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Fracture toughness of copper-base alloys for fusion energy applications

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Abstract

Oxide-dispersion strengthened copper alloys and a precipitation-hardened copper–nickel–beryllium alloy showed a significant reduction in toughness at elevated temperatures (250°C) as compared to room temperature. This decrease in toughness was much larger than would be expected from the relatively modest changes in the tensile properties over the same temperature range. However, a copper–chromium–zirconium alloy strengthened by precipitation showed only a small decrease in toughness at the higher temperatures. The embrittled alloys showed a transition in fracture mode, from transgranular microvoid coalescence at room temperature to intergranular with localized ductility at high temperatures. The Cu–Cr–Zr alloy maintained the ductile microvoid coalescence failure mode at all test temperatures.

1. Introduction

High-strength copper alloys with high thermal conductivity are attractive candidates for some structural applications in ITER. Several classes of copper-based alloys are being examined to determine their fracture toughness at room temperature and up to 250°C.

The first class of copper alloys is dispersion strengthened by internal oxidation. One version of this alloy, called GLIDCOP AL-15, contains 0.15 wt% aluminum that has been internally oxidized to produce small Al_2O_3 particles in a copper matrix. Testing of unirradiated material was conducted to determine the fracture toughness as a function of test temperature and specimen orientation. Preliminary testing [1] had shown that the toughness of the AL-15 material decreased significantly as the test temperature increased from 22°C to 250°C, although the tensile properties showed only a slight change over the same temperature range [2]. This suggested that an environmental effect might be responsible for the decrease in toughness at higher temperatures. Therefore, tests were carried out in vacuum to determine whether this could mitigate the decrease in toughness observed at higher temperatures. A second version of this alloy, GLIDCOP AL-25, which contains 0.25 wt% aluminum, was also examined.

A second class of alloys are strengthened by precipitation. Two alloys are being studied: Cu–Ni–Be [nominal composition Cu–2Ni–0.35Be (wt%)] and Cu–Cr–Zr (nominal composition Cu–0.65Cr–0.15Zr).

2. Experimental procedure

The fracture toughness testing was conducted with small disk compact specimens 12.5 mm in diameter by 4.62 mm thick (0.491 by 0.182 in.) [designated 0.18 T DC(T) specimens]. All specimens were fatigue precracked at room temperature and then side grooved 10% of their thickness on each side prior to testing. Testing was conducted on an 89-kN (20-kip) capacity servohydraulic test machine in laboratory air, or on a 223-kN (50-kip) servohydraulic machine equipped with a vacuum chamber. The vacuum tests were conducted with a vacuum of better than 10^{-6} torr. A thermocouple was spot-welded to the specimen to monitor the temperature during each test. The *J*-integral–resistance (*J*–*R*) curves were obtained, in general accordance with American

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Society for Testing and Materials (ASTM) E 813–89, Standard Test Method for J_{IC}, A Measure of Fracture Toughness, and ASTM E 1152–87, Standard Test Method for Determining *J–R* Curves, using a computer-controlled data acquisition and analysis system operating in strain control. The *J*-integral equations from E 1152–87 were used for the calculations. Tensile properties used in the analyses were taken from the literature [2–4].

Crack growth was monitored by the unloading compliance technique for all tests. An outboard clip gage was used that was seated in grooves machined on the outer diameter of the disk, above and below the loading holes. The experimental techniques developed for testing the small DC(T) specimens have been described elsewhere [5].

To mark the extent of crack growth for some of the preliminary testing the specimens were heat tinted by placing them on a hot plate and heating them until a noticeable color change had occurred. The specimens were cooled to room temperature and then broken open to allow the initial and final crack lengths to be measured. Later tests used fatigue crack extension at room temperature after the tests were completed to mark the final crack front. The crack lengths were measured from the fracture surfaces with a measuring microscope.

