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Ageing effect on the properties of CuCrZr alloy used for the ITER HHF components

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Abstract

The tensile properties and structure changes due to various heat treatments of CuCrZr alloy with the composition specified for high heat flux components of ITER have been measured. The material was solution annealed at 970–1000 °C for 0.5-1 h and water quenched. The ageing temperatures were 350–650 °C and ageing times 1–180 min. Temperature–time diagrams of properties changes have been plotted. This gives guidelines for the selection of manufacturing heat treatments and prediction of the strength and ductility of CuCrZr alloy after applied manufacturing cycles. The effects of secondary heat treatment have also been studied. After first ageing at 480 °C for 2 h, an additional heat treatment at 650–750 °C was applied. The tensile properties after the applied heat treatments are presented.

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1. Introduction

CuCrZr alloy is used as a heat sink material for the high heat flux components of ITER including first wall, limiter and divertor. A solution anneal at 980–1000 °C for 1 h, water quench, and ageing at 450–480 °C for 2–4 h is specified as a reference heat treatment for ITER use [1]. However, the actual manufacturing cycle might be different from such 'ideal' heat treatment. For example, temperatures of 500–800 °C and times of 5–60 min are used for the amour tiles brazing, so, it is important to know the ageing kinetic of CuCrZr, and to know

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the strength and ductility of this alloy after different ageing treatments.

Effects of heat treatments on the properties of CuCrZr alloys have been reported [2,3]. Unfortunately, both the solution anneal temperature and time in [2] were too low (950 °C and 30 min) to produce complete solution anneal of CuCrZr. Moreover, the materials composition was not within the ITER specification. The present work starts with material and solution anneal heat treatment that satisfied ITER specification.

2. Experimental conditions

CuCrZr bars $50 \times 40 \times 200$ mm produced by Zollern Co were used for this investigation. The actual chemical composition and the ITER specification

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	Cu	Cr	Zr	Al	Co	Fe	Р	Pb	S	Zn	
ITER specification	base	0.6–0.9	0.07–0.15	0.0016 ^a	0.06 ^a	0.009 ^a	0.0069 ^a	0.0017 ^a	0.0023 ^a	0.0069 ^a	
Chemical analysis result	base	$\begin{array}{c} 0.85 \\ \pm \ 0.04 \end{array}$	$\begin{array}{c} 0.09 \pm \\ 0.005 \end{array}$	$\begin{array}{c} 0.005 \\ \pm \ 0.003 \end{array}$	< 0.001	$\begin{array}{c} 0.083 \\ \pm \ 0.030 \end{array}$	$\begin{array}{c} 0.0014 \\ \pm \ 0.0004 \end{array}$	< 0.005	$\begin{array}{c} 0.00083 \\ \pm \ 0.00035 \end{array}$	$\begin{array}{c} 0.0040 \\ \pm \ 0.0021 \end{array}$	

Table 1 Chemical composition of CuCrZr used for investigation and ITER specification for this alloy

^a Typical content, not included in specification.

are presented in Table 1. The data show that the main alloying elements are within the ITER specification. The material was solution annealed by the manufacturer at 970 ± 10 °C for 20 min and water quenched. The specimens $8 \times 8 \times 50$ mm were cut from the bar. The ageing heat treatments were performed in quartz capsules filled with argon. Exposure times and ageing temperatures have been recorded for each specimen.

Three series of experiments were performed:

- 1. Effects of ageing temperature and exposure on the tensile strength and hardness of CuCrZr alloy. The ageing temperatures were 350, 400, 450, 500, 550, 600 and 650 °C. The exposure times varied from 1 to 180 min. The ageing temperature was maintained within 5 °C. These tests provided the data to construct time-temperature diagrams of tensile properties as a function of heat treatment.
- 2. Effects of high temperature ageing, simulating brazing temperatures proposed for high heat flux (HHF) component manufacturing, on the tensile properties of CuCrZr alloy. Two types of specimens were used in this series of experiments: (a) solution annealed specimens and (b) solution annealed and aged at 480 °C for 2 h. Both sets of specimens were aged at 650 °C and 750 °C for 1, 5, 10, 30 and 60 min. Tensile properties were then measured at room temperature.
- 3. Simulation of non-perfect cooling rate during 'solution anneal'. The objective of these experiments was to simulate heat treatments that may be realized during manufacturing of relatively large size or encapsulated components, so that fast cooling rates required for perfect solution anneal are not achievable. The same material manufactured by Zollern Co in solution annealed state has been used. The specimens were contained in evacuated capsules and exposed for 30 min at 1000 °C. The capsules were then water cooled; due to the encapsulation, the cooling rate

was in the range 1-3 °C/s. Specimens were aged at 500 °C for 1, 5, 10, 30, 60, 120 and 180 min and tensile tested. The tensile properties of these specimens were compared with another set subjected to solution anneal at 970 °C and aged at the same time and temperatures as the slower cooled specimens.

Tensile tests were performed on an Instron-1195 machine at room temperature (RT). Cylindrical specimens with diameter 3 mm and gage length 15 mm were used for the tests. The crosshead movement rate was 0.5 mm/min. For each specimens ultimate strength (UTS), yield strength (YS), total elongation (TEL) and reduction of area were measured.

Brinell hardness (HB) was measured on some specimens where variations in hardness (and strength) due to heat treatment were expected. The ball diameter was 5 mm, loads were up to 200 kg, and the loadindentation depth diagrams were recorded.

