TEMPERATURE AND STRAIN RATE EFFECTS IN HIGH STRENGTH HIGH CONDUCTIVITY COPPER ALLOYS TESTED IN AIR – D.J. Edwards (Pacific Northwest National Laboratory)*

OBJECTIVE

The objective of this work is to investigate the strain rate and temperature dependence of the tensile properties of GlidCop™ Al25 and Hycon 3HP™ CuNiBe tested in air.

SUMMARY

The tensile properties of the three candidate alloys GlidCop™ Al25, CuCrZr, and CuNiBe are known to be sensitive to the testing conditions such as strain rate and test temperature. This study was conducted on GlidCop™ Al25 (2 conditions) and Hycon 3HP™ (3 conditions) to ascertain the effect of test temperature and strain rate when tested in open air. The results show that the yield strength and elongation of the GlidCop™ Al25 alloys exhibit a strain rate dependence that increases with temperature. Both the GlidCop™ and the Hycon 3HP™ exhibited an increase in strength as the strain rate increased, but the GlidCop™ alloys proved to be the most strain rate sensitive. The GlidCop™ failed in a ductile manner irrespective of the test conditions, however, their strength and uniform elongation decreased with increasing test temperature and the uniform elongation also decreased dramatically at the lower strain rates. The Hycon 3HP™ alloys proved to be extremely sensitive to test temperature, rapidly losing their strength and ductility when the temperature increased above 250°C. As the test temperature increased and the strain rate decreased the fracture mode shifted from a ductile transgranular failure to a ductile intergranular failure with very localized ductility. This latter observation is based on the presence of dimples on the grain facets, indicating that some ductile deformation occurred near the grain boundaries. The material failed without any reduction in area at 450°C and 3.9×10^{-4} s^{-1}, and in several cases failed prematurely.

PROGRESS AND STATUS

Introduction

The tensile properties of the three candidate copper alloys (GlidCop™ Al25, CuCrZr, and CuNiBe) being considered for use in fusion applications are known to be dependent on test conditions such as strain rate and temperature [1,2]. Solomon and coworkers [1] reported that the tensile properties and the fracture toughness of the GlidCop™ alloys were sensitive to test temperature, with the fracture toughness decreasing as much as 50% when the test temperature was raised from room temperature to 150°C. Their mechanical testing was done in open air for both the tensile and the fracture toughness testing, raising the issue of whether an environmental effect is the source of the drastic decrease in fracture toughness. Zinkle and Eatherly [2] recently demonstrated that the mechanical properties of GlidCop™ are weakly dependent on strain rate below test temperatures of 250°C, but at higher temperatures it can become quite significant. Their tests were conducted under high vacuum over a strain rate range of 4×10^{-4} to 6×10^{-2} s^{-1} at temperatures ranging from 25 to 500°C. Alexander and coworkers [3] conducted fracture toughness tests on the unirradiated

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GlidCop™ Al15 in both air and vacuum, and found that testing in air lowered the toughness approximately 50%. The decrease in toughness is therefore thought to be the result of a combination of strain rate sensitivity and possible chemisorption of oxygen to the fracture surface [2-4].

Zinkle and Eatherly also tested Hycon 3HP™ and CuCrZr alloys. Both materials proved to be less sensitive to strain rate than the GlidCop™, but the Hycon 3HP™ alloys were prone to brittle failure when the test temperature exceeded 300°C. The failure mode under these conditions changed from a ductile transgranular mode to a ductile intergranular mode. The behavior of these materials under different strain rates and test conditions suggests fundamental differences in the behavior of these materials that need to be understood.

To compare the effects of testing in air versus vacuum, a series of tensile tests were conducted in air at PNNL on the same materials as those tested by Solomon and coworkers and by Zinkle and Eatherly. The range of strain rates and temperatures were also extended to encompass a broader range of behavior. The tensile yield and ultimate strengths, elongations, and reduction in area are reported, and a brief comparison made with previous work.

