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Final Report on In-Reactor Creep-Fatigue Deformation Behaviour of a CuCrZr Alloy: COFAT 2

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Abstract

The main objective of the present work was to determine experimentally the mechanical response and resulting microstructural changes in CuCrZr (HT1) alloy exposed concurrently to flux of neutrons and creep-fatigue cyclic loading directly in a fission reactor. Using specially designed test facilities for this purpose, in-reactor creep-fatigue tests have been performed at strain amplitudes of 0.25 and 0.35 % with a holdtime of 10s in the BR-2 reactor at Mol (Belgium). These tests were performed at the ambient temperatures of 326K and 323K. For comparison purposes corresponding out-of-reactor creep-fatigue tests were also carried out. In the following we first describe the details of the creep-fatigue experiments. We then present the main results on the mechanical response of the material in the form of hysteresis loops and the maximum stress amplitude as a function of the number of creep-fatigue cycles during the out-of-reactor and the in-reactor tests carried out at different strain amplitudes. Finally, the dependence of the number of cycles to failure (i.e. creep-fatigue lifetime) on the strain amplitudes is shown. The details of microstructure of the specimens tested out-of-reactor as well as in the reactor were investigated using transmission electron microscopy. The main results on the mechanical response as well as changes in the microstructure are briefly discussed. The main conclusion emerging from the present work is that the lifetime of the in-reactor tested specimens is by a factor of about two longer than in the case of corresponding out-of-reactor tests.

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1. Introduction

At present it seems almost certain that the precipitation hardened CuCrZr alloy will be used both in the first wall and divertor components of the International Thermonuclear Experimental Reactor (ITER). In service, both these components will be exposed to an intense flux of fusion (14MeV) neutrons and will experience at the same time thermo-mechanical cyclic loading (i.e. fatigue) as a result of the cyclic nature of plasma burn operations of the system. Consequently, the structural materials in the reactor vessel will have to endure not only the cyclic loading but also the stress relaxation and micro structural recovery (i.e. creep) during the “plasma-on” and “plasma-off” periods.

In an effort to evaluate the impact of cyclic loading (fatigue) on mechanical performance of CuCrZr alloy, the low cycle fatigue behaviour was investigated at temperatures in the range of 295 to 623 K both in the unirradiated and the neutron-irradiated conditions [1,2]. Recently, the creep-fatigue deformation behaviour of CuCrZr alloy has been studied extensively both in the unirradiated and the neutron- irradiated conditions at 295 and 573 K [3]. In these studies, the effects of holdtime and strain amplitudes were investigated. The results of these investigations exhibited a general tendency that the application of holdtime during creep-fatigue loading practically always reduces the number of cycles to failure. Furthermore, this reduction in lifetime tends to be even higher at lower strain amplitudes. These effects of creep-fatigue mode of deformation on the lifetime of CuCrZr alloy remain rather elusive and at present no clear and reliable explanation can be advanced.

The nature of deformation is likely to get even more complex during creep-fatigue loading in the environment of energetic neutrons because of continuous production of lattice defects and their clusters. These defect and defect clusters would interact with the deformation-induced dislocations. As a result, both dislocation dynamics and defect accumulation are likely to get modified. These modifications would directly affect the mechanical performance of the structural materials during creep-fatigue deformation in a fusion reactor like ITER. To our knowledge, practically nothing is known at present either experimentally or theoretically about the nature of plastic deformation and its impact on the mechanical performance and lifetime of CuCrZr alloy under the complicated conditions of concurrent creep-fatigue loading and neutron irradiation. It is relevant to note here that at least the fatigue behaviour of the CuCrZr alloy has been investigated during irradiation with 600 MeV protons [4].

In view of the above described complications involved in the in-reactor deformation experiments, it must be recognized that the results of out-of-rector creep-fatigue experiments on the unirradiated or irradiated materials are totally inadequate for describing the in-reactor deformation behaviour.

It is therefore essential, at least in our view, to perform well controlled creep-fatigue experiments directly in a reactor to determine the dynamic response of the material when it is exposed concurrently to external force (producing dislocations) and to the flux of neutrons (generating lattice defects and their clusters) as a function of the number of creep-fatigue cycle (and irradiation dose). On the basis of these results, a realistic and reliable assessment of mechanical response and lifetime of materials can be made for the case of creep-fatigue loading in the environment of neutrons in a fission or/and fusion reactor.

Recently an experimental facility has been developed jointly by VTT (Finland), SCK.CEN (Belgium) and Risø National Laboratory (Denmark) specifically to study the deformation behaviour and its impact on mechanical response and lifetime of materials during creep-fatigue loading in the environment of fission neutrons. In this test facility provisions are made to implement holdtime both in the tensile and compression sides of the cyclic tests. Using this facility, some in-reactor creep-fatigue tests have already been performed at strain amplitude of 0.5 % with holdtimes of 10 and 100 s. The results of these tests have already been described and discussed recently [5]. The present report describes the results of additional in-reactor creep-fatigue tests carried out at lower strain amplitudes of 0.25 % and 0.35 % with a holdtime of 10 s.

2. Materials and Experimental Procedure

2.1 Materials

The material used in the present investigations was a precipitation hardened CuCrZr alloy supplied by Outokumpu Oyj (Finland) with a composition of Cu-0.73% Cr-0.14% Zr. The alloy was solution annealed at 1233 K for 3 hours, water quenched and then prime aged at 733K for 3 hours. After this heat treatment the material was water quenched again. Finally, the prime aged material was overaged at 873K for 1 hour and then water quenched. Henceforth this heat treatment will be referred to as HT1. The main purpose of this heat treatment was to coarsen the precipitates so that they could be stronger obstacles to dislocation motion during deformation. The final grain size after HT1 heat treatment was found to be 60µm. The size and geometry of the specimens used in the in-reactor creep-fatigue tests are shown in Figure 1(a). A photograph of one of the specimens used in these tests is shown in Figure 1(b).

2.2 Test module and irradiation rig

In order to carry out in-reactor creep-fatigue tests, a special test facility was designed and constructed. The facility consists of a pneumatic fatigue test module and a servo-controlled pressure-adjusting loop. The pressure-adjusting loop operates on a continuous flow of helium gas. The basic principle of the creep-fatigue test module is based on the use of two pneumatic bellows, one to introduce tensile and the other one to introduce compressive stress / strain on the specimen. A linear variable differential transformer (LVDT) sensor is used to measure the resulting extension of the gauge length of the creep-fatigue specimen during the deformation. The strain measured in the gauge length of the specimen is used to control the pre-determined strain amplitude throughout the whole creep-fatigue test. It should be pointed out that the strain measured by the LVDT is calibrated against the strain measured by strain gauges during the out- of-reactor creep-fatigue tests. Details of the design of creep-fatigue test modules and the procedure for calibrating the stress and strain measured in these modules are described in separate reports [6, 7]. The outside diameter of the test module was 50mm and the total length of the module together with the LVDT was 250mm. The accuracy of the load calibration was found to be ± 1% of the actual value of the stress resulting from the applied pressure causing the displacement in the specimen.

To accommodate the test modules and the necessary instrumentation to be able to perform the creep-fatigue tests in the reactor, special irradiation rigs were designed and constructed at Mol. Figure 2(a) shows the simplified layout and operational features of the test module including the instrumentation. The photograph in Figure 2(b) shows the final assembly of two test modules, two specimens and the necessary instrumentation. The whole assembly is loaded in the irradiation rig which is hung in a thimble tube. During the whole test period, the irradiation rig remained submerged in the demineralised reactor pool water. However, to avoid overheating of the specimens due to gamma heating the pool water was circulated in the irradiation rig at a rate of 600 litres per hour from the top of the test assembly. The temperature profile in each test module was measured by three thermocouples placed at three different positions (i.e. LVDT, specimen and bellow) in the rig. (Figure 2). Three dosimeters were placed at the specimen level to measure the neutron fluance.

2.3 In-reactor creep-fatigue tests

The irradiation rig was designed to accommodate two complete test modules so that two independent creep-fatigue tests can be performed at the same time. In addition, a sub size fatigue specimen was attached to each test module which was irradiated in unstressed condition but with the same neutron flux and at the same temperature as for the in-reactor creep-fatigue test.

The irradiation rig containing the assembly of specimens, LVDTs, cooling system and thermocouples was lowered in the reactor core when the local neutron flux near the rig had reached a steady-state level after the reactor start. The rig was manually inserted into the open tube position of BR-2 reactor core. As the rig was lowered in the open tube, the temperature of the test modules increased rapidly due to the gamma heating power of $2Wg^{-1}$. It took about 10 minutes to reach a stable temperature in the modules 1 and 2 of 326 and 323K, respectively. The test module 1 was situated at the top and the test module 2 at the bottom of the assembly in the rig. The temperature of each test module was measured and recorded separately and continuously. The temperature measured on the test module was taken to be the temperature of the specimen.

The creep-fatigue cyclic loading was initiated almost immediately after the temperature in the test modules had stabilized. The test on the specimen in module 1 was identified as Test No.3 and on the specimen in the module 2 as Test No.4. The Test No. 3 and Test No. 4 were carried out in the strain controlled mode with strain amplitudes 0.25 % and 0.35 %, respectively. It should be noted that the in-reactor Test No 1 and Test No. 2 were carried out with strain amplitude of 0.5 % and with holdtimes of 10 and 100 s, respectively. The results of these tests are described and discussed in [5]. The loading cycles were always fully reversed (i.e. R=-1) so that the maximum strain in the tension side of the cycle was the same as the maximum strain on the compression side of the cycle. Time taken to reach from pull to push (i.e. tension to compression) was 50s both in Test No.3 and 4, giving the strain rate of $1x10^{-4}\ s^{-1}$ and $1.4x10^{-4}\ s^{-1,}$ respectively, during the loading period. A holdtime of 10s was implemented both in the tension and compression sides of the fatigue cycles both in Test No. 3 and Test No. 4. Thus, the total time spent in each creep-fatigue cycle was 120s both in Test No. 3 and 4. The displacement dose per cycle was $5.3x10^{-6}$ dpa and $4.6x10^{-6}$ dpa, in the Test No. 3 and Test No. 4, respectively. All the relevant test parameters used in these tests are summarized in Table 1. During the creep-fatigue tests the evolution of the stress and strain in the specimen was continuously recorded. The stress acting on the specimen was determined from the measured value of the gas pressure in the bellows and the magnitude of the strain was obtained from the measured value of extension of the gauge length of the specimen by LVDT sensors attached to the specimen.

Table 1. Test parameters for in-reactor creep-fatigue experiments in BR-2 reactor

Test Parameters	Test No.3	Test No.4
Strain amplitude (%)	0.25%	0.35%
Holdtime in tension and compression (s)	10	10
Total time per complete cycle (s)	120	120
Neutron flux (n/m^2s (E>1 MeV))	$4.7x10^{17}$	4.1x1017
Damage rate (dpa/s)	$4.44x10^{-8}$	3.86x10-8
Irradiation and test temperature (K)	326±1	323±1
Displacement dose at the start of creep-fatigue test (dpa)	$2.5x10^{-5}$	1.7x10-5
Displacement dose per cycle (dpa)	$5.3x10^{-6}$	4.6x10-6
Displacement dose during deformation (dpa)	0.048	0.032
Total displacement dose experienced by the specimen (dpa)	0.048	0.048

2.4 Out-of-reactor creep-fatigue tests

A number of creep-fatigue tests were performed out side of a reactor (i.e. in the absence of neutron flux) on the reference material CuCrZr (HT1) ally in the unirradiated condition. These tests were carried out using the test modules and other testing facilities identical to the ones used in the case of in-reactor tests. It was deemed necessary to carry out these tests so that meaningful comparison could be made between the results of these tests and the results of the in-reactor tests. Furthermore, without such out-of-reactor reference tests, the effect of irradiation on the creep-fatigue lifetime could not be identified. It was therefore decided to carry out out-of-reactor tests at 353K at strain amplitudes of 0.3, 0.4 and 0.5 % with a holdtime of 10s.

In addition to the regular creep-fatigue tests to failure, a number of "interrupted" creep-fatigue tests were performed on the prime aged CuCrZr (PA) alloy at room temperature (295K) in a conventional Instron machine. Tests were conducted in a strain controlled mode with strain amplitude of 0.2 % and with a tension and compression holdtime of 10s. The main purpose of these interrupted tests was to determine the evolution of the maximum stress amplitude and the corresponding dislocation microstructure as a function of the number of cycles. Therefore a number of unirradiated specimens were tested for 1, 2, 5, 100 and 500 cycle. After a given number of cycles, the test was terminated and the specimen was taken out for the microstructural investigations.

2.5 Microstructural investigations

The microstructure of CuCrZr alloy used in the present creep-fatigue experiments was investigated in the following states: (a) unirradiated and undeformed, (b) unirradiated and deformed, (c) irradiated and undeformed and (d) irradiated and deformed concurrently with strain amplitudes of 0.25 % (Test No. 3) and 0.35 % (Test No. 4) with a holdtime of 10s. For the transmission electron microscopy (TEM) examinations, 3mm diameter discs were obtained from the gauge length of the creep-fatigue specimens. In cases where specimens broke during the test, the disc was taken from the portion of the gauge length closest to the fractured surface. Prior to electro polishing, the discs were mechanically polished down to a thickness of ~120µm. Thin foils for TEM investigations were prepared by twin-jet electro polishing in a solution of 25 % phosphoric acid, 25 % ethylene glycol and 50 % water at 9.5 V for about 2 min. at ~293K. Thin foils were examined in a JEOL 2000FX transmission electron microscope.

