EFFECT OF HEAT TREATMENTS ON THE TENSILE AND ELECTRICAL PROPERTIES OF HIGH-STRENGTH, HIGH-CONDUCTIVITY COPPER ALLOYS —

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OBJECTIVE

The objective of this report is to summarize recent tensile and electrical resistivity measurements on several different unirradiated commercial high-strength, high-conductivity copper alloys that are being considered for the divertor structure and first wall heat sink in ITER.

SUMMARY

The unirradiated tensile properties of CuCrZr produced by two different vendors have been measured following different heat treatments. Room temperature electrical resistivity measurements were also performed in order to estimate the thermal conductivity of these specimens. The thermomechanical conditions studied included solution quenched, solution quenched and aged (ITER reference heat treatment), simulated slow HIP thermal cycle (~1°C/min cooling from solutionizing temperature) and simulated fast HIP thermal cycle (~100°C/min cooling from solutionizing temperature). Specimens from the last two heat treatments were tested in both the solution-cooled condition and after subsequent precipitate aging at 475°C for 2 h. Both of the simulated HIP thermal cycles caused a pronounced decrease in the strength and electrical conductivity of CuCrZr. The tensile and electrical properties were unchanged by subsequent aging in the slow HIP thermal cycle specimens, whereas the strength and conductivity following aging in the fast HIP thermal cycle improved to ~65% of the solution quenched and aged CuCrZr values. Limited tensile and electrical resistivity measurements were also made on two new heats of Hycon 3HP CuNiBe. High strength but poor uniform and total elongations were observed at 500°C on one of these new heats of CuNiBe, similar to that observed in other heats.

PROGRESS AND STATUS

Introduction

In a previous semiannual report [1], data from room temperature electrical resistivity measurements and elevated tensile test measurements at several different strain rates were summarized for wrought dispersion strengthened copper (Cu-Al_2O_3) and solutionized and aged CuCrZr and CuNiBe. Since the mechanical and electrical properties of precipitation strengthened alloys such as CuCrZr and CuNiBe are known to be sensitive to heat treatment conditions [1-5], the objective of the present study was to quantify the degradation in electrical conductivity and tensile strength which may occur under some heat treatment conditions. In particular, this study focuses on CuCrZr subjected to several different heat treatment cycles. The current reference design for the International Thermonuclear Experimental Reactor (ITER) utilizes a hot isostatic press (HIP) technique to join the Cu alloy heat sink to the stainless steel structure in the first wall [6]. The joining is performed at a temperature of ~950°C, and the cooling time to reach ~400°C after joining is estimated to be ~1 h due to the large size of the first wall panels. A similar HIP technique is envisioned for fabricating the Cu/W/carbon-carbon composite monoblocks for the divertor. However, higher cooling rates of ~100 to 200°C/min may be achievable with gas cooling on the inside of the Cu tube for these components [6]. The effect of two different HIP thermal cycle heat treatments (furnace cool and gas cool) on the properties of CuCrZr are summarized in this report. The effects of subsequent precipitate aging heat treatments were also studied. Finally, the results of limited tests on two new heats of Hycon 3HP™ CuNiBe are reported and compared to previous results obtained on other CuNiBe heats.

Table 1. Summary of Heat Treatments for CuCrZr Specimens

Alloy and heat treatment	Solutionizing treatment	Aging treatment
Zollern CuCrZr, heat Z822		
solution quenched (as-received)	970°C, 20 min./water quench	---
solution quenched & aged	970°C, 20 min./water quench	475°C, 2 h
HIP furnace cool	980°C, 1 h/ furnace cool (~6 h to cool to 400°C)	---
HIP furnace cool & aged	980°C, 1 h/ furnace cool	475°C, 2 h
HIP fast cool	980°C, 1 h/ gas cool (~100°C/min cooling rate)	---
HIP fast cool & aged	980°C, 1 h/ gas cool	475°C, 2 h
Kabelmetal CuCrZr,heat AN4946		
ITER solution quenched & aged	980°C, 1 h/ water quench	475°C, 2 h

Experimental Procedure

Most of the work reported here was performed on bar stock of CuCrZr that was received in a solution quenched condition. The bar was manufactured by Zollern GmbH, Laucherthal, Germany (heat Z822) with a reported composition of 0.85 wt%Cr, 0.09 wt%Zr and <0.001 wt%P. The bar was solutionized for 20 minutes at 970°C and water quenched. The short solutionizing time and slightly lower solutionizing temperature compared to typical CuCrZr solutionizing conditions was specified by Joint European Tokamak (JET) designers in order to minimize the grain size of the final product. Miniature SS-3 sheet tensile specimens with nominal gage dimensions 0.76 mm × 1.5 mm × 7.6 mm were electro-discharge machined from the bar and subsequently heat treated at several different conditions which are summarized in Table 1. The HIP solutionizing treatments were performed in flowing Ar, and all of the aging treatments were performed in flowing He.

