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Effect of heat treatments on precipitate microstructure and mechanical properties of a CuCrZr alloy

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Abstract

The precipitate microstructure of prime aged CuCrZr was coarsened by overaging to see if the larger precipitates could prevent the initiation of plastic flow localization in irradiated CuCrZr. A number of tensile and fracture toughness specimens of prime aged CuCrZr alloy were given overaging treatments at 873 K for 1, 2 and 4 h. A subset of these specimens were irradiated with fission neutrons in the BR-2 reactor at Mol (Belgium) at 333 K and 573 K to a dose level of ∼0.3 dpa. Both unirradiated and irradiated tensile specimens were tested at 295 K, 333 K and 573 K. Fracture toughness tests were carried out at 293, 333 and 573 K. Transmission electron microscopy was used to investigate the effects of overaging, subsequent irradiation and the effect of deformation. The results indicate that the overaging treatment of 873 K for 1 h produced a precipitate microstructure that improved the plastic instability, overall ductility and fracture toughness.

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1. Introduction

Currently, the precipitation hardened CuCrZr alloy is being evaluated for use in the first wall and divertor components of ITER. In service, both of these components will be exposed to an intense flux of fusion (14 MeV) neutrons while being subjected to thermo-mechanical stresses. Experimental investigations have demonstrated that neutron irradiation of prime aged CuCrZr at temperatures below ∼473 K leads to a substantial increase in strength, formation of a tensile instability, and a severe loss of work hardening ability and uniform elongation [1,2]. Experimental results indicated that while the precipitates in the prime aged condition were present in a high density, they were too small in size and thus too weak to effectively inhibit dislocation motion during deformation. This is based on the fact that the channels are generally clear of precipitates as well as defects and dislocations, indicating that the precipitates were unable to promote any work hardening within the channels and were sheared by the passing of so many dislocations.

It was therefore decided to coarsen the precipitate microstructure by annealing the prime aged CuCrZr so that larger and hopefully stronger precipitates, albeit in lower density, might prove more

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effective at preventing the initiation of plastic flow localization by resisting dislocation motion. The rationale for this approach stems in large part from our observations on the deformation behavior of neutron irradiated oxide dispersion strengthened GlidCop Al25, which was found to be resistant to flow localization because of the high density of particles, dislocations and fine subgrain microstructure. As a starting point, we hoped to achieve a precipitate microstructure in the overaged CuCrZr that was coarsened to a level near that of the GlidCop Al25, that is, particles with an average size of ~7–8 nm with a density of $\sim 10^{22}$ particles per m^3. The following sections present the results of these experimental investigations.

2. Experimental details

The CuCrZr alloy (Cu–0.73%Cr–0.14%Zr) supplied by Outokumpu Oyj (Finland) was solution annealed at 1233 K for 3 h, water quenched and then prime aged (PA) at 733 K for 3 h. After prime ageing the specimens were given further heat treatments to modify the precipitate microstructure. In the first series, the prime aged specimens were annealed in vacuum at 873 K for 1, 2 and 4 h. In the second series a number of prime aged specimens were annealed at 973 K and 1123 K for 4 h. In both series of experiments, the specimens were water quenched after final annealing at various temperatures.

The microstructures of each heat treatment condition were characterized using a JEOL 2000FX transmission electron microscope (TEM). Specimen preparation and the imaging conditions used to characterize the microstructure of these samples have been described in previous work [1,2].

A number of tensile and fracture toughness specimens of the CuCrZr alloy in the prime aged condition and the specimens further heat treated at 873 K for 1 and 4 h were irradiated with fission neutrons in the BR-2 reactor at Mol (Belgium) at 373 and 573 K to a displacement dose level of ~0.3 dpa (displacement per atom). The damage rate during irradiation was $\sim 6 \times 10^{-8}$ dpa s^{-1}.

