is not correct because of the geometry of the specimen and the strain applied. Since the tests are conducted at high total strains (0.8%), we can assume that the time to generate a crack will be small and most of the fatigue life will be crack propagation. Therefore the scatter which usually comes from the crack nucleation period is small in our case. As explained in Section 2.5, once the crack is generated, it has to run a short precise distance, the wall thickness of the tube specimen (450 µm), to produce the failure. Therefore the scatter is probably smaller as compared to the scatter from bulk specimens where the failure criteria is based on a change of the cyclic stress. This change is the overall result of a complex crack propagation mechanism. For these reasons it is believed that the scatter, for the parameters of this experiment, is

low and that the lives measured from single experiments, represent well the general behaviour.

Not much information is available from the literature to compare to our results. Gorynin et al. report some results from bending tests on specimen irradiated at 90 °C to  $10^{21}$  n/cm<sup>2</sup> in SM-2. The authors report an increase of life at a test temperature of 300 °C, after irradiation, especially at low strains. These results cannot be compared to ours, because the test procedure is very different and the heat treatment of the CuCrZr is not described [9].

Work from Singh et al. [3,10] gives some results on the irradiated fatigue life and deformed microstructure of copper alloys. At an irradiation temperature of 100 °C to  $1.5 \times 10^{20}$  n/cm<sup>2</sup> (0.3 dpa), prime aged (the same heat treatment as in this work) CuCrZr specimens tested at 100 °C did not indicate any deleterious effects from the irradiation. But this result is an invalid comparison to this work done at high strain, because the unirradiated strain controlled specimens are compared to stress controlled specimens. Specially at high strains, strain localizations effects could have affected the behaviour of the stress controlled fatigue specimens.

A more recent work from Singh et al. [11] allows a more reliable comparison. Results from strain controlled specimens tested and irradiated at 250 °C to a neutron fluence of  $1.5 \times 10^{20}$  n/cm<sup>2</sup>, are presented. At an imposed strain  $\Delta\epsilon_{tot}$  of 0.8%, the fatigue life of the irradiated specimen is reduced by approximately 20–30%. The number of cycles to failure lies around 2000 cycles, which is similar to the results shown in Fig. 2. At higher imposed strains, the effect of the irradiation is even stronger. The general behaviour shown supports the results presented in this work.

#### 5. Conclusions

A series of experiments have been conducted using an *in situ* fatigue device to study the behaviour of CuCrZr alloy under cyclic deformation and concurrent irradiation with 590 MeV protons, at 100 °C. Three experiments have been completed; an unirradiated test, the *in-beam* fatigue test and a *post-irradiation* fatigue test. The dose reached at the end of the *in-beam* test was 0.23 dpa. The *post-irradiation* tested specimen was irradiated to 0.11 dpa. The main conclusions that can be drawn are as follows:

- The *in-beam* specimen reached the longest life. The *post-irradiation* tested specimen had the shortest life. The *in-beam* test condition gives superior performance in terms of endurance.
- The total plastic strain measured in the *in-beam* specimen was larger than the plastic strain measured in the statically irradiated specimen or in the unirradiated specimen.

- Assuming high strain levels in the CuCrZr components and depending on the dynamic conditions at the first wall of ITER, the effect of the in situ deformation could be beneficial for the fatigue endurance.

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