3. Results and discussion

Statistical processing of tensile data has been performed and 3D figures have been plotted, i.e. dependences of tensile properties vs ageing time and temperature. The strength functions were plotted by the least-squares method. The projections of tensile properties on the time-temperature plane are presented in Fig. 1. The location of experimental points used for statistical analysis is shown in figures. These curves give guidelines for the selection of manufacturing heat treatment and prediction of the strength and ductility of CuCrZr alloy after applied manufacturing cycles. It is concluded from these figures that the strength maximum is reached for ageing at 480 ± 20 °C and exposure times of 1-2 h. Increasing the ageing temperature resulted in a shorter time (1-10 min) to reach maximum strength. The decrease of strength at longer exposure (overageing effect) was observed. The minimum



Fig. 1. Time-temperature (T-T) diagrams of RT ultimate tensile strength (UTS) (a), yield strength (YS) (b), total elongation (TEL) (c) and reduction of area (RA) (d) of aged CuCrZr alloy. (White points indicate the location of experimental data used for interpolation).

of ductility does not precisely coincide with the maximum strengthening, it is slightly shifted to the higher temperatures region compared with the maximum strengthening (see Fig. 1c and d). However, the material remains relatively ductile, the total elongation does not fall below 20%, and the fracture surfaces of tensile specimens are ductile in spite of overageing at high ageing temperatures (650–750 °C). The typical fracture surface of CuCrZr alloy aged at 750 °C for 30 min is shown in Fig. 2.

The thermal stability of solution annealed and aged CuCrZr alloy under overheating is demonstrated in Fig. 3. This shows the changes in ultimate strength and yield strength of optimally solution annealed and aged CuCrZr alloy during additional ageing at 650 °C for times varying from 1 to \sim 70 min. The results show that optimally aged material retains higher strength under additional ageing at 650 °C, but this phenomena disappears



Fig. 2. Typical fracture surface of CuCrZr alloy after RT tensile testing (specimen was solution annealed and aged at 750 °C for 30 min).

with increasing exposure time at the ageing temperature. The conclusion is that the solution annealed



Fig. 3. Overageing effect on the RT tensile strength of CuCrZr alloy with two types of heat treatments: SA and SA + age at 480 $^{\circ}$ C, 1 h. (Anneal temperature is 650 $^{\circ}$ C, exposure is shown in figure).

and aged CuCrZr could be used for the manufacturing of ITER HHF components by brazing (i.e. that overheating during brazing will still result in acceptable properties). The allowable brazing time to retain adequate strength significantly decreases with increasing of temperature. The difference of greater than 50 MPa in strength of solution annealed plus aged material, compared with solution annealed material, remains for ageing time up to almost 20– 25 min at the temperature of 650 °C. The higher strength of solution annealed plus aged material is retained just for a few minutes, ~2–5 min, for ageing or brazing at 750 °C. (These data are not shown in figure.)

The effect of 'slow' cooling rate following the solution anneal heat treatment on the tensile



Fig. 4. Comparison of ageing effect on RT tensile strength of CuCrZr alloy solution annealed at 970 °C with the same alloy additionally annealed at 1000 °C for 30 min and cooled in water while encapsulated (1–3 °C/s).



Fig. 5. Correlations of Brinell hardness with RT yield and ultimate tensile strength of CuCrZr alloy.

strength of aged material is shown in Fig. 4. Microstructural observations reveled significantly more Cr precipitates in comparison with fast cooled material. The extra precipitates resulted in higher initial strength of material annealed in capsule at 1000 °C compared to material solution annealed at 970 °C. After ageing at 500 °C with exposure times exceeding ~20 min, higher strength was observed compared to the initially solution annealed alloy. However, the difference in achievable strength after ageing is not valuable. If manufacturing of HHF components includes a solution anneal procedure with cooling rate of about 1–3 °C/s, the subsequent ageing will result in some decreasing of strength, but the loss of strength will not be a critical.

Measurements of Brinell hardness established a correlation between hardness and tensile strength at RT. Linear functions were used to best fit the experimental data. These correlation results are presented in Fig. 5. The linear equations given in the figure can be used with the hardness measurements for quality control and properties prediction of CuCrZr alloy after the manufacturing cycles of HHF components.

4. Conclusion

The effects of ageing temperature in the range of 350-650 °C and times from 1 min to 3 h on the tensile strength and ductility have been studied. The *T*-*T* diagram has been plotted using 3D statistical fitting of experimental data. *T*-*T* diagrams can be used for tensile properties prediction of CuCrZr if solution annealed at 970 °C is used and if manufacturing heat treatments (brazing, for example)

are combined with ageing procedures or if ageing heat treatment are applied after manufacture.

The strengthening of CuCrZr alloy occurs faster at higher temperatures, with subsequent loss of strength for longer times. The most valuable changes in strength due to age hardening occur during the first 3–20 min. However, the CuCrZr alloy is not very sensitive to overageing. Strength and ductility remains at a relatively high level even after ageing at 650 °C. This allows exploring the advantages of high temperature manufacturing processes (for example brazing).

If initially solution annealed and aged at the most favorable temperature and time (480 °C, 2 h), CuCrZr alloy maintains better thermal resistance compared to solution annealed material. This conclusion was based on tensile data of alloy aged at 650 °C.

A slow cooling rate following solution anneal results in material strengthening under cooling that is probably caused by chromium precipitations. This heat treatment can be realized if the manufacturing route includes high temperature exposure (i.e. solution anneal temperature ~ 1000 °C) and cooling of large components, so that fast cooling is not achievable.

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