The Kabelmetal Cu-0.65%Cr-0.10%Zr specimens were obtained from a 2 cm thick plate that was originally fabricated under the trade name of Elbrodur G by KM-Kabelmetal, Osnabrück, Germany as an F37 (cold-worked and aged) temper, heat #AN4946. A 2 × 3 × 5 cm piece from this plate was solution annealed in flowing argon for 1 hour at 980°C, water quenched, then aged in flowing helium at 475°C for 2 hours (furnace cool) at ORNL, in accordance with the draft ITER heat treatment specifications. Miniature SS-3 sheet tensile specimens were subsequently machined from the solutionized and aged plate.

Two different heats of Hycon 3HP™ CuNiBe produced by Brush-Wellman were investigated. Heat #35562 was fabricated as 3.2 mm thick strip in a cold-worked and aged (TH04 temper) condition. The reported solute composition for this heat was 1.56%Ni, 0.25%Be, 0.1%P. Heat #28626 was supplied in two different heat treatments, and had a reported solute composition of 1.90%Ni, 0.32%Be, 0.1%P. The HT (TH04 temper) material from heat #28626 was supplied as 0.89 mm thick strip. A thicker plate of HT temper material from this heat was reprocessed by Brush-Wellman to the AT (solutionized, air-quenched and aged) condition. The AT heat treatment consisted of solutionizing at 932°C for 1 h (gas quench) followed by aging at 510°C for 3 h. The AT material was fabricated into sheet tensile specimens with an overall length of 12.2 cm and a thickness of 3.2 mm. Miniature SS-3 sheet tensile specimens were cut from the grip regions of these large tensile specimens after they had been tensile tested by McDonnell-Douglas in an induction furnace system at 500°C. Since the 8 min. hold time at 500°C for the tensile tests on the large sheet tensile specimens was much less than the aging time of 3.5 h at 510°C, the precipitate structure in the grip regions should be similar to that of the as-fabricated AT material.

Four-point probe electrical resistivity measurements were performed at room temperature on a total of 2 to 5 different SS-3 sheet tensile specimens for each of the heat treatment conditions, using procedures summarized elsewhere [7]. The temperature was recorded for each measurement and the resistivity data were corrected to a reference temperature of 20°C using the copper resistivity temperature coefficient of $d\rho/dT = 6.7 \times 10^{-11}$ Ω-m/K. Nonuniformities in the width and thickness in the specimen gage region caused the typical experimental uncertainty of individual resistivity measurements to be $\pm$0.5%. The relation 17.241 nΩ-m = 100% IACS (international annealed copper standard) was used to convert the resistivity measurements to electrical conductivity values.

The tensile properties of the SS-3 sheet tensile specimens were measured at room temperature or 500°C at a crosshead speed of 0.0085 mm/s (with the exception of the Kabelmetal CuCrZr specimen which was tested at 0.017 mm/s), which corresponds to initial strain rates of 1.1×10^{-3} and 2.2×10^{-3} in the gage region, respectively. The room temperature tests were performed in air, and the elevated temperature tests on CuNiBe were performed in vacuum (10^{-6} to 10^{-5} torr). Two different heating cycles were used for the 500°C tests on CuNiBe in order to determine if the tensile properties degraded after holding at the test temperature. One specimen was rapidly heated to 500°C (heating time <8 minutes) and tensile tested as soon as it reached the test temperature. A second specimen was heated to 500°C over a period of ~0.5 h and subsequently held at the test temperature for 0.25 h prior to the start of each tensile test. One specimen was tested in an Instron servohydraulic machine for each experimental condition. The tensile properties were determined from graphical analysis of the chart recorder curves. A plastic deformation offset of 0.2% was used for measuring the yield strength.