The prime aged and various heat treated specimens in the unirradiated condition were tensile tested at 323 and 573 K. The prime aged conditions as well as specimens heat treated at 873 K for 1 and 4 h and subsequently irradiated at 373 and 573 K were also tensile tested in the post-irradiation condition. All tests were carried out in vacuum ($<10^{-4}$ Torr) at a strain rate of 1.2×10^{-3} s^{-1}. The specimens irradiated at 333 K were tensile tested at 295 and 333 K whereas those irradiated at 573 K were tensile tested at 573 K. Single edge notched bend SEN(B) fracture toughness specimens of dimensions 3 × 4 × 27 mm were used in the fracture resistance testing. The initial notch and the 20% side grooves were machined using electric wire discharge machining. The applied prefatigued crack length to specimen width ratio (a/W) was about 0.5. Fracture resistance curves were determined using the displacement controlled three point bend test method with a constant displacement rate of 1.5×10^{-2} mm/min. Fracture resistance testing at elevated temperatures was carried out in a silicon oil bath. Load, displacement and crack length measured using the DC–PD method were recorded during the testing and the fracture resistance curves were determined following the ASTM E1737-96 standard procedure.

3. Results

3.1. Pre-irradiation precipitate microstructure

As expected, heat treatments after prime ageing led to significant coarsening of the prime aged precipitate microstructure. Fig. 1 shows examples of the precipitate microstructure in the prime aged condition and after annealing at 1 and 4 h at 873 K. The precipitates in the prime aged condition are small Guinier–Preston (G–P) zones as described previously [3]. The higher temperature annealing replaced the G–P zones with precipitates thought to be predominately incoherent Cr-rich particles, which are commonly observed in this alloy [3–7]. The images in Fig. 1(c) and (d) indicate that extensive precipitation occurs at the twin boundaries, which may be Cu_4Zr based on evidence found in earlier work [4]. As the data presented in Table 1 show, annealing for 1–2 h at 873 K produced a significant coarsening that was accelerated when the annealing time was increased to 4 h. The size distributions of precipitates in the prime aged and overaged conditions are given in [5]. Increasing the annealing temperature to 973 K for 4 h produced an even lower density of larger precipitates, whereas annealing at 1123 K appeared to solution anneal the material and remove all but the sub-micron Cr inclusions, which were probably present in the original solution annealed material.

