

INVESTIGATION OF THE INFLUENCE OF GRAIN BOUNDARY CHEMISTRY, TEST TEMPERATURE, AND STRAIN RATE ON THE FRACTURE BEHAVIOR OF ITER COPPER ALLOYS, K. Leedy and J.F. Stubbins (Univ. of Illinois), D.J. Edwards (Pacific Northwest National Laboratory), R.R. Solomon (OMG Americas) and D. Krus (Brush Wellman)

OBJECTIVE

The objective of this work is determine how the changes in grain boundary chemistry, temperature, and strain rate affect the tensile and fracture behavior of GlidCop™ Al25, Hycon 3HP™ CuNiBe, and Elbrodur CuCrZr.

SUMMARY

In an effort to understand the mechanical behavior at elevated temperatures (>200°C) of the various copper alloys being considered for use in the ITER first wall, divertor, and limiter, a collaborative study has been initiated by the University of Illinois and PNNL with two industrial producers of copper alloys, Brush Wellman and OMG Americas. Details of the experimental matrix and test plans have been finalized and the appropriate specimens have already been fabricated and delivered to the University of Illinois and PNNL for testing and analysis. The experimental matrix and testing details are described in this report.

PROGRESS AND STATUS

Introduction

Although the microstructure and processing are significantly different for the following three alloys, oxide dispersion strengthened (DS) GlidCop™ Al25, and Hycon 3HP™ CuNiBe and Elbrodur CuCrZr (two precipitation strengthened alloys), the general trend for each alloy is that the tensile properties and fracture toughness decrease with increasing test temperature, and in the case of the CuAl25 and Hycon, the fracture toughness drops very rapidly at $T_{\text{test}} \geq 200^\circ\text{C}$ [1-8]. Along with the decrease in strength and toughness is a concomitant loss of ductility characterized by a large decrease in uniform and total elongation. The decrease in toughness is much more rapid than for the tensile properties, and it has been speculated that the poor toughness and possibly the strength are related to either an environmental and/or an impurity effect that alters the flow and fracture properties of these materials. The GlidCop™ alloys are now known to possess a strain rate dependence as the temperature increases, which may also affect their fracture toughness [6].

Literature surveys [9-11] suggest that the zirconium additions may act as a sulfur or oxygen scavenger, thereby reducing the effect of these elements on the fracture and flow properties. While tests at ORNL have indeed shown that CuCrZr does not exhibit as rapid a decrease in toughness as the other two alloys [4,5], the reason for this behavior still remains unclear. There is no evidence to support the idea that an environmental effect is solely responsible for the poor fracture toughness of the CuAl25 since tests conducted in vacuum at ORNL showed a marked decrease in fracture toughness with increasing temperature, though not quite as severe as that measured when tested in air.

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One large uncertainty that needs to be resolved is whether more than one mechanism may be responsible for the observed behavior in these three alloys, or if a different mechanism is operating in each alloy. Factors that complicate the analyses of these alloys and their behavior are the differences in grain size, strengthening phase, and composition. For example, the grains range in size and shape from $1\mu\text{m} \times 20\mu\text{m}$ (elongated shape) in the DS copper to over $30\mu\text{m}$ (equiaxed shape) in the Hycon and CuCrZr. In addition, the precipitate density in the CuNiBe alloys is typically 100-1000 times higher than that present in the oxide dispersion strengthened GlidCop™ alloy, and is accompanied by large coherency strains around the γ'' -phase in the CuNiBe alloys.

These microstructural variables and the testing parameters are known to influence the behavior of the alloys, however their respective roles are not clearly understood. Consequently, a study has been undertaken to begin looking at the influence of test temperature and strain rate on the mechanical properties and how the grain boundary chemistry and microstructure are related. This work is a collaboration between four parties: University of Illinois-UC, Pacific Northwest National Laboratory, OMG Americas, and Brush Wellman, Inc.

Experimental Procedure

Three different copper alloys are to be tested and analyzed. The GlidCop™ Al25 (Heat #C-8064) is in the cross-rolled and annealed condition and boron deoxidized, which is currently considered as the ITER Grade 0 (IG0) condition. The material was purchased from OMG Americas as 1 inch thick plates with a pure copper cladding. The Hycon 3HP CuNiBe (Heat #46546) was supplied by Brush Wellman, Inc. as four 1.25 inch thick plates. All four of the plates were originally in the HT temper (cold worked and aged), however, two of the plates were heat treated again to produce an AT tempered condition (solutionized, quenched, and aged). For this experiment only specimens from the AT temper are to be used. A small piece of Elbrodur G CuCrZr (Heat # AN4946) in the cold worked and aged condition (F37 temper) was used as the third alloy. Specimens from the CuCrZr are to be heat treated according to ITER draft specifications (solutionized 1 hour at 980°C , quenched, aged at 475°C for 2 hours, then furnace cooled). The compositions for the three materials are listed in Table 1.

