OVERAGING OF OUTOKUMPU CuCrZr AT 600°C - D. J. Edwards (Pacific Northwest National Laboratory)* and B.N. Singh (Risø National Laboratory)

OBJECTIVE

The objective of this work is to study the effect of overaging on the microstructure of CuCrZr, its effects on mechanical properties, and how it affects performance under neutron irradiation.

SUMMARY

An attempt is being made to alter the starting microstructure of CuCrZr to produce a microstructure that is not as sensitive to overageing under irradiation as the prime aged condition. Different overageing conditions (600, 700 and 800°C for 4 hours) have been examined in an earlier report, and in this report shorter overageing times at 600°C have been examined. Overageing times of 1 and 2 hours resulted in a microstructure that is similar in overall precipitate size and density to that of the oxide dispersion in GlidCop Al25. These samples will be included in future irradiation experiments to examine the effects of irradiation on the mechanical and physical properties.

PROGRESS AND STATUS

Introduction

Precipitation strengthened CuCrZr is a copper alloy being considered for use in fusion reactor devices due to its thermal and electrical conductivity coupled with reasonable strength and fracture toughness [1-6]. Other alloys such as the oxide dispersion strengthened GlidCop Al25 and the precipitation strengthened CuNiBe, the highest strength alloy by a wide margin, have also been evaluated. These latter two alloys lack the required mechanical properties at irradiation temperatures above ~250°C due to a transition in how they begin to fail. For example, in the extreme case the CuNiBe alloy loses all semblance of ductile failure and fails by brittle intergranular cracking along the grain boundaries at very low stresses. In contrast to this behavior, the CuCrZr alloy remains ductile throughout the same temperature range and maintains reasonable fracture toughness.

While CuCrZr can be heat treated to reasonable strength levels, precipitate stability becomes a serious issue when the irradiation temperature exceeds ~280°C [4]. At this irradiation temperature and higher, the fine-scale precipitate microstructure begins to overage due to enhanced diffusion and cascade dissolution of the precipitates. The coarsening of the precipitates degrades the strength to unacceptable levels, and if the temperature and dose are high enough, can eventually lead to swelling. Part of the problem lies in the small size of the precipitates as related to the volume of a displacement cascade. The precipitates in a prime aged CuCrZr are typically on the order of a 1-4 nm in average size, smaller than or on the order of the size of the displacement cascades formed during neutron irradiation. The small size of the precipitates allows ballistic dissolution to dissolve some of the precipitation and place solute back in solution. As the irradiation temperature increases, the dissolution and enhanced diffusion under irradiation lead to precipitate coarsening in CuCrZr. The variables involved are complex and not completely understood, but phase stability (oxide particles versus lower temperature intermetallic phases as an example) as a function of irradiation temperature can play an important role in how the dispersion changes with irradiation conditions.

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* Pacific Northwest National Laboratory (PNNL) is operated for the U.S. Department of Energy by Battelle Memorial Institute under contract DE-AC06-76RLO-1830.

One approach to improve the stability of the microstructure during irradiation is to alter the starting precipitate size and density with the hope of achieving a balance between the radiation-induced dissolution of the precipitates and the subsequent redistribution of solute. A starting point would be to recognize that the ODS GlidCop alloy possesses an oxide dispersion that is extremely stable under irradiation up to temperatures of 450°C at very high doses. The average size of these particles is around 7 nm with a density of ~10^{22} m^{-3}, about an order of magnitude lower than in the prime aged CuCrZr [7]. The oxide particles are thermally stable up to high temperatures, which likely won't be the case for the precipitates in the CuCrZr. However, a coarser distribution of precipitates in the CuCrZr may achieve a microstructure more resistant to changes under irradiation, or at least postpone any serious degradation in mechanical and physical properties.

To that end, an experiment has been initiated wherein different ageing treatments have been explored to alter the starting microstructure of the CuCrZr. The preliminary analysis of the microstructure has been reported in an earlier semi-annual report [7]. Ageing conditions of 850, 700 and 600°C for 4 hours were given to samples of CuCrZr. The highest ageing temperature essentially left the material in a solution annealed condition with only a few large inclusions present in the microstructure. Both the 700 and 600°C ageing treatments however, produced a very coarse distribution of blocky precipitates with an average size of ~40 and 20 nm respectively. The density of these precipitates was on the order of 0.7 x 10^{21} m^{-3} for the 700°C treatment and 1.5 x 10^{21} m^{-3} for the 600°C treatment. The average sizes of particles represent a size distribution considerably more coarse than the precipitates that are present in the ODS alloy GlidCop Al25. Shorter ageing times were therefore given to the CuCrZr to further explore the possibility of producing a dispersion similar in density and size to that of the GlidCop.