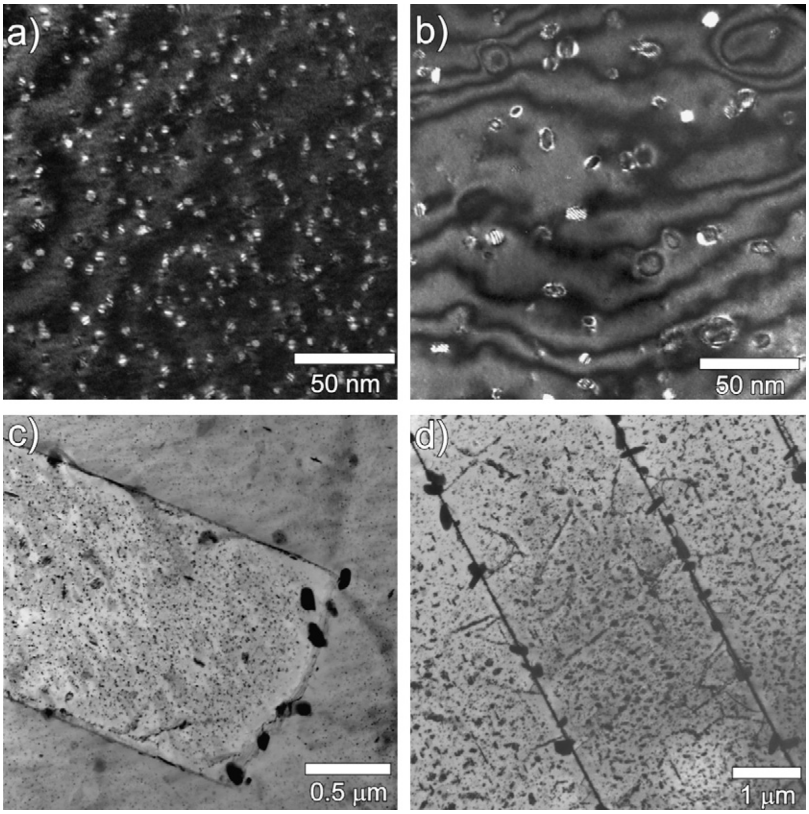

Fig. 1. The image in (a) shows G–P zones present in the prime aged CuCrZr. The images (b) and (c) are from the sample given the 1 h/873 K annealing treatment, (d) is from the sample given the 4 h/873 K annealing treatment. Annealing coarsens the GP zones into larger Cr-particles that precipitate within the matrix as well as on twin and grain boundaries.

Table 1
Measured size and densities of precipitates

Heat treatment	Precipitate size (nm)	Precipitate density ($\times 10^{23}$ m^{-3})
Prime aged (PA)	2.2	2.6
PA + 873 K/1 h	8.7	0.17
PA + 873 K/2 h	9.4	0.18
PA + 873 K/4 h	21.3	0.015
PA + 973 K/4 h	46.4	0.007
PA + 1123 K/4 h	Precipitates removed (effectively solution annealed)	–

3.2. Tensile properties

The engineering stress–strain curves for the PA specimen and specimens heat treated at 873 K for 1 and 4 h tested at 323 and 573 K are shown in Fig. 2. It is clear that for both test temperatures the tensile strength decreases as the annealing time increases from 1 to 4 h. With the exception of the 4 h annealing condition at 873 K, the ductility and work hardening behavior appear to be similar to that of the prime aged material. The loss of strength is consistent with the coarsening response of the precipitate microstructure (Table 1) but this apparently is not enough to alter the work hardening behavior or overall ductility.

The engineering stress–strain curves for specimens irradiated at 333 K and 573 K are shown in Fig. 3. The stress–strain curves for OFHC-copper irradiated and tested under the same condition as the CuCrZr alloy are shown for comparison in Fig. 3. Irradiation at 333 K to a dose level of ~0.3 dpa caused a large increase in the yield strength of the prime aged as well as the two overaged alloys when tested at either room temperature or 333 K. The increase in the yield strength in the prime aged material is accompanied by the formation of a distinct yield point and a severe loss of uniform elongation in tension. It should be noted however that the yield drop is not very prominent either in the case of the prime aged or the two

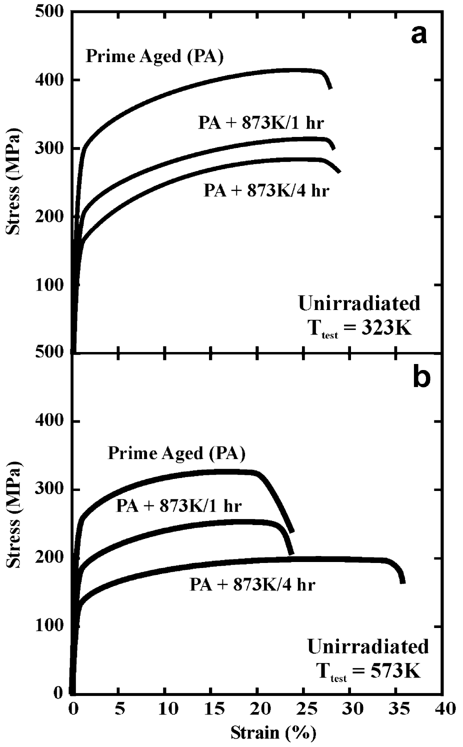

Fig. 2. Stress–strain curves for CuCrZr alloy in the prime aged condition and after annealing at 873 K for 1 and 4 h and tensile tested at (a) 323 K and (b) 573 K.