Fracture toughness tests (J-integral) over a range of three temperatures (RT, 200°C and 300°C) will be conducted to determine the effect of test temperature. Test temperature will be limited to 300°C since the rapid decrease in strength already occurs at lower temperatures. Three-point bend bars will be used for the fracture toughness tests, as well as notched tensile specimens to allow more flexibility in the test matrix. The geometry of the specimens is provided in Figures 1 and 2. Representative fracture surfaces will be thoroughly analyzed to establish the nature of the microstructure, surface and near surface chemistry of the two alloys following fracture using optical metallography/SEM and Auger analysis.

Annealing experiments will also be conducted and subsequent Auger/TEM analysis used to determine if there are changes in grain boundary chemistry resulting from the high temperature exposure. Auger analysis of the specimens may require hydrogen charging and subsequent fracture inside the Auger microscope to eliminate oxidation on the fracture surface.

To better understand the material flow behavior near yield, interrupted tensile tests will be analyzed for microstructural development just past the point of yield, again at a number of representative temperatures (i.e. RT, 200°C and 300°C) at a minimum of two deformation

rates. This will indicate the extent to which flow is initiated and distributed during the initial stages of post-yield deformation. The tensile geometry to be used is the same as that used in previous and ongoing experiments in the Russian Federation as part of a collaboration with the United States, the European Community, and Japan. The details of the tensile specimen geometry are provided in Figure 3.

These three complementary studies should clarify the major contributors to the limited elevated temperature flow and fracture behavior of the two alloys. They will provide a means to differentiate between flow limitations during initial plastic deformation which leads to early localized flow or to localized flow stemming from the influence of grain boundaries or other microstructural features.

FUTURE WORK

In a separate but related study PNNL will perform Auger analysis on samples of the same three materials that have been held under load at the 350°C to see if any changes in grain boundary chemistry occur. Hydrogen charging may be necessary to initiate failure within the Auger microscope, however, the exact details remain to be worked out.

Depending on the outcome of the above studies, possible minor alterations in alloy chemistry or microstructure could lead to improvements in flow and fracture behavior. Such alloy alterations would be suggested by the current study (e.g. adding deoxidants - Zr - to alter grain boundary oxide levels, or inducing a precipitate sizes which would enhance cross-slip and flow distribution). These possibilities could be examined as a next logical step based on the outcome of the current work.

ACKNOWLEDGEMENTS

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REFERENCES

1. R.R. Solomon, J.D. Troxell, A.V. Nadkarni, *J. Nucl. Mater.*, **233-237** (1996) p. 542.
2. S.J. Zinkle and W.S. Eatherly, Fusion Materials Semiannual Progress Report for the Period ending June 30, 1996, DOE/ER-0313/20 (ORNL, 1996), p. 207.
3. S.A. Fabritsiev, S.J. Zinkle, and B.N. Singh, *J. Nucl. Mater.*, **233-237**, (1996) p. 127.
4. D.J. Alexander, Fusion Materials Semiannual Progress Report for the Period ending June 30, 1996, DOE/ER-0313/20 (ORNL, 1996), p. 217.
5. D.J. Alexander, Fusion Materials Semiannual Progress Report for the Period ending Dec. 31, 1996, DOE/ER-0313/21 (ORNL, 1996), p. 175.
6. S.J. Zinkle and W.S. Eatherly, Fusion Materials Semiannual Progress Report for the Period ending Dec. 31, 1996, DOE/ER-0313/21 (ORNL, 1996), p. 165.
7. B.N. Singh, D.J. Edwards, M. Eldrup and P. Toft, Risø-R-937 (EN), (Jan. 1997), (also accepted for publication in the *J. Nucl. Mater.*)

8. B.N. Singh, D.J. Edwards, M. Eldrup and P. Toft, Risø-R-971 (EN), (Feb. 1997), (also submitted to the J., Nucl. Mater.)
9. R.D.K. Misra, C.J. McMahon, Jr. and A. Guha, Scripta Metall., **31** (1994), p. 1471.
10. R. Muthiah, A. Guha and C.J. McMahon, Jr., Materials Science Forum, **207-209** (1996), p. 585.
11. M. Kanno, Z. Metallkde, **79** (1988), p. 684.