Experimental Procedure

Tensile specimens were fabricated from plates of dispersion strengthened GlidCop™ Al25 and Hycon CuNiBe. The compositions of the two materials are given in Table 1. Two types of DS copper plates formed from the same internally oxidized powder were supplied by OMG Americas (formerly SCM Metals). The powder, designated by OMG Americas as LOX-80, was boron deoxidized to remove any free Cu_2O, and the powder was screened to below 80 mesh to narrow the size range of the starting powders. The first plate was in the form of extruded 2.5 cm thick plates that were given a cross-rolling and annealing (CR & Ann) treatment afterwards that corresponds to the designation ITER Grade 0 (IG0). The second GlidCop™ plate was consolidated directly from the powder by hot isostatic pressing, yielding a circular plate approximately 2.5 cm thick. The as-hipped plate was consolidated at ~14:1 reduction ratio at 980°C. No further thermomechanical treatments were given to the plate, simulating the condition of Al25 powder HIP'ped directly to stainless steel, a processing route considered for the ITER first wall. The grain structure of the cross-rolled and annealed material consists of highly elongated grains (1-2 μm in diameter x 10-20 μm in length) in the original extrusion direction, whereas the as-hipped material has a more equiaxed grain structure (20-30 μm diameter) with intergranular aluminum oxide at the grain boundaries.

The CR & Ann Al25 is the result of improved thermomechanical processing that, in conjunction with the LOX-80 starting powder, yields better elongation up to temperatures as high as 350°C [1]. The extruded plate is given an additional warm rolling treatment perpendicular to the original extrusion direction, yielding approximately 60% reduction. The plate is then annealed at 1000°C for 1 hour to stress relieve the plate, producing strength levels closer to that of the as-extruded plate. The final properties of the cross-rolled and annealed plate at room temperature are: σ_{UTS}= 421 MPa, σ_{YS}= 331 MPa, ε_{total} = 27%, and an electrical conductivity 86% IACS. The properties of the as-HIPped plate are almost identical at room temperature.

Four plates of Brush Wellman's Hycon 3HP™ CuNiBe were purchased, two of the plates in the HT condition (fully hardened temper, 69% IACS, hardness R_B= 92) and the second two

Table 1. Composition of materials (wt%)

Al25*	99.65 Cu	.0.25 Al	0.01 Fe	0.01 Pb	0.22 O	~250 ppm B
CuNiBe	99.5 Cu	0.35 Be	1.92 Ni	<0.2 Al	<0.01 Co	--------

* LOX -80 Al25 powder, ITER Grade 0 (IG0)

plates in the AT condition (solution annealed and aged, 65% IACS, R_B= 92). The HT temper involves a solution annealing and quenching step, subsequent cold working, and then ageing to achieve the fully hardened temper, whereas the AT temper does not include the cold working step. While the strengths of both materials are quite high, the lack of cold working in the AT temper yields a reasonably high strength condition with greater ductility. All of these plates were from the same heat (#46546) and originally in the HT temper. The AT plates were made by solution annealing two of the four HT plates and then ageing to achieve the AT temper. Dr. S. Zinkle provided a small piece of Hycon 3HP™ from the same heat (#46546) overaged to produce a higher conductivity condition to include in the testing matrix. The conductivity of this material was 74% IACS, with a σ_{YS}= 633 MPa. Further details on the latter material can be found in the report by Zinkle and Eatherly [2]. The grain structure in the Hycon 3HP™ AT temper is equiaxed with a high density of large beryllides dispersed throughout the matrix, with a grain size of approximately 50-60 μm. The grains in the HT temper are highly elongated as in the CR & Ann Al25, but much larger in size (25 μm dia. x 100 μm long).

The tensile tests were conducted in open air over a strain rate range of 3.9 x 10^{-4} to 1.5 s^{-1} and covering the temperature range of 25 to 450°C. The testing was conducted on an MTS servo hydraulic frame. The specimens were taken to temperature and then held for 5 minutes to allow the temperature to equilibrate through the entire specimen. Since the tests were conducted in open air, thermal convection was sufficient to help equilibrate the temperature in the short period of time. Oxidation of the specimens did occur during the testing, but from comparison with Zinkle and Eatherly's data [2] the oxide scale does not appear to have affected the tests. The specimen geometry and orientation with respect to the original rolling or extrusion direction is the same as that reported earlier for the STS specimens that were included in a joint RF/US/EU/Japan irradiation experiment in the SM2 reactor in Russia [5]. To aid in gripping and aligning the specimens pin holes were drilled in the center of the specimens grips at each end. Preliminary tests on the high strength Hycon 3HP™ alloys revealed that deformation occurred around the pin holes, leading to an underestimation of the yield strength due to a change in the slope of the tensile curve in the elastic region. This was corrected by increasing the pin hole diameter to 3.2 mm to lower the stress concentration at the pin holes. This problem never occurred in the lower strength GlidCop™ alloys (clamping from the grips was sufficient in itself), and no further problems were found in subsequent tests of the Hycon alloys after switching to the larger pin hole diameter. The 0.2% offset yield, ultimate, uniform and total elongation, and the reduction in area (RA) were measured on selected sets of tested specimens from computerized data. The reduction in area was measured in an optical comparator. Fracture surfaces from selected conditions were examined in an JEOL 840 SEM to analyze the failure mode as a function of strain rate and test temperature.