3. Experimental Results

Before describing the results on microstructural evolution and deformation behaviour during the in-reactor creep-fatigue tests, it is crucially important to point out that the experimental conditions during these tests are fundamentally different from those existing during the conventional out-of-reactor tests on the unirradiated or post-irradiated materials. In the conventional tests both the microstructural evolution and the deformation behaviour are entirely dependent on the stress-induced generation of dislocations, their interactions with each other and with other microstructural features such as precipitates, particles and irradiation- induced defect clusters that may provide resistance to dislocation motion. This relatively simple situation is, however, likely to get significantly complicated during the in-reactor deformation experiments. The complications would arise due to the fact that during the in-reactor experiments the generation of stress-induced dislocations and neutron-induced defects and defect clusters would occur concurrently. Consequently, the mobile defects and their clusters would interact with both the grown-in and freshly generated dislocations, modifying the intrinsic dislocation mobility and kinetics of dislocation–dislocation interactions. In addition, the production and accumulation of sessile defect clusters (e.g. loops and stacking fault tetrahedra) would present additional obstacles to dislocation motion.

In view of these complications, caution should be exercised in assessing the results of the in-reactor experiments and comparing them with the results of out-of-reactor conventional deformation experiments. Furthermore, in order to be able to rationalize the results of the in-reactor experiments and to be able to draw some reasonable conclusions from the results, it is important to determine details of the microstructure and deformation behaviour of the unirradiated reference material in the undeformed and deformed (out-of-reactor) states. Therefore, these details are presented in the following subsection (subsection 3.1) before describing the results on the in-reactor deformation behaviour and the resulting microstructure at the end of the in-reactor creep-fatigue tests (subsection 3.2).

3.1 Microstructure and deformation behaviour during out-of-reactor creep-fatigue tests

The dominating feature of the microstructure of the overegged CuCrZr (HT 1) alloy (in unirradiated and undeformed condition) as revealed by the TEM investigation was the presence of a relatively high density of small Cr-rich precipitates. The spatial distribution of these precipitates was found to be fairly homogenous throughout the whole grains and there were no significant variations in the size and density of precipitates from grain to grain. Figure 3 shows the general feature of the precipitate microstructure in the unirradiated and undeformed CuCrZr (HT1) alloy. The average precipitate size and density in the overaged and unirradiated CuCrZr (HT1) alloy were found to be 8.7 nm and 1.7 x 10^{22} m^{-3}, respectively [8].

Prior to carrying out in-reactor creep-fatigue tests, the creep-fatigue deformation behaviour of the unirradiated CuCrZr (HT 1) alloy was determined by performing out-of-reactor tests at 353K. These tests were carried out using the same specimen size and geometry and the same test parameters as used in the in-reactor creep-fatigue tests (section 2.3). Figure 4(a) shows the measured temporal evolution of the strain and stress response of the specimen during the first five cycles of the test carried out with a strain amplitude of 0.3 % and with a holdtime of 10s both in the tension and compression sides of the cycles. It should be noted that as expected the strain level during the holdtime remains constant (at 0.3 %) since the test is carried out in the strain controlled mode. The maximum stress level (both in tension and compression) does not change much either with the number of cycles. This means that the material does not harden much during the first few cycles of the creep-fatigue test. Figure 4 (b) shows the cyclic deformation behavior in the form of hysteresis loops illustrating the dynamic stress-strain relationship during the creep-fatigue test described in Figure 4 (a). These loops describe the deformation behaviour during those first five cycles of the creep-fatigue test shown in Figure 4 (a). The stability and the symmetry of the hysteresis loops shown in Figure 4 (b) confirm the true cyclic nature of deformation during the creep-fatigue test. The results of the out-of-reactor creep-fatigue test performed with strain amplitude of 0.4 % at 353K with a holdtime of 10s are shown in Figure 5. Figure 5 (a) shows the measured temporal evolution of the stress and strain response of the specimen during the first five cycles of the test. The corresponding hysteresis loops illustrating the dynamic stress-strain relationship during the creep-fatigue test is shown Figure 5 (b). The results confirm the true cyclic nature of deformation during the test.

In order to illustrate the stability of the cyclic nature of deformation throughout the whole creep-fatigue test, a number of hysteresis loops for the higher number of creep-fatigue cycles representing increasingly higher level of deformation are shown in Figure 6, for strain amplitudes of (a) 0.3 % and (b) 0.4 % and (c) 0.5 %. The position and shape of the loops indicate that the cyclic deformation remains stable during the first 500 cycles of the test and then becomes clearly unstable beyond about 1000 cycles. For comparison the hysteresis loops for creep-fatigue test carried out at 353K with a strain amplitude of 0.5 % and a holdtime of 10s are shown in Figure 6 (c).

The average value of the maximum stress reached in the tension and compression sides of a deformation cycle (i.e. the maximum stress amplitude) is plotted in Figure 7 as a function of number of cycles during the out-of - reactor creep-fatigue test at 353K with strain amplitudes of (a) 0.3 % and (b) 0.4 % and with a holdtime of 10s. It can be seen in Figure 7 that the material hardens during the first 50 – 60 cycles of the creep-fatigue test. The hardening seems to saturate at about 100 cycles and remains saturated until towards the end of the test. For comparison the results of 0.5 % strain amplitudes are also shown in Figure 7. It should be noted that the magnitude of cycle hardening at all three strain amplitudes is rather small.

The post-deformation microstructure of the CuCrZr (HT1) specimen out-of-reactor creep-fatigue tested at 353K was investigated using TEM. The TEM discs were obtained from the region close to the fractured surface. The TEM examination showed that the post-deformation microstructure was quite uniform throughout the whole volume examined and was composed of precipitates, dislocation segments and some relatively small loops (Figure 8). Most of the dislocation segments were rather short in length with varying degree of curvature. The microstructure showed no indication of build-up of dislocation density in the form of dislocation walls.

As described in section 2.4, a number of out-of-reactors interrupted creep-fatigue tests were carried out on the prime aged CuCrZr (PA) alloy at

295 K with strain amplitude of 0.2 % and a holdtime of 10s. The variation of the maximum stress amplitude measured in these tests as a function of the number of creep-fatigue cycles is illustrated in Figure 9. The results show that a modest amount of hardening continues up to about 500 cycles without showing any sign of saturation in the hardening level.

Thin foils prepared from these creep-fatigue tested specimens were investigated in a transmission electron microscope. Figure 10 (a-d) shows the representative micrographs for specimens tested to (a) 1, (b) 5, (c) 100 and (d) 500 creep-fatigue cycles. The micrographs show the presence of a high density (2.6×10^{23} m^{-3}) of small (2.2 nm) precipitates [6] and a relatively low density of dislocation segments. It can be seen that the density of deformation induced dislocations increases with increasing number of creep-fatigue cycles (Figure 10(a-d)). The micrographs shown in Figure 10 (a-d) suggest that as dislocation density increases, the frequency of dislocation–dislocation interactions increases. However, even after 500 cycles (Figure 10(d)) there is no indication of dislocation segregation in the form of cells and dislocation walls. The fact that the dislocation density increases with increasing number of cycles is consistent with the increase in hardening with increasing number of cycles (Figure 9).

3.2 In-reactor creep-fatigue deformation behaviour

3.2.1 Mechanical response

The measured temporal evolution of strain and stress response of the specimen during the first five cycles of the in-reactor creep-fatigue test on CuCrZr (HT1) specimen at 326K with a strain amplitude of 0.25 % and a holdtime of 10s (Test No.3) is shown in Figure 11(a). The test parameters used in the experiments are quoted in Table 1. The results shown in Figure 11(a) suggest that the peak values of the measured stress levels both in the tension and compression sides of the cycle do not increase much with increasing number of cycles. The strain level, on the other hand, remains constant during each cycle simply because the test is performed in the strain-controlled mode. It should be noted that no significant amount of stress relaxation occurs during the holdtime period of 10s neither in the tension nor in the compression side of the loading cycles. The cyclic deformation behaviour in the form of hysteresis loops is shown in Figure 11(b) illustrating the dynamic stress-strain relationship during the creep-fatigue Test No.3. The loops shown in Figure 11(b) describe the deformation behaviour during those first five cycles of the creep-fatigue test shown in Figures 11(a). The stability and the symmetry of the hysteresis loops shown in Figure 11(b) confirm that during the in-reactor test the true cyclic nature of deformation is achieved and maintained.

Figure 12 (a) shows the results of the in-reactor creep-fatigue Test No. 4 carried out on CuCrZr (HT1) specimen with a strain amplitude of 0.35% with a holdtime of 10s. The irradiation and test temperature in this case was 323K. The temporal evolution of strain and stress response during this test is shown in Figure 12(a) for the first five cycles of the cyclic loading. Just like in the case of Test No.3 (Figure 11(a)), the measured peak stress level both in the tension and compression sides of the cycle does not increase much with increasing number of cycles. The measured stress response (Figure 12(a)) does not show any significant amount of stress relaxation during the holdtime. The dynamic stress-strain relationship during the cyclic loading for the first five cycles during the Test No.4 is shown in Figure 12(b) in the form of hysteresis loops. The stability and the continued symmetry of these hysteresis loops confirm once again that during the in-reactor creep-fatigue test (Test No.4) a proper cyclic nature of deformation is achieved and maintained during the test.

Since both Test No.3 and 4 were carried out at strain amplitudes of 0.25 % and 0.35 % until the end of life of specimens (see section 2.3), a number of hysteresis loops for the higher number of creep-fatigue cycles during Test No.3 and 4 are shown in Figure 13. The position and the symmetry of hysteresis loops clearly show that a proper cyclic nature of deformation is maintained during the whole test period of the in-reactor Test No.3 and 4. For comparison, the results of the in-reactor test carried out with strain amplitude of 0.5 % at 363K are shown in Figure 13 (c) [5].

The variation of the maximum stress amplitude measured in the in-reactor tests (Test No.3 and 4) is shown in Figure 14 as a function of number of cycles (Figure 14(a)) and displacement dose level (Figure 14(b)). The maximum stress amplitude is the average value of the peak stress reached in the tension and compression sides of a loading cycle. For comparison purposes, the results of in-reactor creep-fatigue test carried out at 363K with a strain amplitude of 0.5% and a holdtime of 10s are also shown in Figures 14(a) and 14(b). The following features of the results shown in Figure 14(a) are worth noting: (a) the material hardens with the number of cycle faster during in-reactor tests(Test No.3 and 4) than during the out-of-reactor test, (b) the level of hardening during the in-reactor tests(Test No. 3 and 4) is higher than that during the out-of-reactor test, (c) the rate of hardening per cycle during the in-reactor tests is somewhat higher than that during the out-of-reactor tests. The hardening comes to saturate between 100 and 300 cycles during the in-reactor tests as well as during the out-of-reactor test and (d) the specimens during the in-reactor tests (No. 3 and 4) begin to show sign of softening already beyond about 100 cycles whereas in the case of the out-of-reactor test the onset of softening does not occur until beyond about 1000 cycles.

In order to examine the impact of continuous production of defects and their clusters by fission neutrons on the mechanical response of the specimens during creep-fatigue cyclic loading, the measured maximum stress amplitude is plotted against displacement dose in Figure 14(b) for Test No.3 and 4. The results of the in-reactor Test No. 1 with 0.5 % strain amplitude [5] at 363K are also included in Figure 14 (b). The main features of the hardening behaviour illustrated by the results presented in Figure 14 (b) is naturally very similar to that in Figure 14(a) since the number of creep-fatigue cycles and the accumulated displacement dose level are directly related. It is interesting to note, however, that the hardening levels both in Test No. 3 and Test No. 4 saturate at a rather low level of displacement dose (i.e. between 1 x 10^{-3} and 2 x 10^{-3} dpa). This means that the damage accumulation beyond the dose level of about 10^{-3} dpa does not seem to affect the mechanical response of the material during the in-reactor creep-fatigue tests.

All the results on creep-fatigue lifetime measured as a function of strain amplitudes during out-of-reactor as well as during in-reactor tests are summarised in Figure 15. The results clearly demonstrate that the number of cycles to failure (N_f) (i.e. the lifetime) decreases with increasing strain amplitudes. The numerical values of the lifetime data presented in Figure 15 are quoted in Table 2.