Specimens of the AL-15 material were fabricated from the middle of the thickness of an as-wrought plate measuring 165 mm wide by 12.7 mm thick by approximately 3 m long that was produced by SCM Metal Products for the ORNL Fusion Energy Division in 1987 [3]. This plate had been warm worked during the consolidation of the −20 mesh powder. The plate was then extruded at about 820°C with an extrusion ratio of 25 : 1. Specimens were oriented in the T–L orientation so that crack growth was in the extrusion direction, or in the L–T orientation for crack growth perpendicular to the extrusion direction.

The AL-25 material was also fabricated by SCM Metal Products. The heat number was C-8064, and the material is referred to as IG0 (ITER Grade 0). The disk compact specimens were fabricated in stacks of four through the thickness of the plate, in either T–L or L–T orientations.

The Cu–Ni–Be alloy (C17510, trademark name Hycon 3 HP) was supplied by Brush-Wellman. Two heats were supplied in different strengths, primarily due to differences in their HT temper (cold-worked and aged) heat treatments. The high-strength heat 33667Y1 had a yield strength of about 720 MPa (104 ksi) at room temperature and an electrical conductivity of ∼ 66% IACS. A second heat 46546AA2 in a slightly lower strength condition had a room-temperature yield strength of about 620 MPa (90 ksi) with a corresponding higher electrical conductivity of 72% IACS [4]. Specimens from both these heats were fabricated in stacks of five through the thickness of the nominally 25 mm thick (1 in.) plates, in either the T–L or the L–T orientation, for crack extension parallel or perpendicular to the rolling direction, respectively.

The disk compact specimens prepared from the lower-strength heat 46546AA2 could not be successfully fatigue precracked. In all cases, the cracks deflected out of the plane of the starter notch, and began to grow perpendicular to the notch soon after they had initiated. This occurred for both specimen orientations, and persisted even when some specimens were side-grooved before precracking was attempted. This tendency for the crack to deflect out-of-plane has been observed previously for this material, in the L–T orientation [6,7]. The specimens from the high-strength material, heat 33667Y1, precracked readily, in either specimen orientation. Thus, all the data reported for alloy C17510 are from this high-strength condition and heat only.

The Cu–Cr–Zr alloy, called Elbrodur N, was provided by McDonnell-Douglas Aerospace in the form of plate 20.3 mm (0.8 in.) thick. The material was produced by KobelMetall in the T37 temper (yield strength of 370 MPa) by solution annealing at 950–1000°C, water quench, cold work, and aging at 425–500°C. The specimens were fabricated as stacks of four through the thickness of the plate, in either the T–L or L–T orientations.

3. Results and discussion

The results of the fracture toughness testing are shown in Fig. 1 and in Table 1. For the dispersion-strengthened AL-15 material, the fracture toughness decreased markedly as the test temperature increased. The toughness was also quite different depending on the specimen orientation, with specimens from the L–T orientation being much tougher than the T–L specimens. The toughness at room temperature was unaffected by the change from air to vacuum, but a higher toughness in vacuum than in air was observed at 250°C.

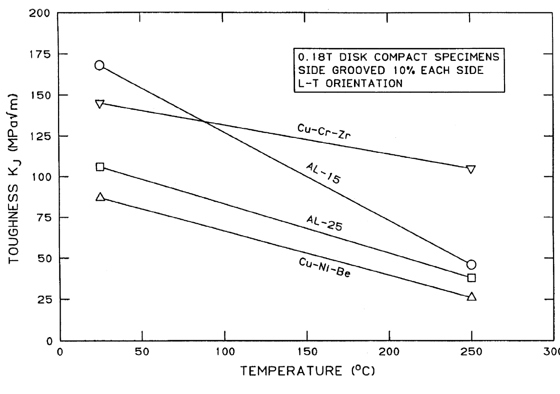

Fig. 1. Fracture toughness versus test temperature for the copper alloys.