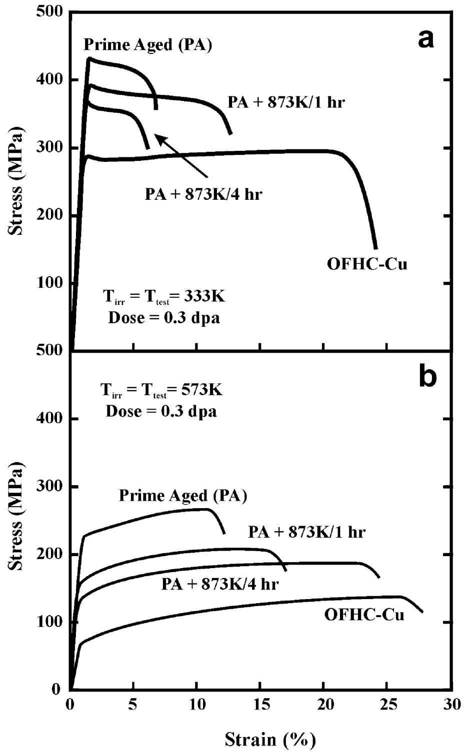

Fig. 3. Stress–strain curves for CuCrZr alloy irradiated to a dose level of ~0.3 dpa in the prime aged and overaged at 873 K for 1 h and 4 h conditions and tensile tested at (a) 333 K and (b) 573 K.

overaged specimens irradiated and tested at 333 K. The results of post-irradiation tensile tests on specimens irradiated and tested at 573 K (Fig. 3(b)) demonstrate that the CuCrZr alloy becomes noticeably softer due to irradiation at 573 K to 0.3 dpa in both the prime aged and the overaged conditions. All the strength and ductility data for the results reported in Figs. 2 and 3 are tabulated in [5].

Microstructural characterization of the CuCrZr alloys with different heat treatments and irradiated at 333 and 573 K to 0.3 dpa has not yet been completed other than a preliminary investigation of the deformation response in the irradiated and deformed samples. Evidence of plastic flow localization in the form of cleared channels (see Fig. 4 for examples) was found in all of the irradiated and deformed specimens, which leads us to conclude that the loss of uniform ductility are likely due to the deformation being confined to the narrow channels.

3.3. Fracture toughness properties

The data measured during fracture toughness testing of three point bend specimens, e.g., load, load line displacement and potential drop values, are presented in normalised form to make a more informative comparison with the results of individual tests and material conditions. The normalised load–displacement curves (Fig. 5) give qualitative information on the effects of heat treatment, test temperature etc. on fracture behaviour of three point bend specimens.

Typical normalised load–displacement curves for CuCrZr alloy in the unirradiated and neutron irradiated conditions are shown in Fig. 5. The three point bend load–displacement curves show a general trend which is similar to that exhibited by the tensile stress–strain curves, i.e., decrease in the strength level due to overaging compared to that in prime aged condition. At the ambient temperature the unirradiated CuCrZr alloy shows relatively flat normalised load–displacement curve without any clear maximum indicating an extensive plastic deformation and crack tip blunting without clear crack extension, i.e., the CuCrZr base alloy has relatively high fracture toughness. In the overaged conditions the normalised load–displacement curves are at lower load levels but the curves have a similar general form to that in the prime aged