Table 2. Number of Cycles to failure (N_f) for CuCrZr (HT1) alloy creep-fatigue tested out-of-reactor and in-reactor at different strain amplitudes

Material	Dose (dpa)	Irradiation and test temperature (K)	Hold time (s)	Strain amplitude (%)	Cycles to failure (N_f)
CuCrZr HT1	0.016	363	10	0.5	2270
CuCrZr HT1	0.054	343	100	0.5	2553
CuCrZr HT1	0.032	323	10	0.35	6861
CuCrZr HT1	0.048	326	10	0.25	8926*
CuCrZr HT1	-	353	100	0.5	1636
CuCrZr HT1	-	353	10	0.4	3291
CuCrZr HT1	-	353	10	0.3	7341

* terminated at the end of the reactor period

Photographs of the specimens used in the in-reactor creep-fatigue (a) Test No. 3 and (b) Test No.4 are shown in Figure 16. It should be noted that these specimens did not break into pieces during the in reactor tests. It is also worth mentioning that these specimens did not show any sign of necking during the test. However, the specimen tested at the strain amplitude of 0.35 % (Test No. 4) did fracture (see Figure 14 and 16 (b)) during the test. The crack in the specimen can be clearly seen in Figure 16 (b). In the specimen tested with the strain amplitude of 0.25 % there was no sign of crack on the surface of the specimen (Figure 16(a)). It should be noted that this test was terminated at the end of the reactor period.

3.2.2 Microstructure of in-reactor deformed specimens

To help understand the mechanical response of CuCrZr (HT1) alloy during in-reactor creep-fatigue tests reported in the preceeding section (3.2.1), the microstructure of the specimens tested with strain amplitudes of 0.25 % (Test No.3) and 0.35 % (Test No. 4) was investigated using transmission electron microscopy technique. It is worth noting here, however, that the results of these investigations provide information about the microstructure only at the end of the in-reactor tests. In other words, the nature of the microstructural evolution as a function of the number of creep-fatigue cycles or the increasing level of displacement dose remains unknown. In spite of this limitation, however, the details of the microstructure even at the end of the in-reactor tests provide very valuable information about physical processes operating during cyclic deformation. In this section we first report the defect microstructure accumulated in the CuCrZr (HT1) specimen during irradiation alone, i.e. in the absence of concurrent plastic deformation. This is followed by the results on various aspects of the microstructure that has survived in the specimens at the end of the in-reactor Test No. 3 and Test No. 4 with strain amplitudes of 0.25 % and 0.35 %, respectively. Results of similar investigations on specimens tested in the in-reactor Test No. 1 and 2, with a strain amplitude of 0.5 % are described in Ref.[5].

In order to establish the impact of deformation induced mobile dislocations on the accumulation of defect clusters produced concurrently during in-reactor creep-fatigue tests, it is crucially important to determine the damage accumulation in the absence of dislocation generation (i.e. without plastic deformation). To achieve this goal, TEM discs were taken from the end of the "head" of the creep-fatigue specimen used in the in-reactor Test No. 3. Since the diameter of the specimen "head" is 3 times larger than the gauge diameter (see Figure 1a), the stress acting on this part of the specimen will be only one-ninth of the stress experienced by the gauge section of the specimen. Thus, the discs used for TEM investigations did not experience at any time during the in-reactor Test No. 3 stress beyond the yield stress of the material [5]. Hence the damage accumulation observed in these discs may be considered to have occurred in the absence of deformation-induced mobile dislocations. It should be noted here that the irradiation conditions (i.e. damage rate, irradiation temperature and displacement dose level) for these TEM discs, on the other hand, were identical to those experienced by the material of the gauge section of the specimen used in the in-reactor Test No. 3.

The weak beam dark field image obtained from the irradiated but undeformed specimen used in the Test No. 3 is shown in Figure 17. It should be noted that the specimen was irradiated at 326K (Test No. 3) to a displacement dose level of 0.048 dpa. The micrograph illustrates that the irradiation induced defect clusters have evolved in a homogeneous fashion. As described in [5], the cluster population shown in Figure 17 are most likely to be a mixture of small interstitial loops and staking fault tetrahedra. Note that the microstructure also contains a relatively high density of rather small precipitates [5]. Figures 18 and 19 shows the weak beam dark field images of the in-reactor specimens tested with strain amplitudes of 0.25 % (Test No. 3) and 0.35 % (Test No. 4), respectively, at 326K and 323K. The microstructures shown in Figure 18 and 19 represent the end of life microstructures at a dose level of 0.048 dpa. These figures illustrate that a high density of irradiation induced defect clusters in the forms of small interstitial loops and stacking fault tetrahedral accumulate during the creep-fatigue deformation and in a homogeneous fastion. It is, on the other hand, rather surprising that these specimens creep-fatigue tested with amplitudes of 0.25 % and 0.35 % did not show the presence of any significant number of deformation–induced dislocations.

It is interesting that the examination of thin foils obtained from specimens tested both with strain amplitudes of 0.25 % (Test No. 3) and 0.35 % (Test No. 4) revealed the evidence for localized deformation in the form of relatively wide bands. These bands have the appearance as well as characteristic features of “cleared channels” commonly observed in specimens tensile tested either in-reactor or post-irradiation out-of-reactor tests [9, 10]. Since the channels observed in the present experiments contain some residual defect clusters, loops and the precipitates, these channels have been called “diffuse” cleared channels (DCCs) [5]. Figure 20 shows an example of DCCs observed in the specimen tested with the strain amplitude of 0.25 % and a holdtime of 10s. It can be seen that the channels seem to have eminated from a grain boundary, are fairly wide and are widely spaced. Figure 21(a) shows an example of DCCs eminating from the incoherent part of a twin boundary. An enlarged view of a portion of the DCC in Figure 21(a) (marked by a box) is shown in Figure 21 (b) illustrating the presence of some loops, a few dislocation segments and finely distributed small precipitates. Figure 22 shows the evidence for the presence of DCCs in the specimen (in-reactor) creep-fatigue tested with a strain amplitude of 0.35 % and a holdtime of 10s (Test No. 4). Two sets of DDCs can be clearly seen in Figure 22. The DCCs are almost a micron wide. It is interesting to note that the DCCs appear to have a fairly regular spatial distribution and seem to have eminated from a twin boundary. It should be mentioned that DCCs have been observed also in the in-reactor creep-fatigue tested specimens with strain amplitude of 0.5 % both with 10s and 100s holdtime [5].

4. Discussion

A comparison of the results shown in Figure 7 for the out-of-reactor tests with that of the in-reactor tests shown in Figure 14 clearly demonstrates that the general trend of the evolution of the stress amplitude with the increasing number of creep-fatigue cycles is very similar in the two cases. However, it is also very clear that the level of stress amplitude for the whole test period increases with the strain amplitude both during out-of-reactor and in-reactor tests. It is interesting to note here that the rate of hardening with the number of creep-fatigue cycles is very similar during out-of-reactor and in-reactor tests. This implies that under during the in-reactor creep-fatigue tests under the conditions of the present experiments the irradiation induced defects and their clusters do not seem to play any significant role in the evolution of the stress amplitude as a function of the number of creep-fatigue cycles. This is very surprising particularly in view of the fact that during these in-reactor tests a high density of defect clusters accumulates (see Figures 17-19). At the strain amplitude of 0.5 %, for example, the density of stacking fault tetrahedra at the end of the in-reactor creep-fatigue test has been found to be 3.4×10^{23} m^{-3} [5].

The comparison of results shown in Figure 7 and Figure 14 also suggests that the maximum stress amplitude in the case of in-reactor tests is higher than that in the case of out-of-reactor tests for all three strain amplitudes. It is interesting to note that this increase due to irradiation occurs already in the first cycle of the test and the increase is maintained at that level throughout the whole test. This increase in the initial stress amplitude is most likely to occur partly due to dislocations produced during the first cycle of the test and partly due to defect accumulation during the time period prior to initiating the test. As shown in Table 1, the accumulated displacement dose during this period is about 1.7 - 3.7×10^{-5} dpa. At this dose level and under these irradiation conditions, a cluster density of about 5×10^{21} m^{-3} is likely to be formed in pure copper [11]. In addition, during this period, the grown-in dislocations are likely to get lightly decorated by the gliding interstitial loops produced directly in the displacement cascades. Both of these factors are likely to contribute to the increase in the maximum stress amplitudes [12, 13].

In order to estimate the role of dislocations produced during creep-fatigue cycles in controlling the evolution of the maximum stress amplitude, a number if interrupted creep-fatigue tests were carried out on the prime aged CuCrZr (PA) alloy with a strain amplitude of 0.2 %. The tests were carried out at 295K. The evolution of the maximum stress amplitude is shown in Figure 9 as a function of the number of creep-fatigue cycles. The corresponding dislocation microstructure is shown Figure 10 (a-d) for specimens deformed for 1, 5, 100 and 500 cycles. As can be seen in Figure 10, the dislocation density increases with increasing number of cycles. Thus, the increases in the maximum stress amplitude with the number of creep-fatigue cycles seen in Figure 9 can be taken to be due to the increase in dislocation density (Figure 10). The interrupted tests carried out on the same CuCrZr (PA) alloy with the strain amplitude of 0.5 % has shown [3] the generation of very high dislocation density already during first creep-fatigue cycle (Figure 16 (a) in [3]). The maximum stress amplitude already during the first cycles is also much higher than in the case of Figure 9 for the 0.2 % strain amplitude. Thus, it is reasonable to conclude that the variation in the level of the maximum stress amplitude with increasing strain amplitudes in Figure 7 and 14 is most probably due to increase in the dislocation density at higher strain amplitude at a given number of cycle.

As mentioned earlier, the irradiation does cause some additional hardening during the first 100 to 200 cycles. However, the most potent effect of irradiation during creep-fatigue tests appears to be on the dynamic recovery and the cyclic softening during the in-reactor tests. During these creep-fatigue tests with three different strain amplitudes most of the lifetime of these specimens is dominated by the processes of recovery and softening. This implies that all the dislocations produced during the first 100 to 200 cycles are somehow slowly lost by some recovery process that remains unknown to us at present. Note that practically no dislocations were found neither in the specimens used in the out-of-reactor tests nor used in the in-reactor tests carried out at different strain amplitudes.

Another feature of the microstructure of the in-reactor tested specimens which is worth commenting upon is the presence of the so called diffuse cleared channels(DCCs).Generally, it is thought that the localized deformation in these channels may be responsible for causing the reduction in the creep-fatigue lifetime by assisting nucleation and growth of cracks. The present results show that if anything, the lifetime during the in-reactor tests is longer than that during the out-of-reactor tests where no channels are formed (see Figure 15). At present, we are unable to provide a sound explanation for these results.

5. Summary and Conclusions

The present work is an extension of an earlier work described and discussed recently in [5]. The main objective of this series of investigations was to study the creep-fatigue deformation behaviour of a precipitation hardened CuCrZr alloy presently considered to be a candidate material for its application in the first wall and divertor components of ITER. It was considered to be appropriate and realistic to study this problem by performing cyclic deformation experiments directly in a nuclear reactor where the effects of simultaneous application of stress and irradiation on deformation behaviour can be determined directly. Undoubtedly such experiments are complicated, time consuming and expensive. However, the present series of experiments have clearly demonstrated that such experiments can be successfully carried out and are very valuable form scientific as well as technological point of views.

In the earlier work the effect of holdtime on the creep-fatigue lifetime was investigated at strain amplitude of 0.5 % with holdtimes of 10s and 100s. The number of cycles to failure was found to be very similar in both cases. In the present work the effects of relatively low strain amplitudes of 0.25 % and 0.35 % with a holdtime of 10s on the deformation behaviour and lifetime have been studied. Both out-of-reactor and in-reactor creep-fatigue tests were carried out.

The cyclic hardening as a function of the number of creep-fatigue cycles in the out-of-reactor and in-reactor experiments is very similar at the strain amplitudes of 0.25 %, 0.35 % and 0.5 %. The cyclic hardening saturates at about the same number of cycles (e.g. 50 to 300) at all strain amplitudes both in the in-reactor and out-of-reactor experiments.

The level of maximum stress amplitude throughout the whole test period increases with increasing strain amplitude both in the out-of-reactor and in the in-reactor tests. The results of the interrupted creep-fatigue tests indicate that this increase occurs primarily due to deformation induced increase in the density of dislocations.

The rate of cyclic hardening per cycle is very similar for both the out-of-reactor and the in-reactor tests. This strongly suggests that the accumulation of the irradiation induced defects with increasing dose (i.e. increasing number of cycles) do not have any significant effect on the hardening rate during the in-reactor creep-fatigue tests. A closer look at the results shows, however, that the level of hardening is increased during the first cycle of the in-reactor creep-fatigue tests due to irradiation. Furthermore, this increase in hardening during the first cycle of the test is maintained throughout the whole test period.

The density of residual dislocations at the end of the creep-fatigue tests was found to be very low both in the out-of-reactor and in-reactor tested specimens at different strain amplitudes. No segregation of dislocations in the form of dislocation-cells and cell walls was observed. It should be pointed out that dislocations have been observed in the out-of-reactor interrupted creep-fatigue tests at 0.2 % strain amplitude (Figure 10) and 0.5 % strain amplitude (Figure 16 in [3]) but only during the early part of the tests (i.e. only lower number of cycles).

An interesting feature of the microstructure observed in the in-reactor tested specimens is the evidence of the flow localization in the form of the so called diffuse cleared channels (DCCs). It should be noted these channels are not observed in the out-of-reactor tested specimens. Generally, it is thought that the localized deformation in these channels may cause reduction in the creep-fatigue lifetime. However, the present results show just the opposite in that the lifetime during the in-reactor tests is longer than that during the out-of-reactor tests where no channels are formed. At present no clear explanation can be given for this observation.