Results

Table 2 summarizes the results of the room temperature electrical resistivity measurements. All three of the CuNiBe thermomechanical conditions had electrical conductivities near 67% IACS. This conductivity is comparable to that previously measured [1,7] in other heats of Hycon CuNiBe (64-72% IACS). As expected, the solution quenched CuCrZr had a low conductivity of ~36% IACS. The Kabelmetal solution quenched and aged CuCrZr had a superior electrical conductivity compared to that of Zollern solution quenched and aged CuCrZr (83% vs. 76% IACS), presumably due to the short solutionizing time and lower solutionizing temperature along with the higher Cr concentration for the Zollern material. The HIP slow furnace cool condition produced a modest degradation in the electrical conductivity of the Zollern CuCrZr compared to the solution quenched and aged condition (~68% vs. 76% IACS). The conductivity remained slightly degraded following aging.

The tensile properties obtained in the present study are summarized in Table 3. Previous tensile tests on the AT CuNiBe (heat 28626) material performed at 500°C using large sheet tensile specimens indicated that the uniform and total elongations were 3.1 to 6.1% [8]. The present tensile tests, performed at 500°C with miniature SS-3 type sheet tensile specimens, produced yield strengths that were in good agreement with the large sheet tensile specimen results. However, the measured uniform and total elongations were significantly smaller than that reported for the larger specimens. This indicates that the small size of the SS-3 sheet tensile specimens (~10 to 20 grains across the gage thickness) accentuates the intergranular deformation mode that occurs in CuNiBe [1,7] for elevated test temperatures. There did not appear to be a pronounced effect of hold time at 500°C on the tensile properties. Although the tensile test performed with the shorter hold time had a somewhat higher elongation, in both cases the total elongation was well below 2%.

Table 2. Room Temperature Electrical Properties Measured in the Present Study (see Table 1 and text for detailed heat treatments)

Alloy and heat treatment	Meas. resistivity at 20°C	Electrical conductivity
Zollern CuCrZr, heat Z822		
solution quenched	48.29 nΩ-m	35.7% IACS
solution quenched & aged	22.55 nΩ-m	76.4% IACS
HIP furnace cool	25.41 nΩ-m	67.9% IACS
HIP furnace cool & aged	24.94 nΩ-m	69.1% IACS
HIP fast cool	39.57 nΩ-m	43.6% IACS
HIP fast cool & aged	25.91 nΩ-m	66.5% IACS
Kabelmetal CuCrZr, heat AN4946		
ITER solution quenched & aged	20.67 nΩ-m	83.4% IACS
Hycon 3HP CuNiBe		
solution quenched & aged (AT), heat 28626	25.62 nΩ-m	67.3% IACS
cold-worked & aged (HT), heat 28626	25.45 nΩ-m	67.7% IACS
cold-worked & aged (HT), heat 35562	25.41 nΩ-m	67.9% IACS

Table 3. Summary of Tensile Properties Measured in the Present Study (see Table 1 and text for detailed heat treatments)

Alloy and heat treatment	Temperature	σ_y (MPa)	UTS (MPa)	e_u (%)	e_{tot} (%)
Zollern CuCrZr					
solution quenched	20°C	118	243	42	52
solution quenched & aged	20°C	310	418	17	25
HIP furnace cool	20°C	—	—	—	—
HIP furnace cool & aged	20°C	68	217	23	29
HIP fast cool	20°C	37	216	56	61
HIP fast cool & aged	20°C	206	328	18	23
Kabelmetal CuCrZr					
solution quenched & aged	20°C	316	414	16	24
Hycon CuNiBe, heat 28626					
solution quenched & aged (AT)	500°C*	399	414	0.6	0.6
solution quenched & aged (AT)	500°C**	414	445	1.3	1.3

*heated to 500°C within ~30 min. and held at temperature for 15 min. prior to starting tensile test
**heated to 500°C within ~ 8 min.; tensile test started immediately after reaching 500°C.

The solution quenched and aged heat treatments for the Zollern and Kabelmetal CuCrZr produced comparable room temperature tensile properties, with yield strengths of ~310 MPa, ultimate strengths of ~420 MPa and uniform elongations of ~16%. The slow cooling rate associated with the HIP furnace cool treatment resulted in a very low yield strength even after subsequent aging. It is interesting to note that the yield strength for the HIP furnace cool and aged condition was even lower than that for the solution quenched condition. A very low yield strength was also obtained in the HIP fast cooled specimens. In this case, however, the room temperature strength could be increased to 200-210 MPa (i.e., ~65% of the solution quenched and aged yield strength) by subsequent aging.

