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Influence of the manufacturing heat cycles on the CuCrZr properties

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Abstract

The use of CuCrZr in the proper aged condition is hindered by the manufacturing process of components, which may involve several high temperature steps, which could then degrade the thermo-mechanical properties. To address this issue the European ITER Party have carried out a R&D programme aimed at assessing the effects of different bonding heat cycles. To this aim, CuCrZr in the solution-annealed-water quench condition was subjected to different heat treatments at various temperatures and holding times. The main outcome was that aging temperatures up to 550 °C could still guarantee a tensile strength of 300 MPa. Higher joining temperatures (up to 600–650 °C) require a certain amount of cold work after the annealing process and prior to the joining cycle in order to achieve an acceptable strength. Up to these temperatures the thermal conductivity is not degraded.

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1. Introduction

The precipitation hardened copper–chromium–zirconium (CuCrZr) alloy reaches an optimum in strength after a thermo-mechanical treatment involving first a solution annealing at high temperature (above 960 °C) to dissolve the alloying elements, then a water quench to keep the alloying elements in supersaturated solid solution at room temperature, and finally an aging treatment at intermediate temperatures to decompose the supersaturated solid solution into a fine distribution of precipitates. After solution annealing, the CuCrZr can be cold worked to further improve the final mechanical properties.

The use of CuCrZr in the proper aged condition, however, is hindered by the manufacturing process of plasma facing components (PFCs), which may involve high-temperature steps, which could then degrade the thermo-mechanical properties.

To address this issue the European ITER Party have carried out a R&D programme aimed at assessing the effects of different bonding heat treatments. The adopted strategy was to combine the aging heat treatment with the hot isostatic pressing (HIP) manufacturing step. The basic idea was to use the CuCrZr alloy in the solution-annealed + water quench condition and have it aged during the HIP process.

The optimum aging cycle should be performed at 475 ± 5 °C for 3 h; on the other hand higher temperatures are preferred for a reliable HIP joint. The proper optimisation of this combined HIP and aging process was the objective of this work.

To this aim, CuCrZr in the solution-annealed-water quench condition was subjected to different heat treatments at various temperatures (475, 500, 550, 600, 700 °C) and holding times (5, 10, 20, 30, 40, 60, 120, 180, 240, 300, 360 min).

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The present paper outlines the thermal and mechanical properties (namely: hardness, thermal conductivity and tensile strength) of CuCrZr after different manufacturing cycles. A discussion on the possible manufacturing routes is also presented based on the results obtained.

2. Study on the manufacturing heat cycles

The basic idea was to use the CuCrZr alloy in the solution-annealed + water quench condition and have it aged during the HIP joining step.

The CuCrZr material, which was used in this study, was ELBRODUR grade made by Kabelmetall Company in the following forms:

- round bar ∅ 25 mm for the tensile test, thermal diffusivity measurements and thermal capacity measurements;
- round bar ∅ 30 mm for the hardness test.

It was supplied in the solution-annealed + water quenched + cold worked + aged condition ('as-received' condition). This condition represents the best thermomechanical properties, which can be obtained with this material.

Vickers hardness (HV) tests were carried out at room temperature in the as-received condition according to the ASTM 92-82 rules, using a load with a mass of 30 kg. The results are reported in Table 1. Three tests were performed on two different samples. Each number is a mean value over five indents. The estimated tensile strength (UTS) was computed with the following formula:

$$HV \leqslant 136 \rightarrow UTS = HV \times 3.136 + 29.4 \text{ MPa},$$

$$136 \leqslant HV \leqslant 350 \rightarrow UTS = HV \times 3.352 \text{ MPa}.$$

The above empirical relationship was developed for steel and was found here to give a good estimation also for the tensile strength of CuCrZr. The samples to be used in the tests were subjected to the following re-solution thermal treatment to obtain a material ready for the aging treatment ('solution-annealed + water quench' condition):

- heating from 20 to 970 °C within 90 min;
- steady state at 970 °C for 20 min;
- water quench.

The solution-annealed + water quench condition represents the typical starting point of any manufacturing process for PFCs. Hardness tests were carried out in this condition and are reported in Table 2. Three tests were performed on each sample. Each number is a mean value over five indents.

The possible HIP joining cycle was simulated by a number of different heat treatments performed at different temperatures and with different hold times. These thermal treatments were performed in an inert fan furnace having a temperature precision of ±1 °C and using the following procedure:

- heating from 20 °C up to the set temperature with an increasing rate of 5 °C/min;
- steady state at the set temperature for the required time;
- air cooling out of the furnace.

Hardness tests were carried out to assess the mechanical properties obtained after each treatment. Three tests were performed on three different samples per each treatment. Each test consisted of five indents. Table 3 reports the overall average of hardness after each heat treatment.