Table 1
Fracture toughness of unirradiated copper alloys

Material	Specimen	Orientation	Temperature (°C)	J_Q (kJ m^{-2})	K_{JQ} (MPa $\sqrt{m}$)	Tearing modulus
GLIDCOP AL-15	FJ4	T–L	25	51	78	42
	FJ1		250	3	20	9
	GC1		250 vacuum	11	34	13
	GC5	L–T	25	241	168	87
	GC11		25 vacuum	220	161	75
	GC6		250	19	46	30
	GC7		250 vacuum	48	72	37
GLIDCOP AL-25	IG34	T–L	25	70	91	51
	IG35		250	7	28	16
	IG25	L–T	25	96	106	94
	IG26		250	13	38	26
Cu–Ni–Be	CL0	T–L	25	58	82	5
	CL2		150	23	51	4
	CT0	L–T	25	64	87	8
	CT6		250	6	26	1
Cu–Cr–Zr	CZ09	T–L	25	108	112	38
	CZ12		250	87	98	41
	CZ01	L–T	25	190	145	51
	CZ05		250	99	105	43

The increase in test temperature from 25°C to 250°C caused a significant decrease in the fracture toughness in either air or vacuum.

The significant decrease in the toughness of the oxide-dispersion strengthened alloys as the test temperature increases is a surprising response, as the change in the tensile properties over this same range of temperatures is modest (about 25%) [2]. These results are similar to toughness data reported by SCM Metal Products for the AL-25 alloy [6], a higher alloyed variant of oxide-dispersion strengthened copper which has 0.25 weight percent aluminum. Interestingly, impact tests of notched specimens of AL-25 [5] do not show a decrease in absorbed energy over a similar range of test temperatures. The fact that the toughness is degraded in the quasi-static fracture toughness test but not under dynamic conditions suggests that an environmental effect such as oxygen embrittlement of grain boundaries may be responsible for the drop in toughness at higher temperature. It was thought that the fracture toughness may not be so impaired in a vacuum environment. However, although there is a slight improvement in the toughness at 250°C under vacuum conditions as compared to air, the toughness is still much lower than one would expect on the basis of the small changes in the tensile properties over the same temperature range.

The results for GLIDCOP AL-15 show that the toughness of L–T specimens is much greater than that of T–L specimens. The processing used in the fabrication of this material results in the alignment of particles and the creation of an aligned grain structure parallel to the rolling or extrusion direction. Specimens in the T–L orientation will have crack extension parallel to this microstructure. This will result in a greatly reduced resistance to crack extension by providing a path that favors crack growth, whether by a ductile fracture mechanism, as will occur at room temperature, or by an intergranular mechanism, as may be occurring at high temperature. Preliminary fractography indicates this change in fracture mode occurs for the T–L specimens tested in air. Additional examination of the specimens tested in vacuum and in the L–T orientation is needed.

The AL-25 material also shows a decrease in toughness at the higher test temperature (see Table 1). Again, the material in the L–T orientation is tougher than in the T–L orientation, although the difference is not as great as in the AL-15 material. The IG0 version of AL-25 incorporates cross-rolling of the plate during the size reduction process. Therefore, the grain elongation is less pronounced in the longitudinal direction, as compared to the AL-15 alloy.

Fractographic examinations of the AL-15 and AL-25 specimens showed a ductile microvoid coalescence mode of fracture for specimens tested at room temperature, in air or vacuum. A pronounced stretch zone was present at the tip of the fatigue precrack. Testing at 250°C in air or vacuum resulted in what appeared to be an intergranular fracture, with no stretch zone at the crack tip. The fracture mode did not resemble classic intergranular fracture, likely due to the anisotropic structure produced during fabrication of the oxide-dispersion strengthened material. It may in fact be intersubgranular, but is clearly very different than the microvoid coalescence observed at room temperature.