Discussion

Table 4 compares the room temperature thermal stress figures of merit, $M=\sigma_y k_{th}(1-\nu)/\alpha E$, for CuCrZr in various heat treatment conditions with other high-strength, high conductivity copper alloys. The M values were calculated using pure copper data [9] for Young's modulus (E), Poisson's ratio (v) and the coefficient of thermal expansion (α), and by utilizing the Wiedemann-Franz relation to convert the electrical conductivity measurements to thermal conductivity (k_{th}). The HIP furnace cool and aged specimens exhibited a very low thermal stress figure of merit, although this parameter may not be the most appropriate guidepost if moderate amounts of deformation can be tolerated in the design. The CuCrZr thermal stress figure of merit for the HIP fast cool and aged condition is ~60% of the solutionized and aged value. This degradation in strength and thermal conductivity must be considered in designs for the divertor structure that use CuCrZr. A similar moderate degradation in strength has been reported for CuNiBe following a HIP furnace cool heat treatment [5]. The electrical conductivity was not changed within the accuracy of the experimental measurement in the study by Singh, et al. Dispersion strengthened copper did not appear to be degraded by a HIP furnace cool heat treatment [5].

Table 4. Thermal Stress Figures of Merit for High-Strength, High Conductivity Copper Alloys

Alloy and thermomechanical condition	M at 20°C (kW/m)
Kabelmetal CuCrZr (ITER solution quenched & aged)	33
Zollern CuCrZr (solution quenched & aged)	30
Zollern CuCrZr (HIP furnace cool & aged)	6
Zollern CuCrZr (HIP fast cool & aged)	17
GlidCop Al25 (IG0)	36
Hycon 3HP CuNiBe (AT, heat 46546)	45
Hycon 3HP CuNiBe (HT, heat 46546)	60

Several relevant studies on the time-temperature-transformation (TTT) kinetics of precipitation in Cu-Cr alloys have been published [2-4]. The key finding from these studies is that cooling rates >20°C/s are necessary to fully quench the Cu-Cr solid solution. Additional relevant studies such as quenching to intermediate temperatures, followed by conventional aging heat treatments were also performed. Considering the estimated cooling rates following HIP joining procedures for ITER first wall and divertor components of ~0.1-0.2°C/s and ~2°C/s, respectively [6], it may be concluded that the Cr solute will not be quenched and therefore inferior properties compared to water-quenched and aged specimens will occur (as observed in the present study).

As demonstrated in Tables 2 and 3, the degradation in conductivity and strength is particularly severe in the as-cooled CuCrZr specimens that have been subjected to the HIP thermal cycle. Subsequent aging does not have a significant effect on the properties of the HIP furnace cooled specimens, whereas a substantial improvement in strength and conductivity occurs in the HIP fast cooled specimens. Therefore, a post-joining heat treatment at ~475°C should be used for CuCrZr alloys fabricated into divertor monoblocks, where the cooling rate after HIPping is relatively fast. On the other hand, a post-joining heat treatment is not recommended for the large first wall HIPped components, since no improvement in properties is expected.

A recent study on CuCrZr subjected to a HIP furnace cool and aged heat treatments reported a modest degradation in the yield strength and no significant change in electrical conductivity compared to solution quenched and aged specimens [5]. A much larger strength degradation for HIP furnace cooled + aged CuCrZr specimens was observed in the present study (Table 3). The source of this discrepancy can be understood by noting that the room temperature yield strength of the solutionized and aged specimens in the previous study [10] was only ~30% that of the solutionized and aged specimens in the present study.

The effect of neutron irradiation on the tensile and electrical properties of these CuCrZr alloys in different heat treatment conditions will be studied in a series of 3 reactor irradiation experiments that are scheduled to begin in the summer of 1997. Further details on this irradiation experiment will be given in the next semiannual report volume.

ACKNOWLEDGEMENTS

The Zollern CuCrZr bar used in this study was supplied by George Kalinin (ITER-Garching joint work site) via Kevin Slattery (McDonnell-Douglas). The Kabelmetal CuCrZr plate was supplied in the F37 temper condition by K. Slattery, McDonnell-Douglas. The two Hycon 3HP™ CuNiBe heats were supplied by K. Slattery, McDonnell-Douglas.

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