Results and Discussion

The mechanical properties for all of the tested conditions for the GlidCop™ alloy are listed in Tables 2 and 3. The reduction in area were measured only for the CR & Ann GlidCop™

Table 2. Temperature and strain rate dependence for cross-rolled and annealed GlidCop™ Al25 (IGO).

Strain Rate (s^{-1})	Test Temp. (°C)	YS (MPa)	UTS (MPa)	E_u (%)	e_{tot} (%)	RA (%)
3.9×10^{-4}	25	324	399	13	22.2	54.8
	150	289	333	9.8	29.1	53.6
	250	242	270	6.2	27.6	40.2
	350	184	201	1.8	33.9	36.2
1.5×10^{-3}	25	348	436	14.3	25.0	49.4
	150	304	345	9.0	25.7	55.0
	250	248	286	5.7	32.1	39.2
	350	210	226	2.6	39.5	30.7
	450	155	163	1.0	47.4	36.8
1.5×10^{-1}	25	360	443	14.4	26.5	51.4
	150	315	382	11.5	28.6	55.0
	250	296	333	7.7	28.3	56.3
	350	256	274	6.0	31.8	41.0
	450	218	244	4.9	36.9	54.7
1.5	25	359	454	14.0	25.1	50.5
	150	319	393	10.5	24.3	55.2
	250	304	349	9.9	26.6	60.4
	350	261	303	6.2	27.5	61.7
	450	242	272	5.3	26.5	62.2

Al25. The tensile properties of the dispersion strengthened alloy are generally better in the cross-rolled and annealed condition than in the as-hipped state. The strengths of the two materials are similar throughout the range of conditions tested, but with a noticeable difference in the uniform and total elongation behavior. The uniform elongation decreased in both materials the higher the test temperature, but was higher in the case of the as-hipped material. The total elongation, on the other hand, was higher in all conditions for the cross-rolled and annealed GlidCop™, and increased substantially the higher the test temperature and the lower the strain rate. In contrast, the as-hipped material exhibited lower total elongation as the temperature increased and the strain rate decreased. The reduction in area measured for the CR & Ann Al25 also improved as the strain rate increased but the influence of test temperature was varied. At strain rates of 1.5×10^{-3} s^{-1} and lower the RA tended to decrease as the test temperature increased, but at higher strain rates the RA began to increase with increasing test temperature. The difference in ductility between the CR & Ann and as-hipped Al25 illustrates the effect that the markedly different grain structures have on the behavior of the material. The equiaxed grain structure of the as-hipped material improves the uniform elongation at the higher temperatures and the highest strain rate, yet the as-hipped material fails much sooner.

Figure 1 shows a comparison of the effect of testing in air versus testing in vacuum. The yield strength of the CR & Ann material is plotted as a function of strain rate and the strain rate sensitivity parameter *m* calculated from the equation :

$$\sigma = C\dot{\varepsilon}^{m}$$

where C is a constant [6]. The data for the tests conducted under vacuum were taken from the work of Zinkle and Eatherly. Below 250°C the sensitivity to strain rate is fairly small, but above 250°C it begins to be more important. In both studies it was found that the strain rate sensitivity increased from 0.01 at room temperature to 0.07 at 300-450°C. Although Zinkle and Eatherly's data suggest that testing in vacuum caused the shift to occur at lower temperatures, the lack of any 300°C data at higher strain rates under vacuum does not allow any conclusions to drawn. Based on the limited results the strain rate sensitivity does not appear to be affected by the testing environment for the CR & Ann Al25. The strain rate sensitivity was also measured for the as-hipped Al25, and it was found that the yield strength in this condition was less sensitive to strain rate, giving a strain rate sensitivity from 0.01 at room temperature to 0.03 at 450°C. Keeping in mind that the as-hipped material failed sooner than the CR & Ann material, the data still indicates that the highly wrought grain structure of the CR & Ann condition strongly influences the behavior of the material. If the properties of the as-hipped material could be improved, it might make a more suitable processing route for the dispersion strengthened material.

The Hycon 3HP™ alloys proved to be more susceptible to the influence of test temperature. The data in Tables 4-6 show that both the uniform and total elongation as well as the

Table 3. Temperature and strain rate dependence for as-hipped GlidCop™ Al25.