The Cu–Ni–Be material also shows a decrease in toughness when tested at higher temperatures, even at only 150°C (Table 1). Again, the L–T orientation appears to be tougher than the T–L orientation, although only limited testing has been conducted. Fractographic examination showed that at room temperature the fracture mode was microvoid coalescence, but at 250°C the fracture was intergranular. However, the intergranular fracture surface produced during the high-temperature fracture was a ductile one, with the grain boundaries covered with small, shallow dimples, much smaller than the transgranular dimples that formed for the specimen tested at room temperature. A similar transition in fracture morphology from ductile transgranular to ductile intergranular was observed in tensile tests performed on this same heat of material, although the transition in the tensile specimens only occurred for test temperatures above 300°C [4].

The Cu–Cr–Zr alloy generally had the best toughness of the alloys studied. At 250°C the toughness was only slightly lower than at room temperature. This material also showed some anisotropy, with the L–T orientation being tougher than the T–L orientation. However, the toughness was high in both orientations, and at 250°C this alloy showed much greater toughness than any of the other materials. Fractographic examination of the specimens tested at room temperature showed microvoid coalescence with a pronounced stretch zone at the precrack tip. At a test temperature of 250°C the fracture mode was again microvoid coalescence, but the dimples appeared to be shallower and less well-formed, and the stretch zone was much less apparent.

The beneficial effects of Zr additions on the high-temperature ductility and toughness of Cu-based alloys have been observed previously [8–12]. However, the mechanism by which the Zr improves the high-temperature ductility is not clear. Kanno [9] suggests that the Zr additions result in the formation of Zr sulphide particles, which getter the S and thus prevent S segregation to the grain boundaries. It was proposed that the presence of S produced intergranular fracture and low ductility at high temperatures in Cu alloys that did not contain Zr. Misra et al. [10] examined a Cu–Co–Be alloy and suggested that the Zr segregates to the grain boundaries and prevents the dynamic embrittlement of these boundaries by oxygen which occurs in the absence of Zr [1]. They suggest either a reduction in the diffusivity of O due to the presence of Zr, or a scavenging effect of the Zr which will tie up the O. This suggests that if O is responsible for the reduced toughness observed in the copper alloys, the toughness should still be high when fracture occurs in a vacuum, if the partial pressure of O can thus be reduced to sufficiently low levels. Tests of a Cu–Co–Be–Ni alloy at 200°C supported this hypothesis, as no embrittlement was observed for tests in vacuum, but significant embrittlement was observed in air [11]. However, similar studies of a Cu–Cr alloy [12] attributed the high-temperature embrittlement to S, and the beneficial effect of Zr to the scavenging of the S. Thus, the embrittling mechanisms may differ in different alloy systems.

If the toughness is still low in vacuum, as was the case for the AL-15 material, then O may not be responsible, and surface-active impurities such as S present in the alloy may be causing embrittlement, if in fact the mechanism for the loss of toughness in the dispersion-strengthened alloys and the Cu–Ni–Be alloy is the same. Although the toughness of the AL-15 and AL-25 at 250°C in vacuum is higher than in air, the toughness is still much lower than would be expected from the tensile properties, so it is not clear which mechanism is responsible for embrittlement in the dispersion-strengthened materials.

Other elements have also been observed to prevent high-temperature embrittlement in Cu and Cu alloys, including Ti, B [13], Y [14], Ce, Ca, or La [15]. These effects were attributed to the removal of S from the alloys by the formation of sulphide particles, thus preventing grain boundary embrittlement by S at high temperatures [13–15].