Table 1 Compositions (wt% unless otherwise noted) of the three alloys being investigated in this study.

Material					
GlidCop™ Al25	0.25 Al	23 ppm Fe	6 ppm Pb	~250 ppm B	10 ppm S typical
Hycon 3HP™ CuNiBe**	0.35 Be	1.92 Ni	<0.01 Co	<0.01 Fe	<0.03 Cr
Elbrodur G CuCrZr***	0.65 Cr	0.10 Zr	-----	-----	-----

* Heat # C-8064, OMG Americas Inc.

** Heat # 46546, Brush Wellman Inc.

*** Heat # AN4946, KM-Kabelmetal

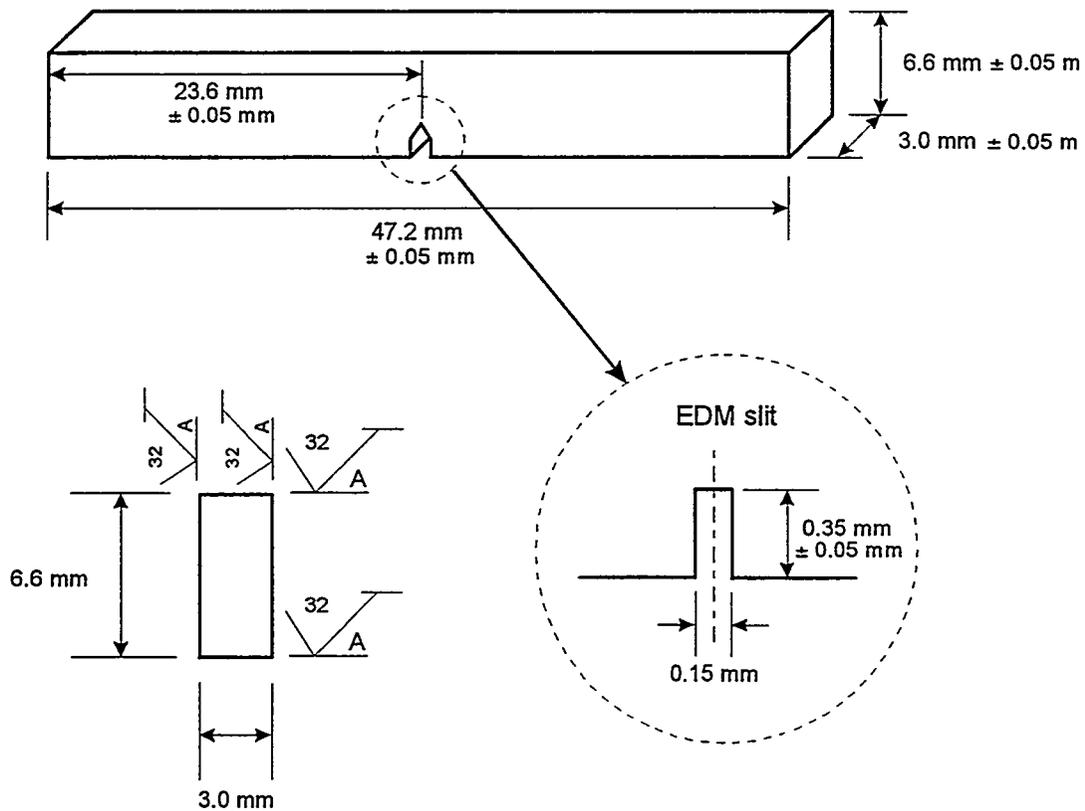


Fig. 1. Geometry and size of bend bars for fracture toughness testing.