Strain Rate (s^{-1})	Test Temp. (°C)	YS (MPa)	UTS (MPa)	e_u (%)	e_{tot} (%)
3.9×10^{-4}	25	281	395	18.8	22.3
	150	252	321	13.0	16.5
	250	221	270	6.4	8.5
	350	187	216	2.7	4.2
1.5×10^{-3}	25	298	417	17.5	27.5
	150	247	323	13.2	18.8
	250	226	277	8.2	9.0
	350	206	232	3.6	4.5
	450	169	183	2.6	3.9
1.5×10^{-1}	25	316	427	16.3	26.3
	150	275	364	13.7	23.8
	250	243	315	12.7	26.1
	350	200	277	12.0	17.6
	450	197	233	7.8	13.0
1.5	25	311	439	17.4	24.1
	150	283	379	14.9	25.9
	250	260	329	12.4	26.7
	350	249	292	11.6	20.7
	450	208	252	10.0	19.7

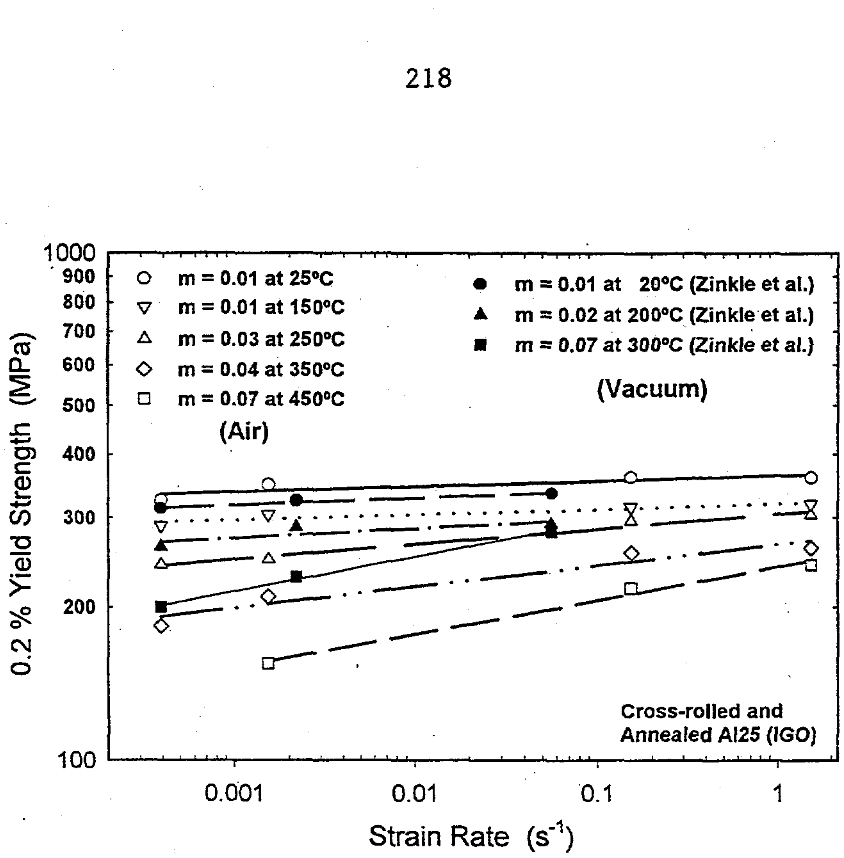

Figure 1. Strain rate dependence of CR & Ann Al25 tested in air and vacuum at different temperatures.

reduction in area (measured for the AT temper only) decrease substantially as the test temperature increases. The Hycon 3HP™ alloys are all susceptible to severe reductions in their ductility as the test temperature increases above 350°C, and the same behavior was observed by Zinkle and Eatherly, though testing under vacuum appears to shift the embrittlement to somewhat higher temperatures. The embrittlement or loss of ductility worsens as the strain rate decreases, suggesting that an environmental effect and/or diffusion controlled mechanism is playing a role. Figure 2 shows a plot of the data from this study and that of Zinkle and Eatherly's work. With the limited data available, the yield strength of the Hycon 3HP™ alloys do not appear to be strain rate sensitive. In all cases the strain rate sensitivity parameter (ignoring those conditions where the specimens failed prematurely) was around 0.01-0.02, irrespective of being tested in air or in vacuum. A change in failure mode was observed at the higher test temperatures and lower strain rates, changing from a ductile transgranular mode to a brittle intergranular with some ductile dimpling present on the grain facets. The reduction in area also decreased drastically, in agreement with the shift to an intergranular failure mode.