In the following brief report, the results of overageing at 600°C for 1 and 2 hours will be presented. These results will be compared to the results obtained for the 4-hr aging treatments.

Experimental

Tensile specimens fabricated from Outokumpu CuCrZr (produced by Outokumpu Oy) were given three separate heat treatments to study the effect of overageing on the microstructure and mechanical properties. The heat treatments are listed below in Table 1. All heat treatments were done in vacuum (<10^{-4} torr). The microstructure was examined by transmission electron microscopy using a JEOL 2000FX.

TABLE 1 Heat treatments used to study overaging in Outokumpu CuCrZr

Prime Aged (PA): Solution annealed at 960°C / 3 hrs / WQ + aged 460°C / 3 hrs / WQ
600°C treatment: PA + 600°C / 4hrs / WQ
600°C treatment: PA + 600°C / 2hrs / WQ
600°C treatment: PA + 600°C / 1hrs / WQ

Results and Discussion

The overageing at 600°C for 1 and 2 hours produced a different precipitate distribution compared to the 4 hour overageing treatment reported earlier [1]. Micrographs are shown in Figure 1 giving examples of the different microstructures produced by the 3 different ageing times. The size distributions shown in Figure 2 illustrate the differences as a result of the shorter overageing times. The 1 and 2-hr treatment yielded a size distribution with a noticeably smaller average size and a narrower distribution than that produced in the 4-hr treatment. The shorter overageing times yield a size distribution similar to that of the GlidCop Al25. Grain boundary