Although the irradiation does not cause strong hardening during the in-reactor creep-fatigue tests, it seems to have very potent effect on the dynamine recovery and cyclic softening during the in-reactor creep-fatigue tests. It seems that the dislocations produced during the first 100 to 200 cycles are lost later at higher number of cycles by some recovery processes. These processes remain unknown to us at present.

Finally, it should be noted that the number of cycles to failure determined in the present in-reactor creep-fatigue tests at the strain amplitudes of 0.25 %, 0.35 % and 0.5 % is higher (by a factor of about two) than in the corresponding out-of-reactor tests. Unfortunately, the reason for this improvement in the creep-fatigue lifetime in the irradiation environment remains unknown at present.

Acknowledgements

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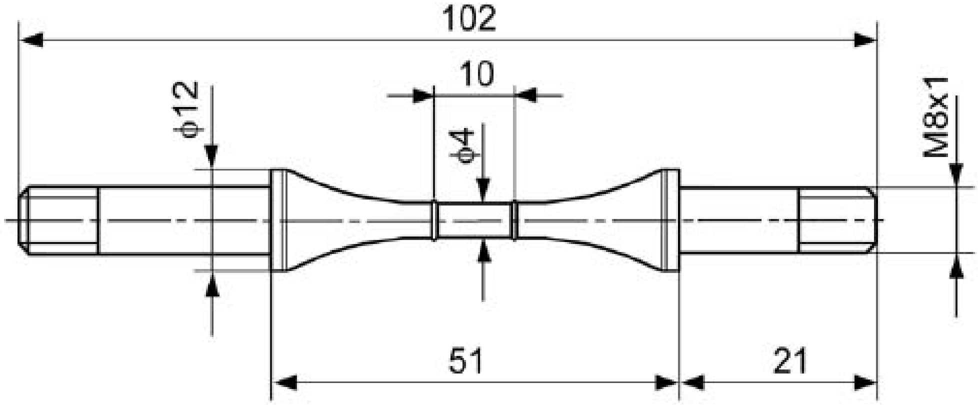
(a)



(b)

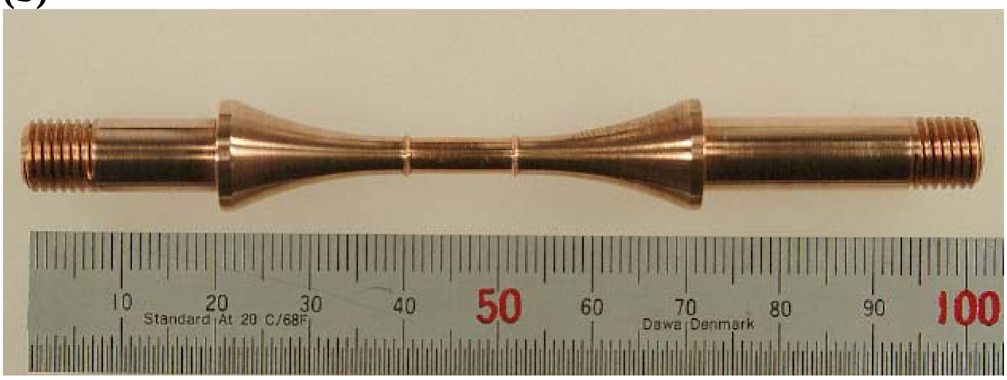
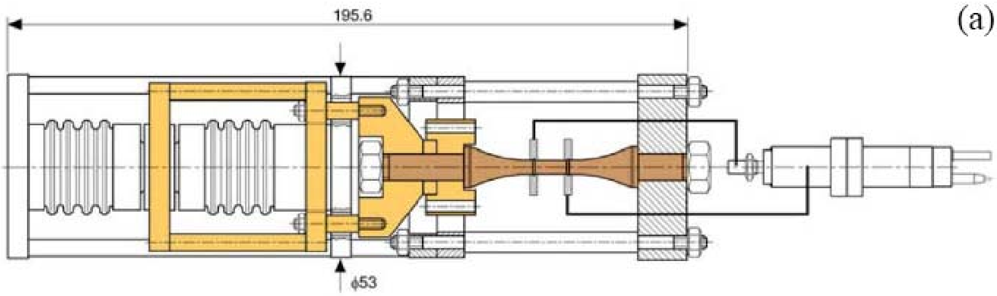


Figure 1. (a) Size and geometry of creep-fatigue specimens used in the out-of-reactor and in-reactor creep-fatigue tests and (b) a photograph of one of the creep-fatigue specimens used in these tests.

(a)



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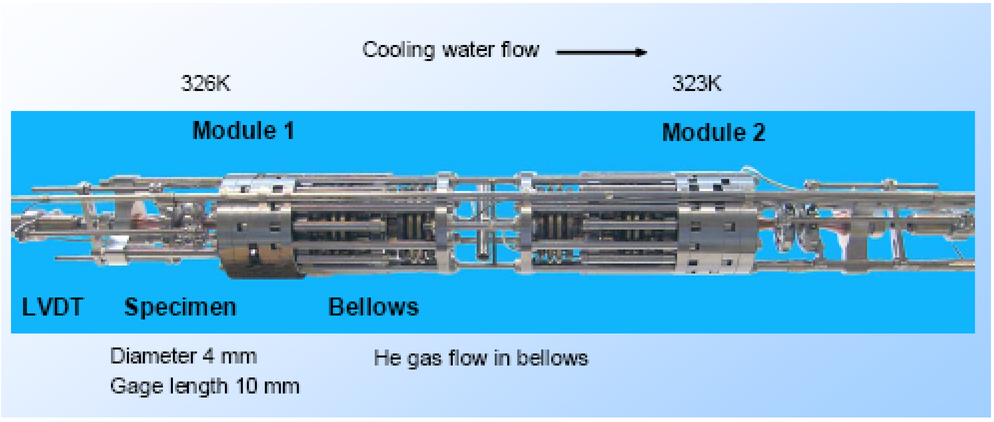

Figure 2. (a) Simplified layout and operational features of the test module and (b) the final assembly of the two complete test modules in the instrumented irradiation rig.

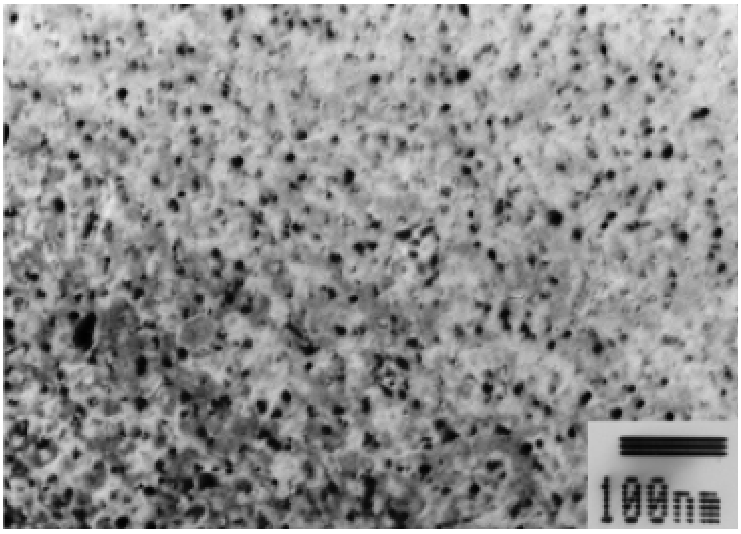

Figure 3. Transmission electron micrograph showing precipitate microstructure of the unirradiated and undeformed CuCrZr alloy in the overaged(HT1) condition.

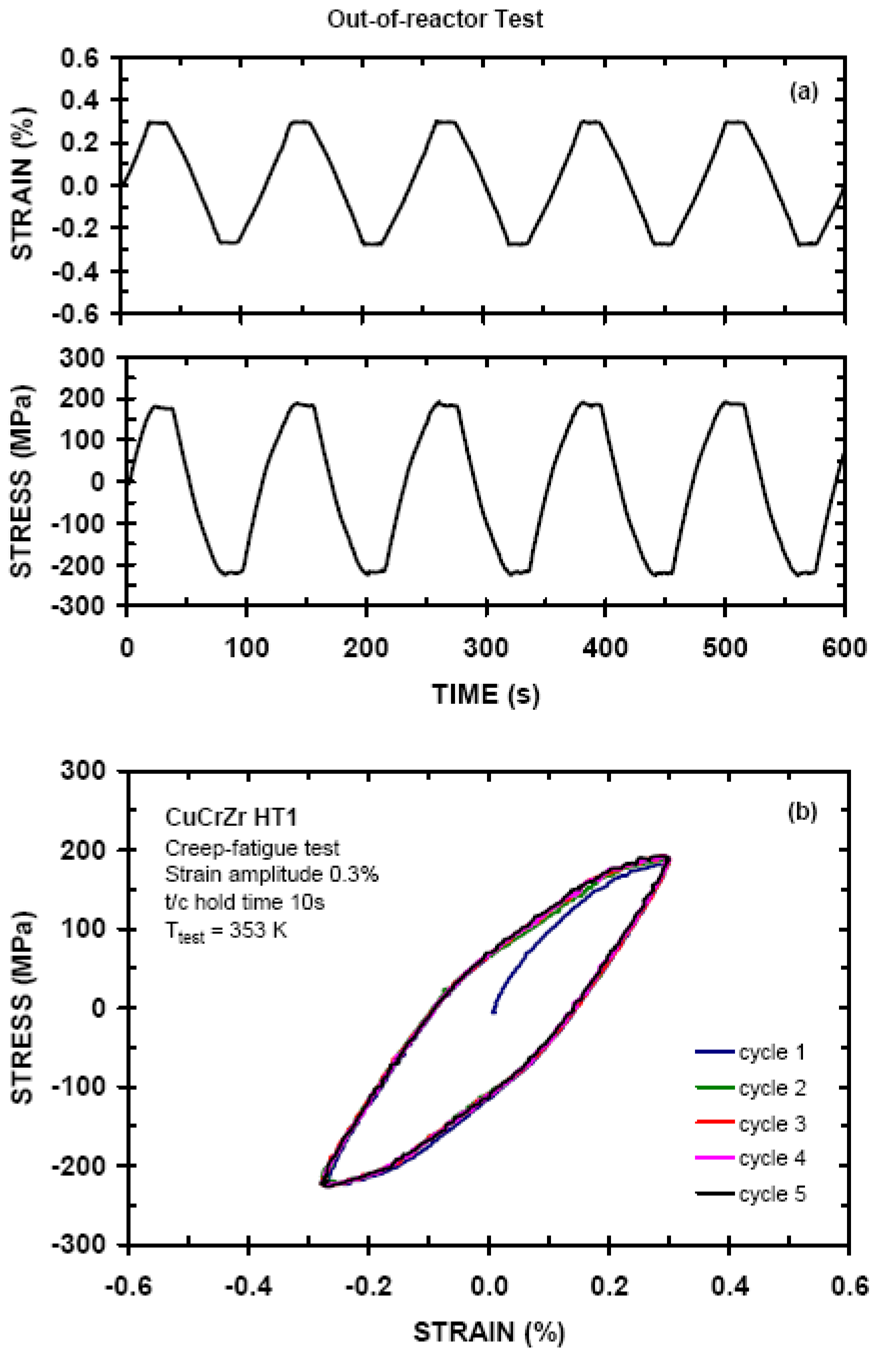

Figure 4. (a) Strain and stress response as a function of test time and (b) cyclic deformation behaviour in the form of hysteresis loops obtained during the first five cycles of the creep-fatigue test performed at 353K on the unirradiated overaged CuCrZr (HT1) alloy with a strain amplitude of 0.3 % and a holdtime of 10s both in tension and compression sides of the loading cycles.