Some heat treatments (namely at temperature 500, 550 and 600 °C and different holding times) were selected among those that showed the most relevance for a manufacturing process. On these selected heat treatments a more detailed characterisation was performed. Table 4 summarises the results of the thermal conductivity and tensile tests. The thermal conductivity was derived by thermal diffusivity measurements obtained by

Table 1
HV of CuCrZr at room temperature in the as-received condition

Test	Sample 1	Sample 2
1	159	160
2	157	157
3	161	161
Average	159	159
Overall average	159	
Estimated tensile strength (MPa)	533	

Table 2
HV of CuCrZr at room temperature in the solution-annealed + water quench condition

Test	Sample 1	Sample 2	Sample 3	Sample 4
1	66	77	67	66
2	67	75	65	66
3	67	77	65	65
Average	67	76	66	66
Overall average		69		
Estimated tensile strength (MPa)		246		

Table 3
HV of CuCrZr at room temperature after different heat treatments, starting condition: solution-annealed + water quench

Hold time (min)	Temperature (°C)				
	475	500	550	600	700
5			108	107	77
10			113	104	75
20			117	100	70
30		125			
40			111	93	68
60	123	110		93	64
120	125	104	97	88	62
180	130	115	106	90	
240	120	111			
300	114	112			
360	119				

means of the laser flash technique. Each number is a mean value over three tests. As regards tensile tests, each number is a mean value over five tests. The thermal conductivity increases with progressive aging and overaging of the material, as expected. The tensile strength, after having reached the maximum at the reference aging heat treatment, decreases progressively with the material overaging.

To evaluate the contribution of cold work to the final mechanical properties, hardness tests were also carried out with CuCrZr starting from the as-received condition and then subjected to different heat treatments. Three tests were performed on three different samples per each treatment. Each test consisted of five indents. Table 5 reports the overall average of hardness after each heat treatment. One can notice that even when no hold time is applied (0 min) a decrease of hardness is observed. This is due to a certain degree of overaging of the CuCrZr during the heating and cooling phase.

Table 5
HV of CuCrZr at room temperature after different heat treatments, starting condition: as-received

Hold time (min)	Temperature (°C)		
	550	600	700
0	156	145	115
20	142	126	103
40	139	124	101
60	135	122	99
120	132		
180	133		
240	128		

3. Discussion of the results and conclusions

The reference aging treatment for CuCrZr is 3 h at 475 °C and in fact the highest hardness of 130 HV was obtained exactly after the same thermal cycle. This value is lower than that of the as-received condition (159 HV), the difference of $\simeq$30 HV is due to the contribution of the cold work which is a non-recoverable loss after the solution-annealed + water quench treatment. Furthermore, starting from the as-received condition the hardness after 60 min at 700 °C is equal to 99 HV. Starting from the solution-annealed + water quench condition the hardness after 60 min at 700 °C is equal to 67 HV. This confirms that the contribution of the cold work can be estimated in about 30 HV (that is about 100 MPa at room temperature). Assuming a minimum acceptable value of 100 HV for the mechanical strength of the CuCrZr, which should guarantee a tensile strength in excess of 300 MPa at room temperature, a HIP temperature as high as 550 °C would be acceptable.

The contribution of the cold work prior to HIP could open the way to a further increase of the manufacturing temperature up to 600–650 °C provided that the starting

Table 4
Yield stress, tensile strength and thermal conductivity measured after different aging treatments (starting condition: solution-annealed + water quench)

Heat treatment	Test temperature (°C)					
	20		100	400		
	$\sigma_{0.2}$ (MPa)	σ_{UTS} (MPa)	λ (W/m K)	$\sigma_{0.2}$ (MPa)	σ_{UTS} (MPa)	λ (W/m K)
None			225.4			257.1
500 °C, 60 min			315.6			331.2
500 °C, 120 min	191	348	336.3	161	192	344.4
500 °C, 180 min			325.4			336.7
550 °C, 60 min			332.9			341.5
550 °C, 120 min	166	295	339.0	130	181	344.1
550 °C, 180 min			338.7			343.3
600 °C, 60 min			328.8			336.7
600 °C, 120 min	142	278	335.9	103	153	342.6
600 °C, 180 min			343.4			341.5

Table 6
Possible manufacturing strategies for PFCs

Strategy 1	Strategy 2	Strategy 3
CuCrZr in any condition	CuCrZr solution-annealed + water quench	CuCrZr solution-annealed + water quench + cold work
Armour/heat sink joining at solution annealing temperature (>960 °C for more than 30 min) Fast cooling with rate higher than 1 K/s down to 400 °C Aging treatment	HIP at a temperature up to 550 °C	HIP at a temperature up to 600–650 °C

condition of the CuCrZr material is solution-annealed + water quenched + a proper amount of cold work. The drawback of this strategy is that the joining between the heat sink and the armour is performed when the heat sink has already a certain mechanical strength. In monoblock geometry this may cause high residual stresses that may break the armour tile. For this geometry, it could be therefore preferred to join the armour to a cooling tube that is as soft as possible. This is the case for the CuCrZr in the solution-annealed + water quench condition.

The optimum aging of CuCrZr is obtained if this material is water quenched after annealing. The fast cooling rate is required to prevent the overaging of the material already during the cooling phase. When this is avoided, then it is possible to obtain a fine distribution of Cr and Zr precipitates during the following aging treatment. This fine distribution guarantees the best mechanical properties. A previous study was carried out at the Joint Research Centre at Ispra aimed at evaluating the minimum cooling rate required from the annealing temperature. In that work it was demonstrated that a successful aging of the CuCrZr can only be achieved if the cooling rate is at least 2 K/s from 970 to 870 °C and then proceeds faster than 1 K/s [1–3]. This result opens the way to another possible manufacturing strategy, that is performing the joining step at the annealing temperature followed by sufficient fast cooling of the component. The proper mechanical properties for CuCrZr can then be achieved by a final aging heat treatment.

Table 6 summarises the most promising strategies to manufacture PFCs while achieving sufficient high thermo-mechanical properties in the CuCrZr material. Three of them are identified depending on the starting conditions of CuCrZr and on the joining temperatures. All of them should guarantee a final tensile strength of CuCrZr in excess of 300 MPa at room temperature as well as an optimum thermal conductivity.

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