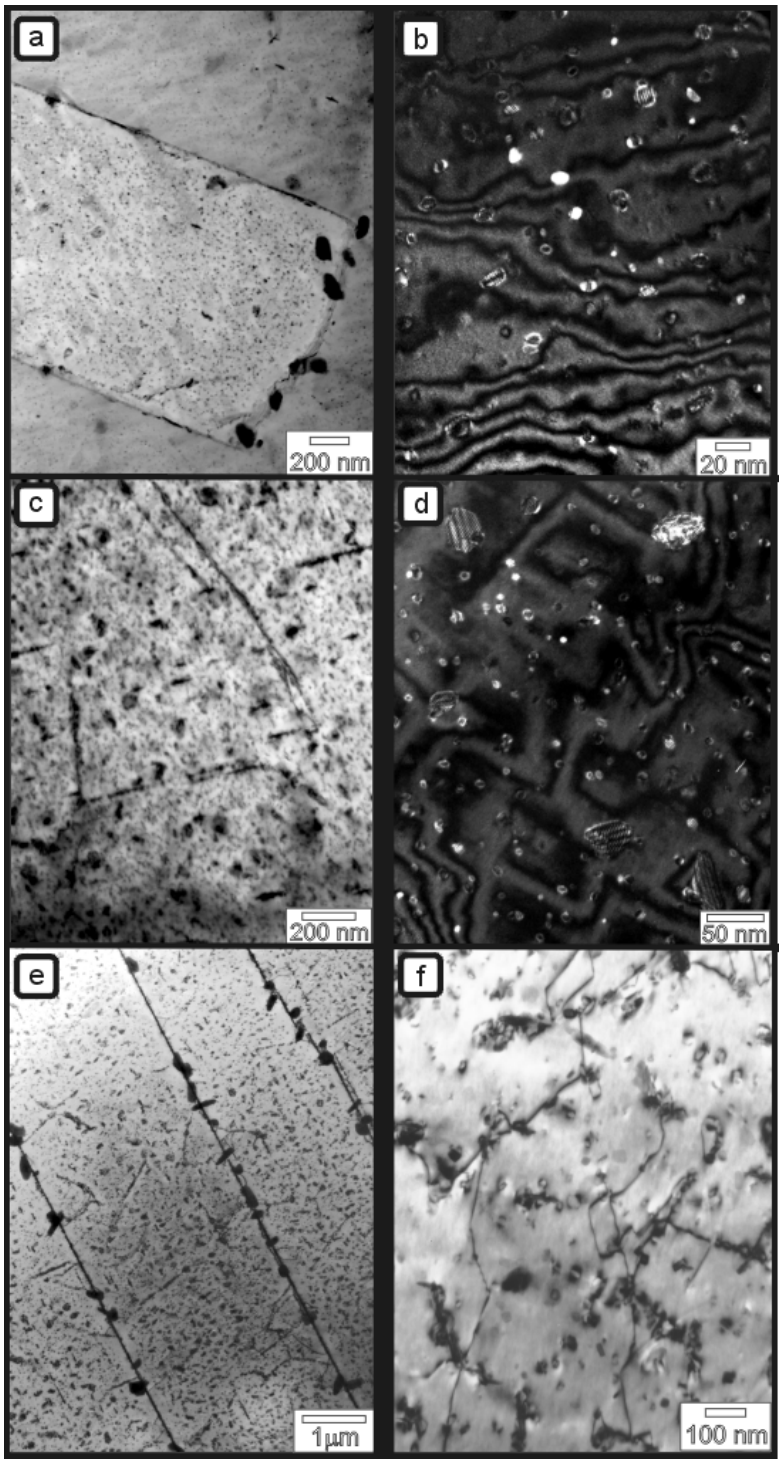

Figure 1. Comparison of the precipitate microstructure after overageing at 600°C for 1, 2 and 4 hours. The micrographs in (a) and (b) are for the 1-hr treatment, (c) and (d) are for the 2-hr treatment, and (e) and (f) are for the 4-hr treatment shown in the previous report [7].

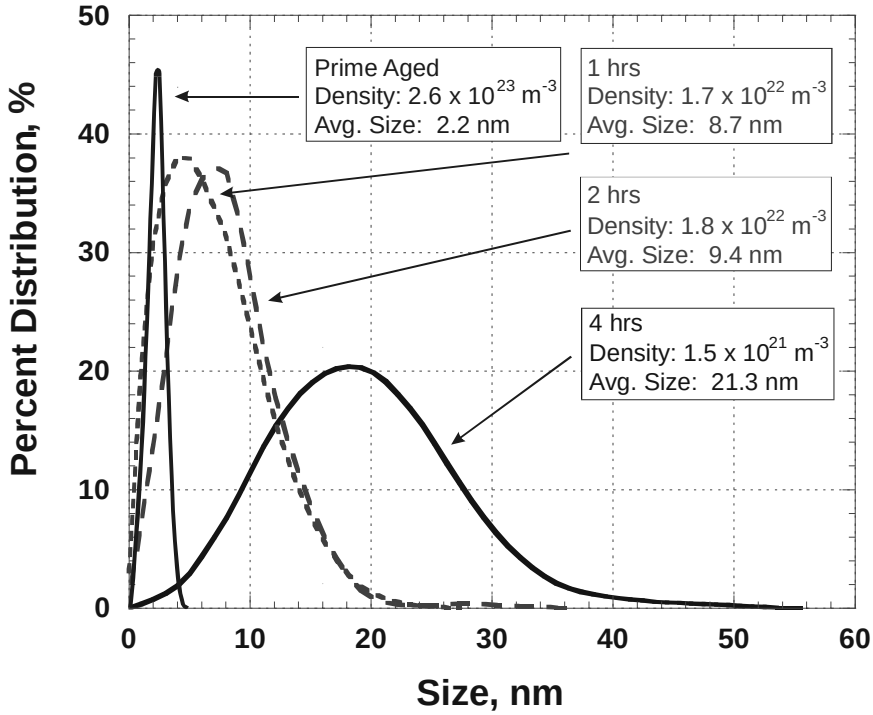

Figure 2. Size distributions are shown for the CuCrZr in the prime aged condition and after being overaged for 1, 2 and 4 hours. Note that the 1 and 2-hr treatments produce a size and density similar to that measured for the GlidCop Al25 alloys in the unirradiated condition (average size ~7 nm, density ~10^{22} m^{-3}).

precipitation and denuding along these areas occurred in all overageing conditions. Precipitation was also found to occur along annealing twins as shown in Figures 1a and 1e.

FUTURE WORK

Further work is necessary to characterize the precipitate types that have formed in the different aging treatments. Samples of the unirradiated material will be shipped to PNNL to be characterized in the 2010 Field Emission Gun ATEM. Tensile testing and electrical resistivity measurements will be conducted at Risø on the unirradiated samples to determine the effect of the aging treatments and how it relates to the observed microstructure. Samples will also be included in future irradiation experiments to study the effect of irradiation on the microstructure, mechanical and physical properties.

ACKNOWLEDGEMENTS

The present work was partly funded by the European Fusion Technology Programme. The authors wish to thank B. F. Olsen, N.J. Pederson and J.L. Lindbo. DJ Edwards would like to thank Risø National Laboratory for the support and assistance during his visit. His work was also partly supported by the U.S. Department of Energy under contract DE-AC06-76RLO 1830 with the Battelle Memorial Institute at the Pacific Northwest National Laboratory.

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