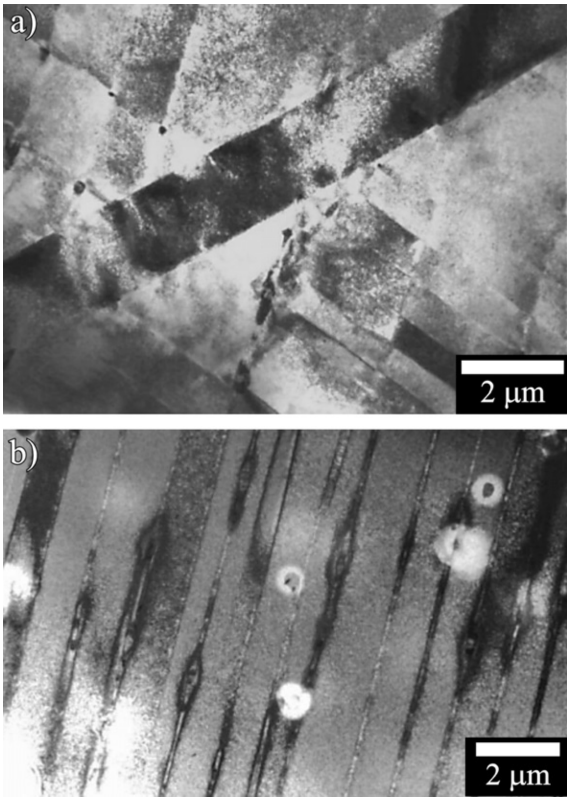

Fig. 4. Examples of cleared channel formation in specimens irradiated at 333 K to ~0.3 dpa and deformed at 333 K: (a) prime aged and (b) overaged at 873 K for 4 h.

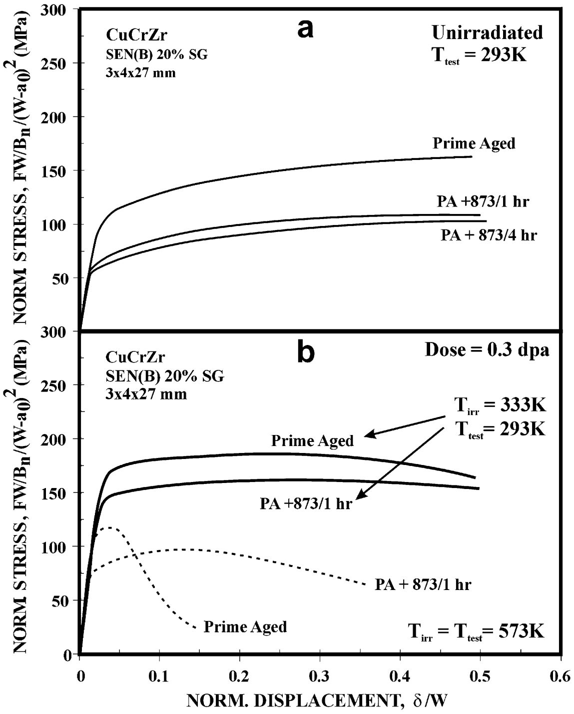

Fig. 5. Normalised load–displacement curves for CuCrZr alloy (a) in unirradiated and prime aged and overaged (at 873 K for 1 h and 4 h) conditions tested at 293 K and (b) irradiated at 333 K and 573 K and tested at 293 K and 573 K, respectively, to a dose level of ~ 0.3 dpa in the prime aged and overaged (at 873 K for 1 h) conditions.

condition. In the neutron irradiated condition the load levels of the normalised load–displacement curves increase and the general form is more flat and goes through a maximum when compared to that in the unirradiated condition at ambient temperature. This indicates that the CuCrZr alloy also had relatively high toughness after irradiation at the ambient temperature. The most pronounced change is observed when the CuCrZr alloy is irradiated and tested at 573 K, i.e., in both prime aged and overaged conditions the normalised load–displacement curves show a clear maximum indicating relatively low fracture toughness.