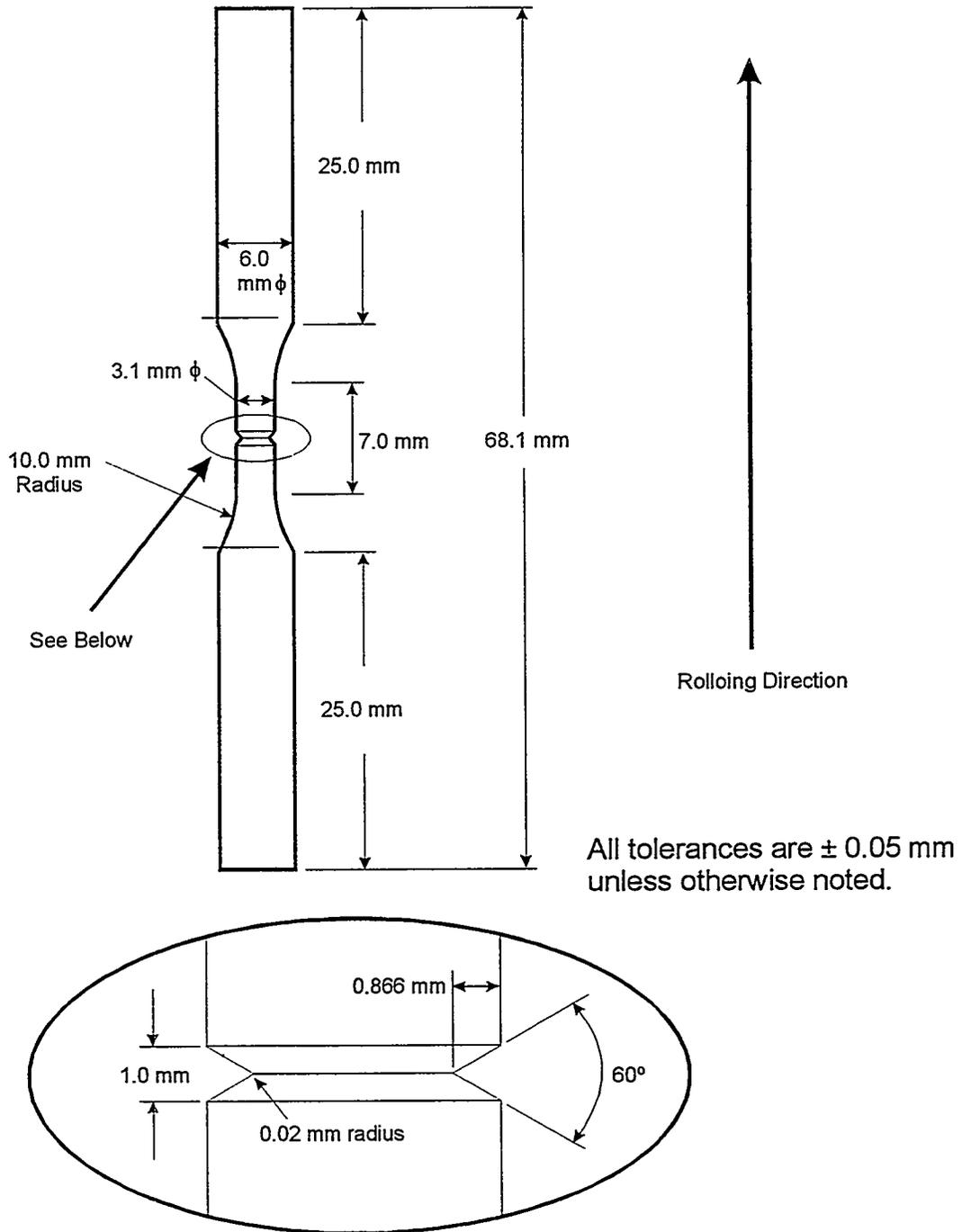


Fig. 2. Geometry and size of notched tensile specimens.

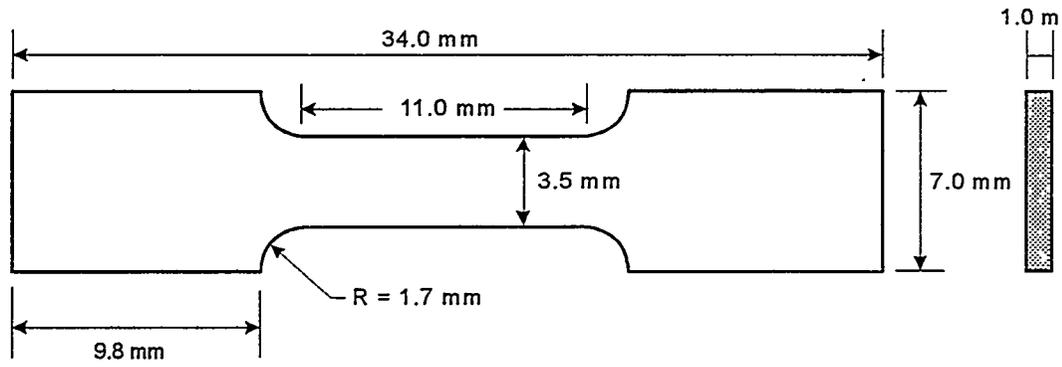


Fig. 3 Geometry and size for tensile specimens.