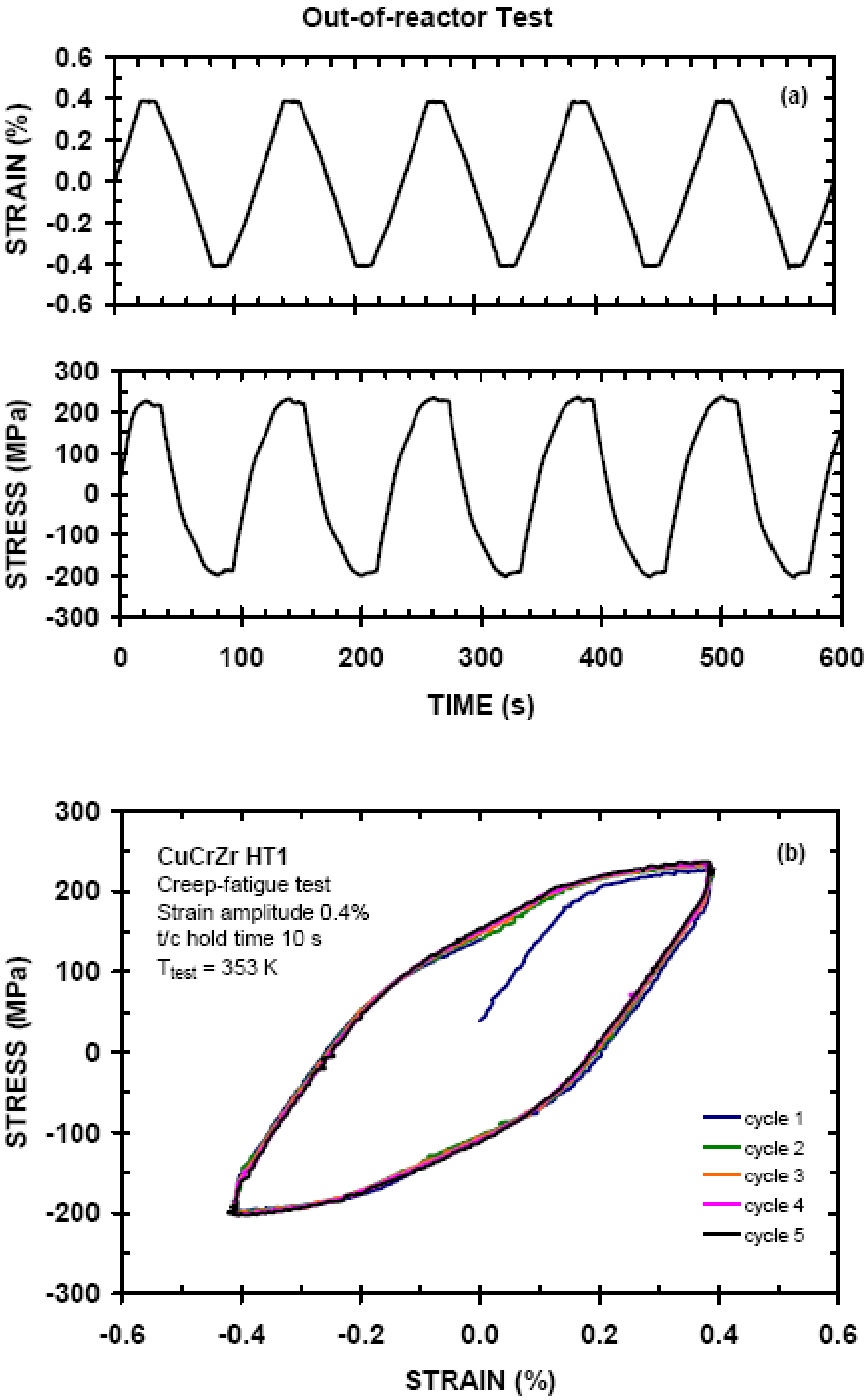

Figure 5. Same as in Figure 4, but for the test carried out with a strain amplitude of 0.4 %.

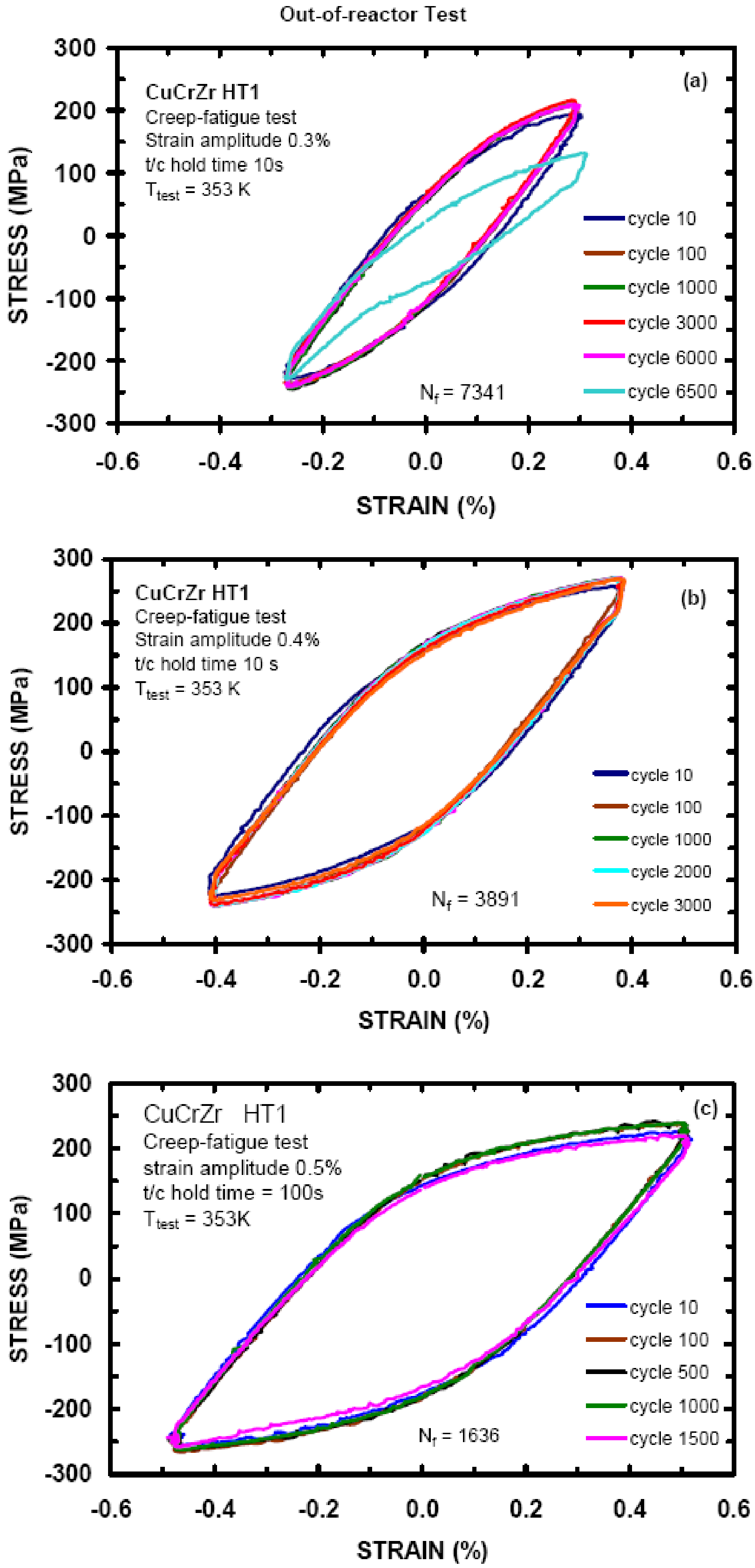

Figure 6. (Figure caption on page 28)

Figure 6. The cyclic deformation behaviour in the form of hysteresis loops obtained for the higher number of creep-fatigue cycles during the out-of-reactor tests carried out at 353K with strain amplitude of (a) 0.3 %, (b) 0.4 % and (c) 0.5 %. Note that the test with strain amplitudes of 0.3 % and 0.4 % were carried out with a holdtime of 10s (Figures 6 (a) and 6 (b)) where as the test with 0.5 % strain amplitude (Figure 6 (c)) was carried out with a holdtime of 100s.

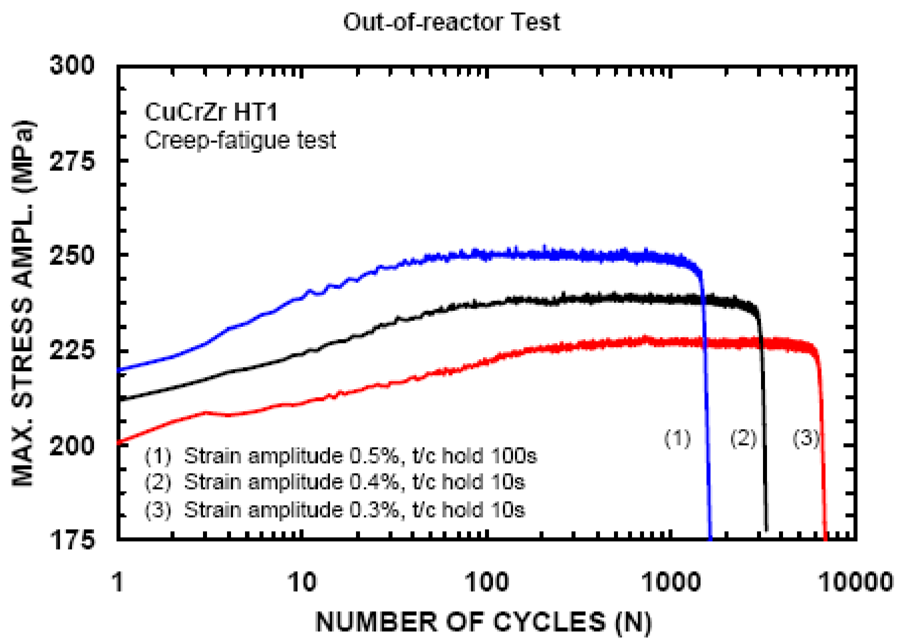

Figure 7. Variation of the maximum stress amplitudes with the number of creep-fatigue cycles during the out-of-reactor creep-fatigue tests carried out at 353K with different strain amplitudes.

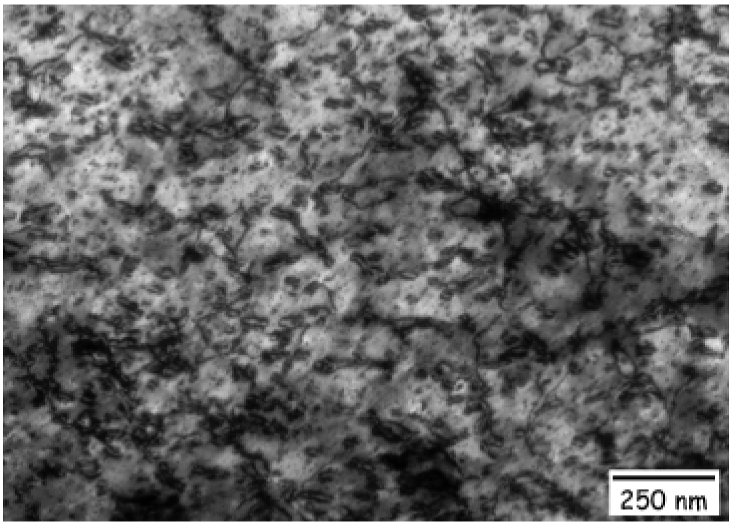

Figure 8. Post-deformation microstructure of CuCrZr (HT1) alloy after out-of-reactor creep-fatigue test in the unirradiated condition at 353K with a strain amplitude of 0.4 % and a holdtime of 10s.

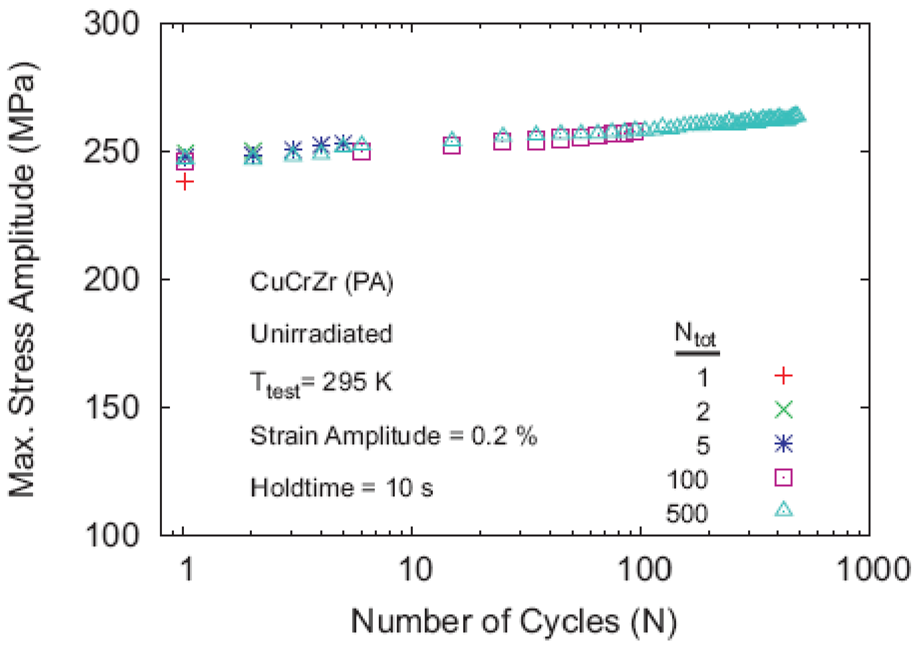

Figure 9. Variation of the maximum stress amplitude with the number of cycles during the interrupted out-of-reactor creep-fatigue testes carried out at 295K with a strain amplitude of 0.2 % and a holdtime of 10s. Note that the CuCrZr specimens used in these tests were in the prime aged (PA) condition. N_{tot} is the total number of cycles after which the tests were interrupted.