If O is responsible for a dynamic embrittlement [10,11], then tests conducted at high strain rates should not show embrittlement, since sufficient time for diffusion of the O will not be available. To test this hypothesis, Charpy impact specimens were fabricated from the dispersion strengthened AL-25 alloy, in either the T–L or L–T orientations. Some of the specimens were fatigue precracked at room temperature prior to testing. The specimens were then tested with an instrumented Charpy impact test system at temperatures from 22°C to 300°C. No decrease in absorbed energy with increasing test temperature was observed for the AL-25 material, in either the blunt-notched or fatigue-precracked condition. The precracked specimens absorbed much lower levels of energy, due in part to the sharper fatigue precrack, and in part to the reduced ligament remaining after the cracks were grown from the machined notch. However, the sharpness of the notch did not affect the trend of the results as a function of temperature, indicating that notch acuity is not a factor. The specimens in the L–T orientation showed higher energy absorption than the specimens in the T–L orientation, in agreement with the fracture toughness data, another indication of

[1] Note that 'dynamic' is used in the sense that the presence of a stress is needed for embrittlement to occur, and not in reference to a strain rate during testing. This is compared to a 'static' embrittlement, which would occur merely due to exposure to the embrittling species, even in the absence of an applied stress.

the anisotropy in these materials. These results are similar to data previously reported for AL-25 IG0 material by SCM Metal Products [6].

The lack of embrittlement under dynamic testing conditions suggests that S is not responsible for the embrittlement observed in the dispersion-strengthened alloys, unless the S embrittlement mechanism is in some way time dependent. Again, it is not certain that all of these alloys are embrittled by the same mechanism. Further work is needed to determine the physical mechanism responsible for the dramatically lower fracture toughness observed for the GLIDCOP and Cu–Ni–Be alloys at 250°C.

It should be noted that most of the *J–R* data generated with this small disk compact specimen do not satisfy all of the validity requirements of the ASTM standards, and so these data are not valid. However, although the data are invalid according to ASTM E 1152, they are not incorrect. The size limitations imposed are conservative, and the *J*-integral values are quite likely still true measures of the materials' toughness, as long as the limits are not exceeded by too great a margin. The *J–R* curves are directly applicable to structures of the same thickness as the specimens, and are of great value in elucidating the materials' responses to test temperature. This will be useful information for evaluating candidate structural materials for ITER applications.

4. Summary

Fracture toughness tests have been conducted on several copper-based alloys being considered for applications in ITER, using small disk compact specimens, and the following preliminary results have been obtained.

1. Dispersion-strengthened copper alloys AL-15 and AL-25 show anisotropic fracture toughness, with higher toughness in the L–T orientation, and lower toughness in the T–L orientation. The anisotropy is significantly less in the AL-25 (IG0) alloy, presumably due to the cross-rolling during the alloy fabrication.
2. The fracture toughness of oxide-dispersion strengthened AL-15 and AL-25 decreases significantly as the test temperature increases from 22°C to 250°C. The toughness at 250°C is higher for AL-15 specimens tested in vacuum than when tested in air, suggesting that environmental effects (oxygen chemisorption) may be at least partially responsible for the low toughness in air at 250°C. However, the reduction in toughness from room temperature to 250°C is much greater than one would expect, based on the modest changes in the tensile properties over this temperature range.
3. Impact tests of full-size Charpy impact specimens of AL-25 do not show embrittlement as the test temperature increases from 22°C to 300°C. This is true for both blunt-notched and fatigue-precracked specimens.
4. The fracture toughness of a high-strength version of the Cu–Ni–Be alloy C17510 is anisotropic, with the L–T orientation being tougher than the T–L orientation. This material also shows a significant decrease in toughness as the test temperature in air increases from 22°C to 250°C.
5. The fracture toughness of a Cu–Cr–Zr alloy is typically greater than that of the other alloys studied. In addition, this material shows only a small reduction in toughness as the test temperature increases from 22°C to 250°C. Some anisotropy is present, with the L–T orientation being tougher than the T–L orientation.
6. The mechanism for the reduction in toughness of the dispersion-strengthened and the Cu–Ni–Be alloys is not clear. Different mechanisms may be operating in the different materials. The mechanism for the beneficial effect of Zr in the Cu–Cr–Zr alloy is also not certain, although it may be related to the formation of S-containing particles that thus prevent a sulphur-induced embrittlement from occurring at the grain boundaries.

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