Fracture resistance curves of CuCrZr alloy in the unirradiated and the neutron irradiated conditions are shown in Fig. 6. In the unirradiated condition clear stable crack growth was observed only in the prime aged condition at 473 K where the initiation fracture toughness J_Q value was about 180 kJ m^{-2}. In all the other conditions strong crack tip blunting was observed. In the irradiated condition crack tip blunting without any stable crack growth was also observed when irradiation and fracture toughness tests were carried out at the ambient temperature. Relatively low initiation fracture toughness values of about 33 kJ m^{-2} and 82 kJ m^{-2} were observed in the prime aged and the overaged conditions, respectively, when irradiated and fracture toughness tested at 573 K. It is noted that only those J_Q values for the prime aged CuCrZr alloy which were irradiated and tested at 573 K are valid according to limitations ($J_{\max} = b_0\sigma_y/20$, where b_0 is remaining ligament and σ_y is flow stress) set for specimen dimensions. Limiting values of $J_{\max}$ for the prime aged and overaged conditions are about 90 and 83 kJ m^{-2} at the ambient temperature and 45 and 35 kJ m^{-2} at 573 K, respectively. The numerical values of all the data reported in Figs. 5 and 6 are tabulated in [5].

4. Summary and conclusions

An overaged condition was established that yielded a reasonable density of larger, and presumably stronger particles than found in the prime aged

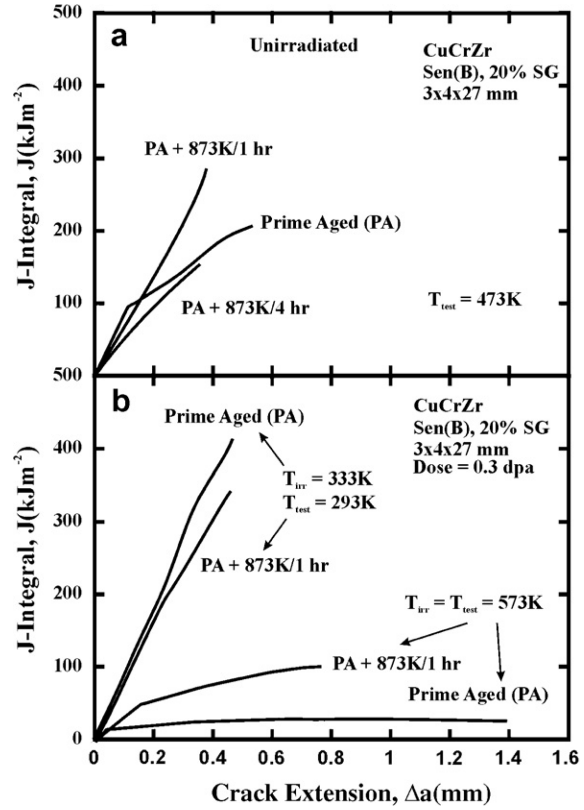

Fig. 6. Fracture resistance curves for CuCrZr alloy (a) in unirradiated and prime aged and overaged (at 873 K for 1 h and 4 h) conditions tested at 473 K and (b) irradiated at 333 K and 573 K and tested at 293 K and 573 K, respectively, to a dose level of ~0.3 dpa in the prime aged and overaged (at 873 K for 1 h) conditions.

condition. The coarsening of the precipitates due to overaging produced a significant decrease in the tensile strength and some increase in fracture toughness properties of the alloy. The irradiation at 333 K to a dose level of ~0.3 dpa caused significant hardening both in the prime aged and overaged specimens while severely reducing the ductility and work hardening. The tensile response and post-deformation microstructure of the irradiated alloy indicate that the lower density of larger particles produced by overaging at 873 K for 1 h improved the resistance to plastic instability and led to some increase in the overall ductility. The overaging heat treatment PA + 873 K/1 h also improved the fracture toughness properties when compared to those of the prime aged CuCrZr alloy in irradiated conditions at 333 and 573 K. Despite these positive changes in the mechanical response, microstructurally the material was found to deform by dislocation channeling in all of the conditions studied. These results suggest that some improvement in the mechanical response of irradiated CuCrZr can be realized if care is taken to alter the final thermo-mechanical treatment to introduce a precipitate dispersion that is coarser than that produced by prime ageing while still maintaining a precipitate structure adequate to inhibit the motion of dislocations within the channels.

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