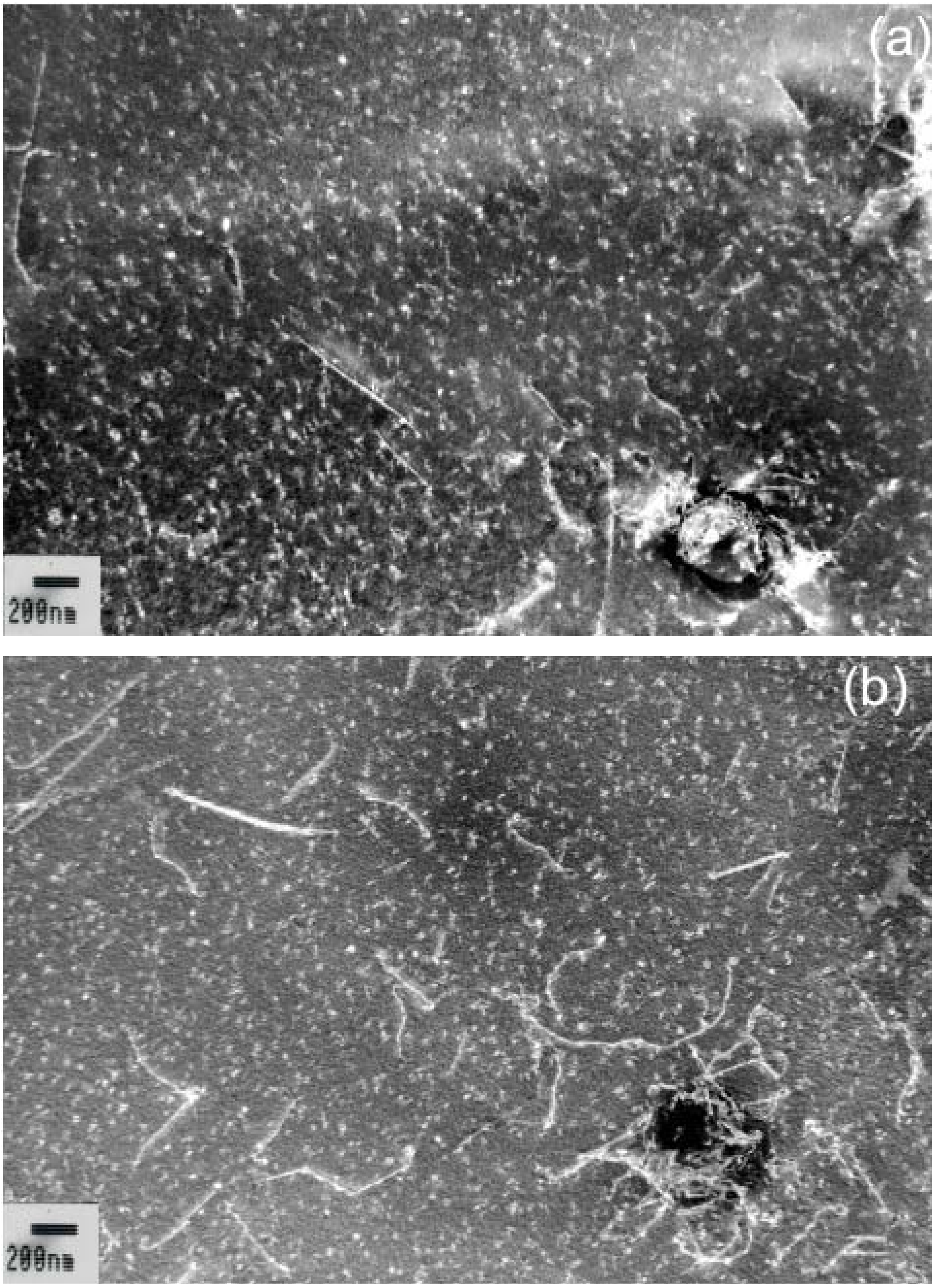

Figure 10.(continued)

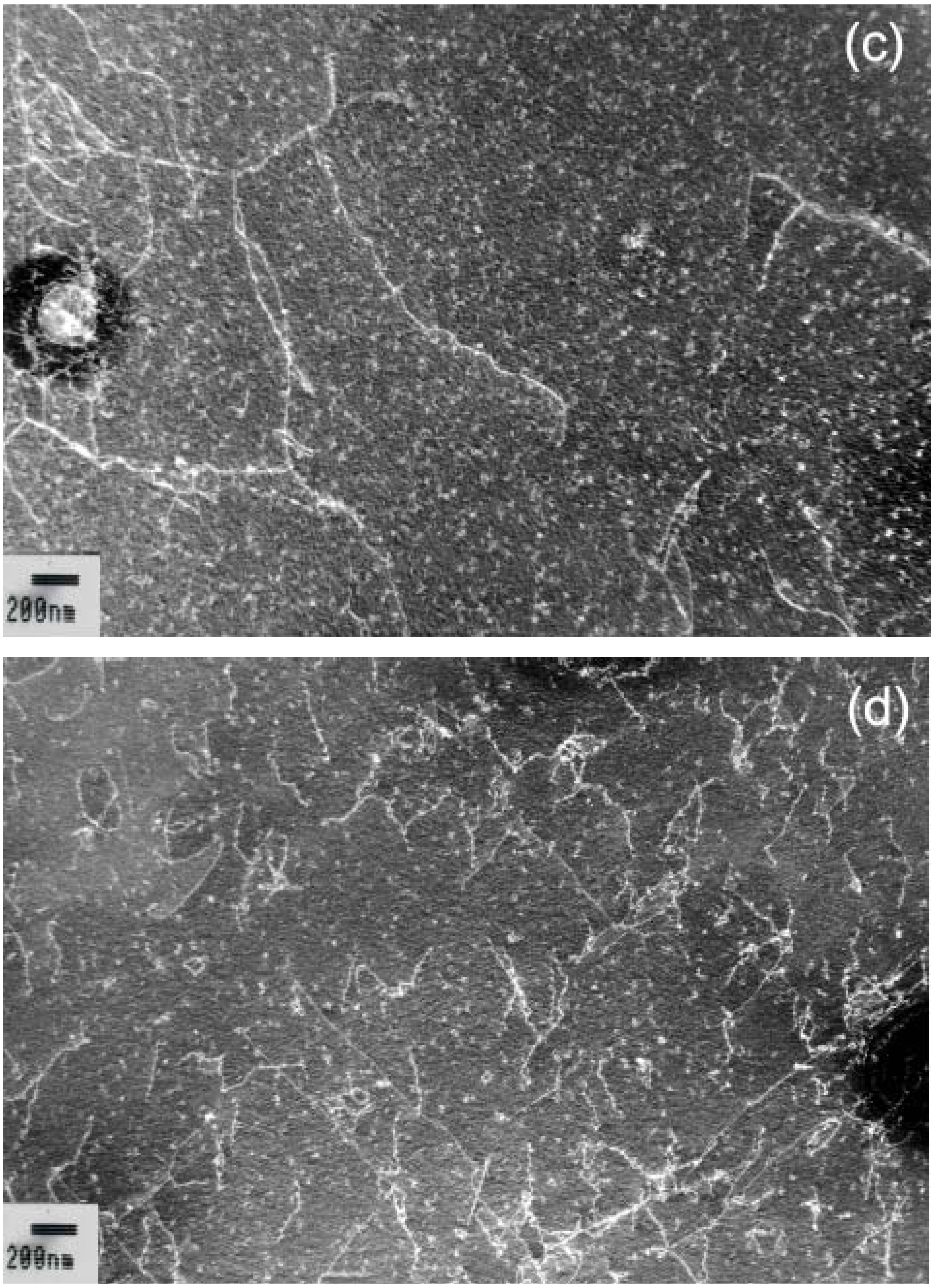

Figure 10. Post-deformation microstructure in the CuCrZr (PA) specimens tested to (a) 1, (b) 5, (c) 100 and (d) 500 cycles at 295K with a strain amplitude of 0.2 % and a holdtime of 10s. Note that dislocation density increases with increasing number of cycles.

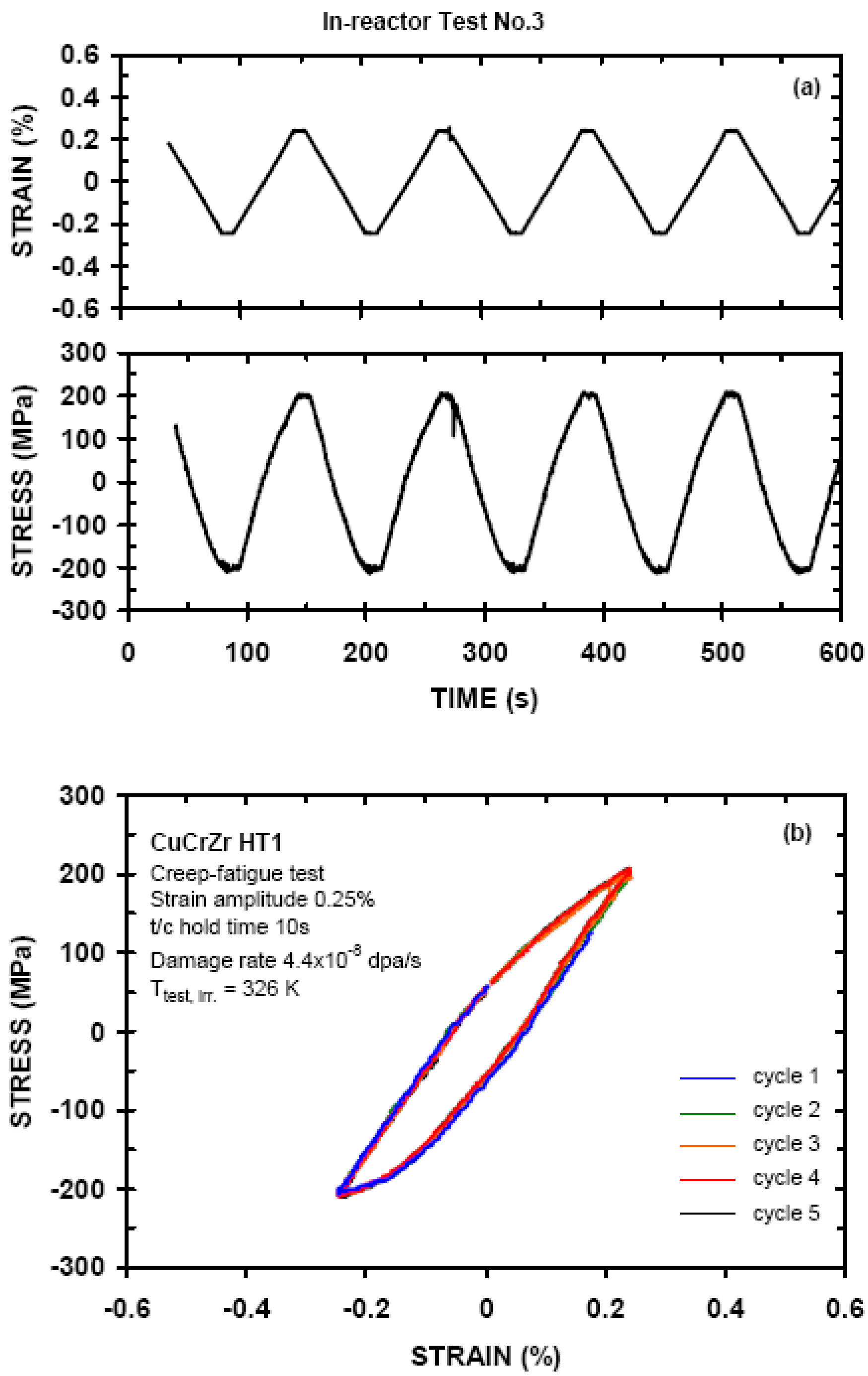

Figure 11 (a) Strain and stress response as a function of test time and (b) cyclic deformation behaviour in the form of hysteresis loops determined during the first five cycles of the in-reactor creep-fatigue test (Test No. 3) performed at 326K with a strain amplitude of 0.25 % and a holdtime of 10s.

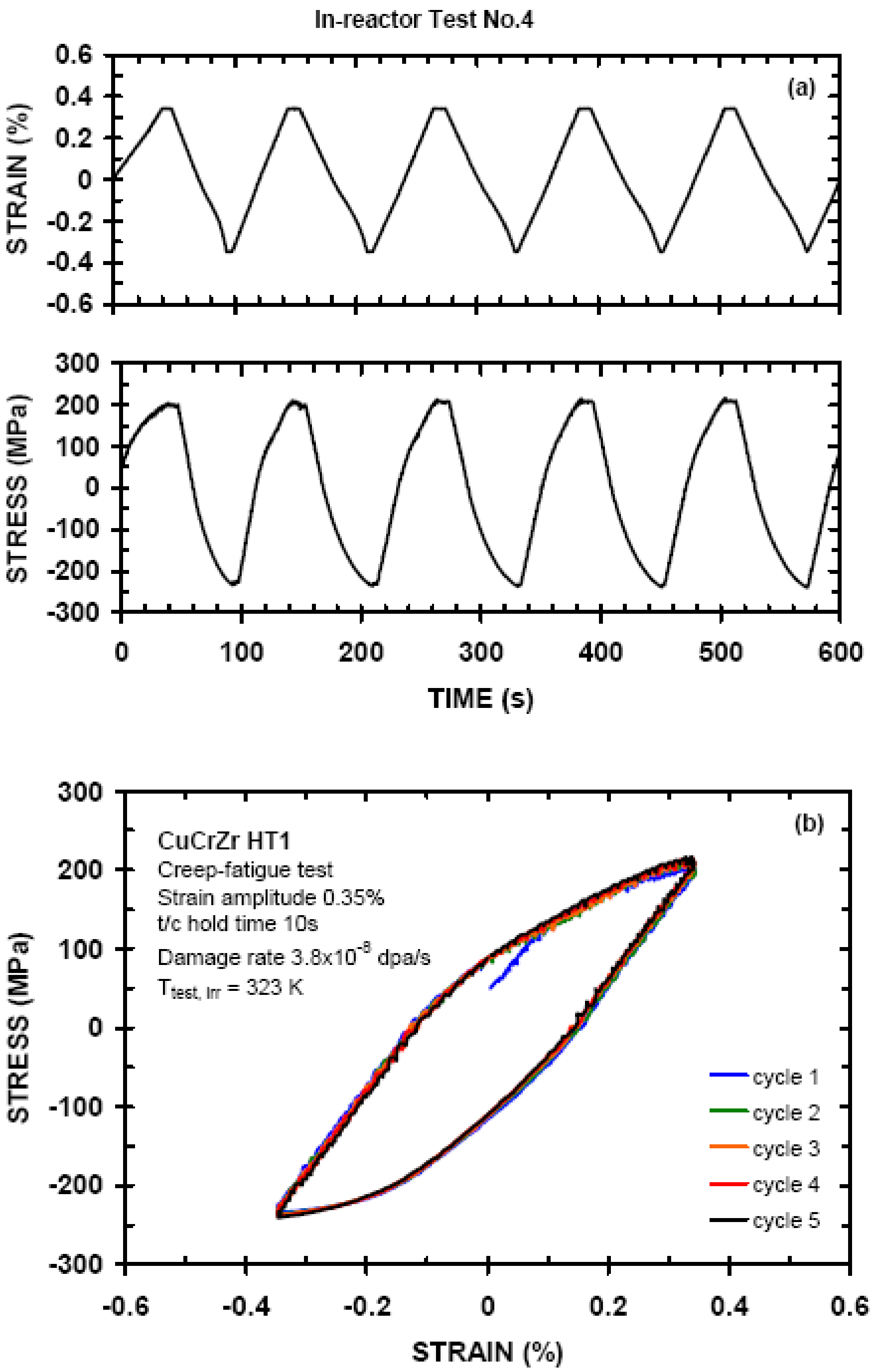

Figure 12. Same as in Figure 11, but for the test carried out at 323K with a strain amplitude of 0.35 % (Test No. 4) and a holdtime of 10s.

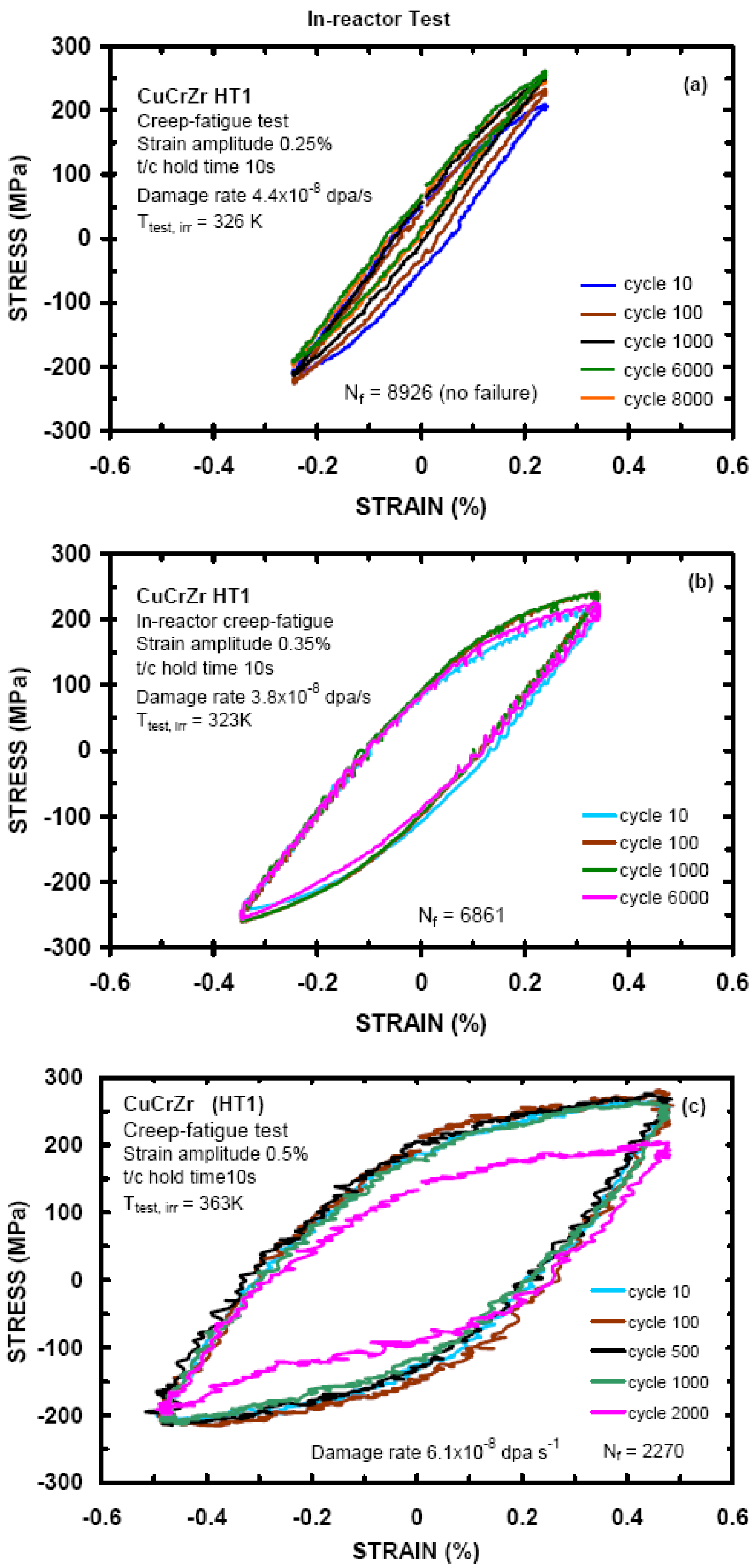

Figure 13. (Figure caption on page 35)

Figure 13. Hysteresis loops obtained for the higher number of cycles (and higher displacement doses) during in-reactor creep-fatigue tests carried out at strain amplitudes of (a) 0.25 %, (b) 0.35 % and (c) 0.5 %. Note that the results for the strain amplitude of 0.5 % are taken from [5]. All test were carried out with a holdtime of 10s.

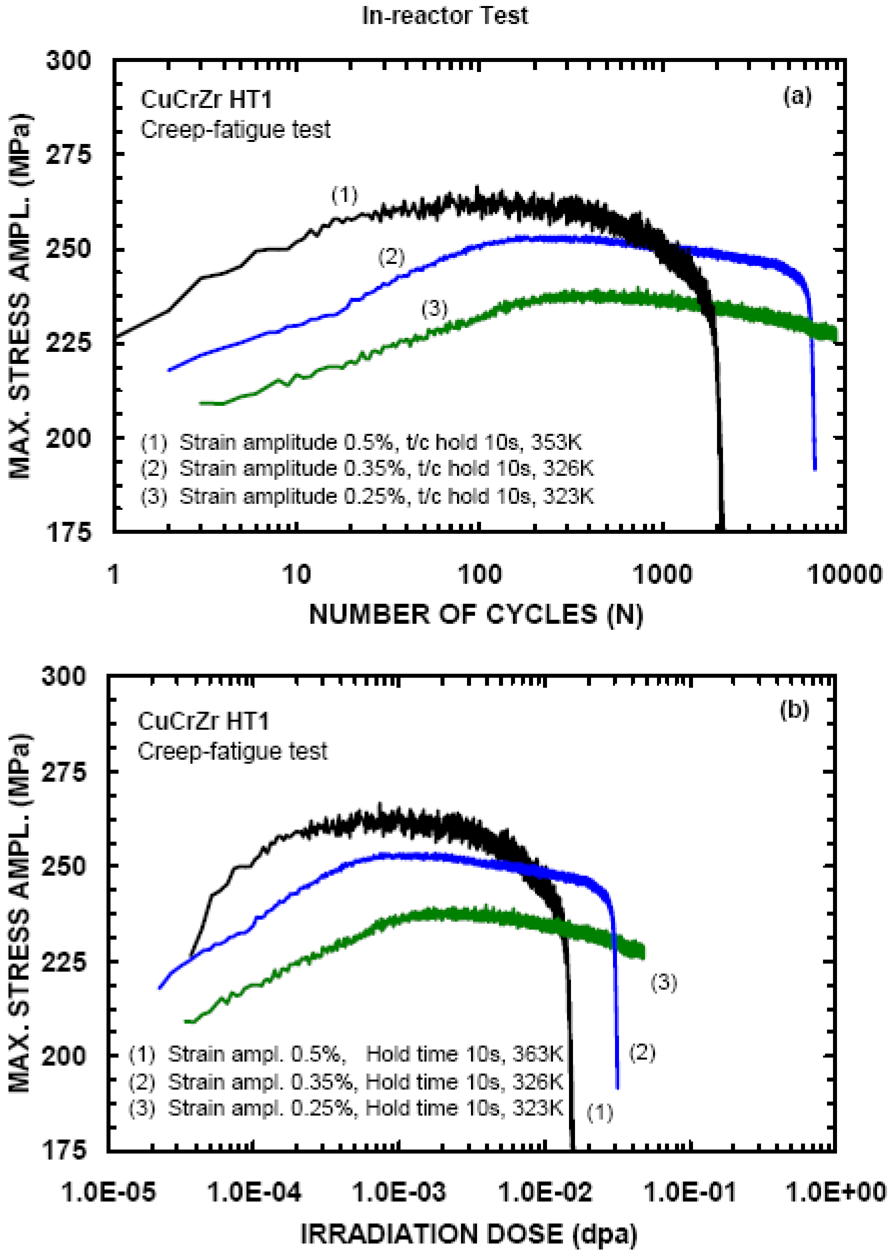

Figure 14. Variation of the maximum stress amplitudes (a) with number of cycles and (b) irradiation dose for the in-reactor creep-fatigue tests carried out with 0.25 %, 0.35 % and 0.5 % strain amplitudes and a holdtime of 10s at temperatures of 326K, 323K and 363K, respectively. Note that the results for the strain amplitude of 0.5 % are taken from [5].

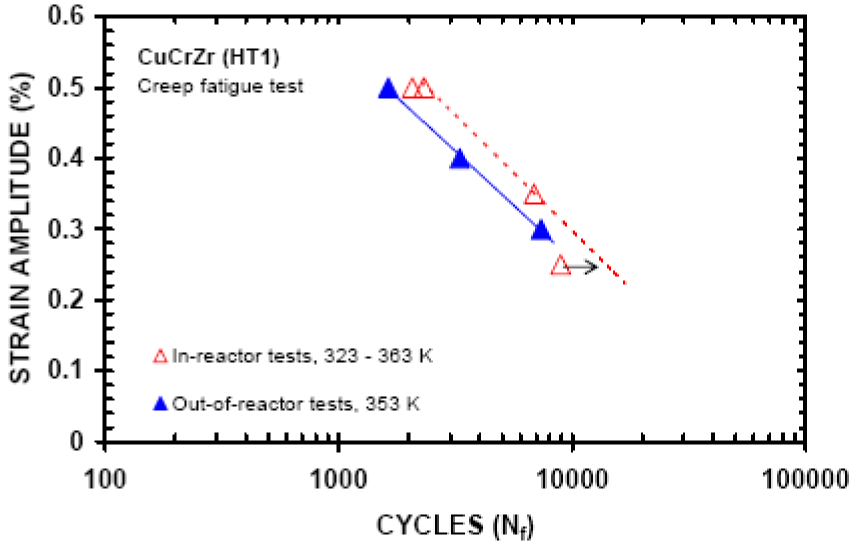

Figure 15. Dependence of the number of cycles to failure (N_f) on the strain amplitudes for the out-of-reactor and the in-reactor creep-fatigue tests. Note that at all three strain amplitudes the creep-fatigue lifetime (i.e. N_f) is somewhat longer during the in-reactor tests than that during the out-of-reactor tests.

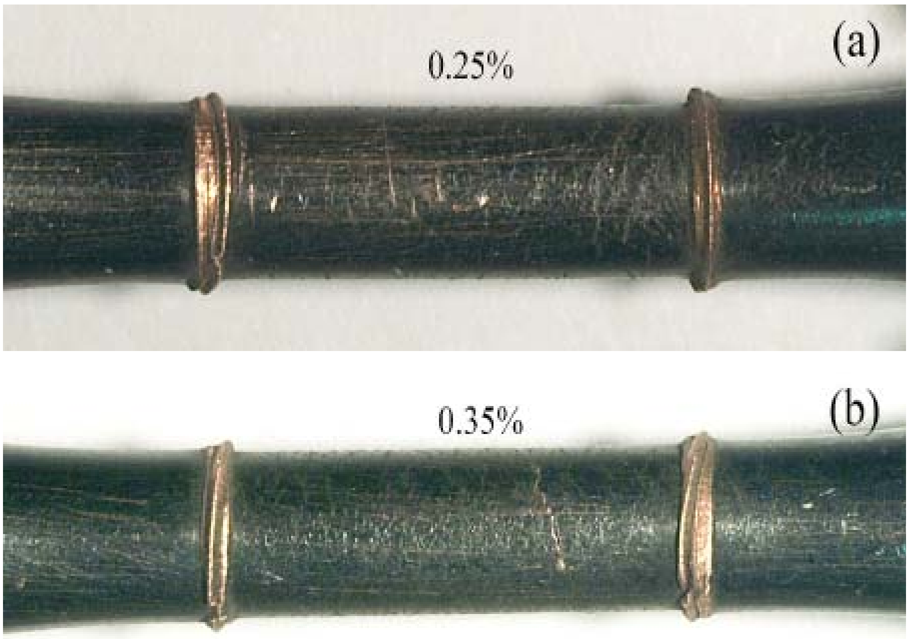

Figure 16. Photographs of CuCrZr (HT1) specimens after in-reactor creep-fatigue tests at strain amplitude of (a) 0.25 % (Test No. 3) and (b) 0.35 % (Test No. 4). Both tests were carried out with a holdtime of 10s. The specimen tested at the strain amplitude of 0.25 % (Test No. 3) did not show any sign of cracking (see also Figure 14, curve marked 2) whereas a crack can be clearly seen in the specimen tested at the strain amplitude of 0.35 % (Test No. 4).

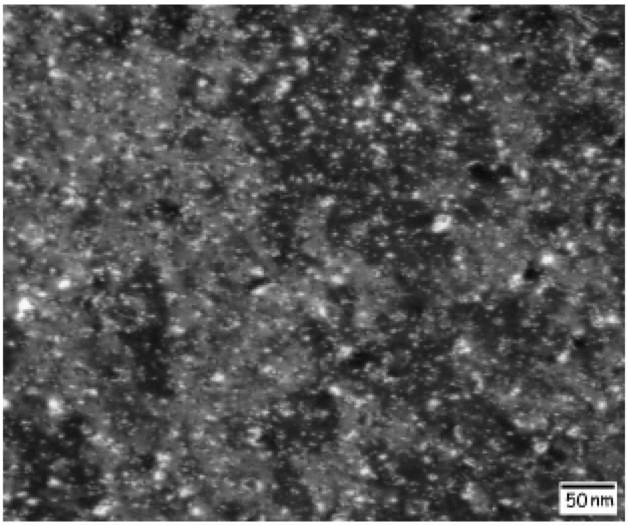

Figure 17. The weak beam dark field image illustrating the presence of small defect clusters and precipitates in the CuCrZr (HT1) specimen in the as-irradiated (and unperformed) condition.

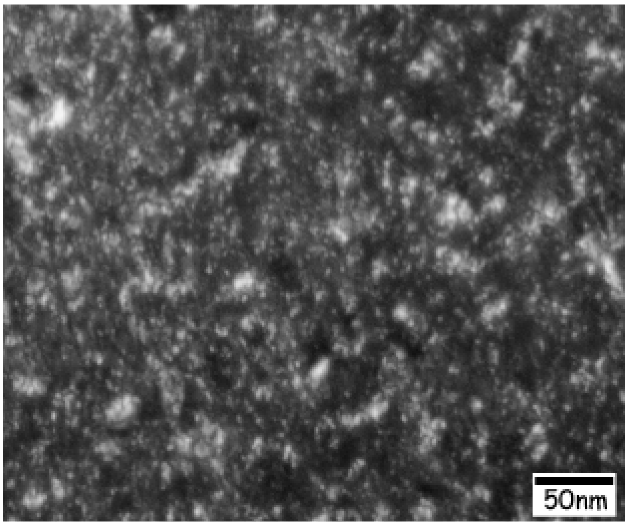

Figure 18. The weak beam dark field image illustrating the presence of small defect clusters and precipitates in the in-reactor deformed specimen with strain amplitude of 0.25 % (and a holdtime of 10s) (Test No. 3). Note that the spatial distribution of the irradiation induced defect clusters remains fairly homogeneous even at the end of the creep-fatigue test.

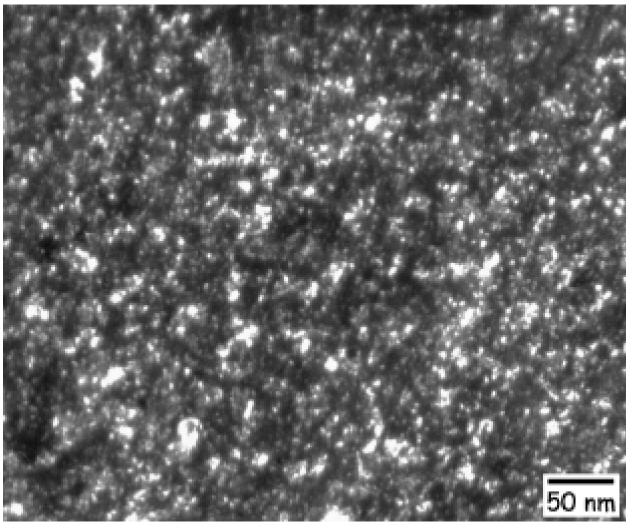

Figure 19. Same as in Figure 18, but for the specimen in-reactor creep-fatigue tested with a strain amplitude of 0.35 % (Test No. 4) with a holdtime of 10s.

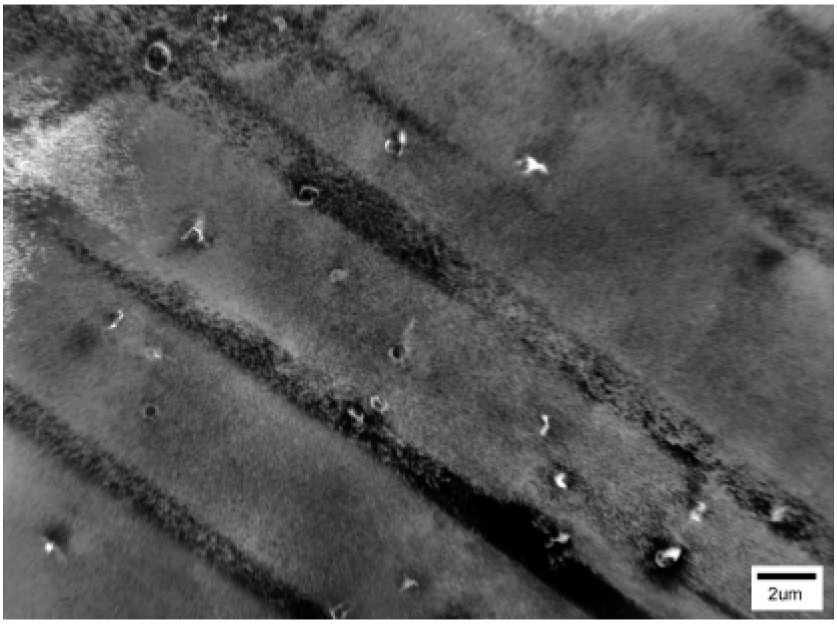

Figure 20. An example of diffuse cleared channels observed in the specimen in-reactor creep-fatigue tested with a strain amplitude of 0.25 % (Test No. 3). Note that the bands seem to be associated with a grain boundary.

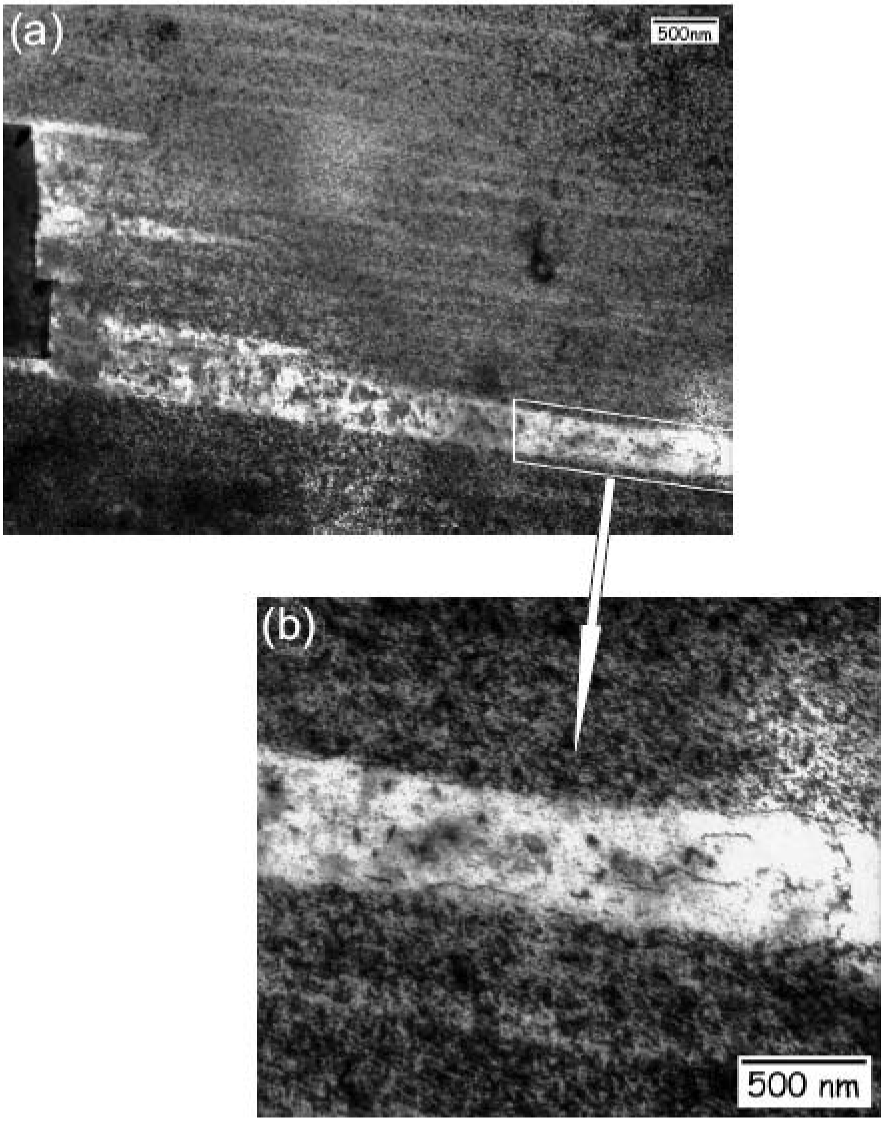

Figure 21. Another example of diffuse cleared channels in the in-reactor creep-fatigue tested (Test No. 3) specimen with a strain amplitude of 0.25 % and a holdtime of 10s. Note that the bands are associated with the incoherent part of a twin boundary (Figure 21(a)). Figure 21(b) shows an enlarged view of a portion of one of the bands (marked with a box) in Figure 21 (a). A few dislocation segments and a few remaining defect clusters can be seen in the figure. The high density of precipitates is also visible in Figure 21(b).

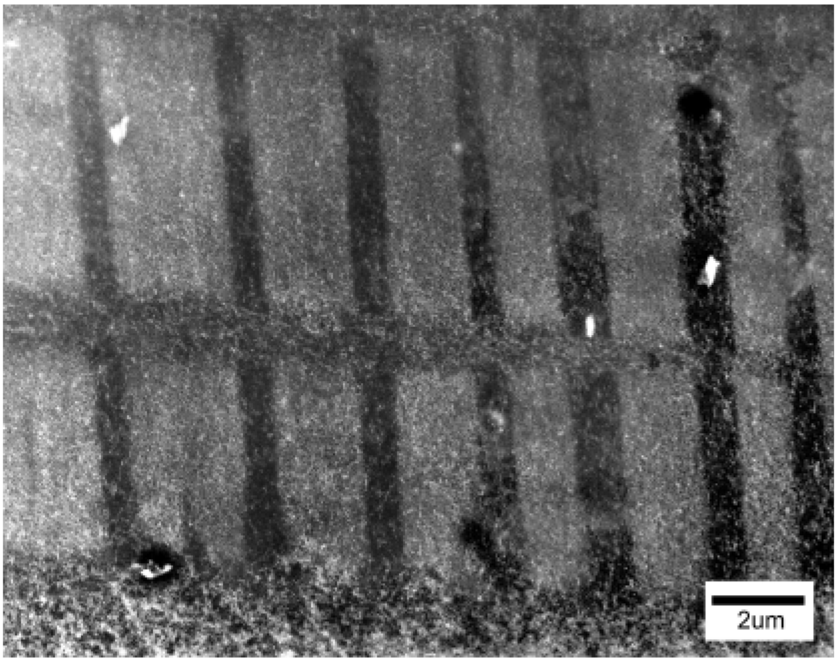

Figure 22. An example of diffuse cleared channels in the in-reactor creep-fatigue tested specimen with a strain amplitude of 0.35 % and with a holdtime of 10s. Two sets of channels can be seen in Figure 22. One set of the channels are associated with the coherent part of a twin boundary.

Risø's research is aimed at solving concrete problems in the society.

Research targets are set through continuous dialogue with business, the political system and researchers.

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