None of the three heat treatments offered any real advantage in terms of retention of ductility or strength. One surprising result is that the HT temper, which is nominally in the fully hardened condition, is actually weaker and less ductile than the AT temper. The elongation of the HT temper is closer to that of the higher conductivity heat treatment of heat # 46546. One would expect that the cold working step included in the HT temper would produce a higher strength and lower ductility compared to the AT temper. Since the higher conductivity heat treatment yielded a higher strength than the HT temper processing, it seems likely that the strength of this alloy is very sensitive to prior heat treatments and processing.

CONCLUSIONS

Testing in air versus vacuum does not appear to change the behavior of the strain rate sensitivity of the CR & Ann Al25. Both the CR & Annealed Al25 and the as-hipped Al25 exhibit a decrease in strength as the test temperature is raised, but the as-hipped material is less strain rate sensitive than the CR & Annealed condition. This appears to be related to the difference in the grain size and morphology of the two materials, but there may be other factors such as the presence of aluminum oxide at the grain boundaries in the as-hipped material.

The Hycon 3HP™ alloy suffers from severe loss of strength and ductility whether tested in air or vacuum. The limited data suggests that testing in air shifts the embrittlement to lower temperatures, and certainly testing at lower strain rates causes the material to behave worse. *Ignoring the specimens that failed prematurely at the high temperatures,* the data shows that the AT temper material is not strain rate sensitive, in agreement with the study of Zinkle and Eatherly.

Table 4. Temperature and strain rate dependence for Hycon 3HP™ CuNiBe AT condition (Heat #46546).

Strain Rate (s^{-1})	Test Temp. (°C)	YS (MPa)	UTS (MPa)	E_u (%)	e_{tot} (%)	RA (%)
3.9×10^{-4}	25	558	727	17.1	19.5	24.5
	150	511	679	13.5	14.7	17.8
	250	508	606	3.2	3.4	6.8
	350	--------	395	------	------	5.5
1.5×10^{-3}	25	578	741	15.9	19.5	25.2
	150	557	689	12.2	15.0	18.9
	250	529	628	3.9	4.8	7.1
	350	477	512	0.42	0.54	4.2
	450	--------	371	------	------	0
1.5×10^{-1}	25	588	744	16.7	20.1	28.1
	150	558	698	14.3	17.5	25.5
	250	538	660	10.2	11.4	14.8
	350	500	597	3.0	4.0	7.3
	450	498	541	0.9	1.03	4.3
1.5	25	597	747	16.1	21.0	27.0
	150	559	703	14.4	18.6	24.9
	250	535	671	11.9	14.2	18.1
	350	545	619	4.9	6.1	7.4
	450	530	547	0.48	0.51	4.3

Table 5. Temperature and strain rate dependence for
Hycon 3HP™ CuNiBe HT condition (Heat #46546).

Strain Rate (s^{-1})	Test Temp. (°C)	YS (MPa)	UTS (MPa)	E_u (%)	e_{tot} (%)
3.9×10^{-4}	25	562	675	10.0	15.3
	150	520	608	9.5	14.9
	250	500	562	5.7	6.4
	350	421	498	------	0.75
1.5×10^{-3}	25	563	679	10.9	15.5
	150	538	623	8.6	13.2
	250	513	584	6.1	8.4
	350	499	518	------	0.71
	450	-------	370	------	------
1.5×10^{-1}	25	593	694	10.6	16.8
	150	545	646	8.6	14.0
	250	537	610	7.8	12.2
	350	489	555	5.1	6.5
	450	486	505	0.74	0.78
1.5	25	602	694	10.4	16.0
	150	545	630	10.5	16.2
	250	539	612	8.5	12.5
	350	509	565	6.6	10.1
	450	480	532	------	2.1

Table 6. Temperature and strain rate dependence for
Hycon 3HP™ CuNiBe high conductivity condition (Heat #46546).

Strain Rate (s^{-1})	Test Temp. (°C)	YS (MPa)	UTS (MPa)	e_u (%)	e_{tot} (%)
1.5×10^{-3}	25	620	703	9.9	15.2
	150	575	657	7.9	12.7
	250	541	598	5.6	7.0
	350	[redacted]			
	450				
1.5×10^{-1}	25	611	704	11.1	17.0
	150	596	677	8.2	12.6
	250	550	623	7.6	13.0
	350	[redacted]			
	450				
1.5	25	627	710	10.8	16.8
	150	601	676	7.9	12.2
	250	565	629	8.3	13.0
	350	[redacted]			
	450				

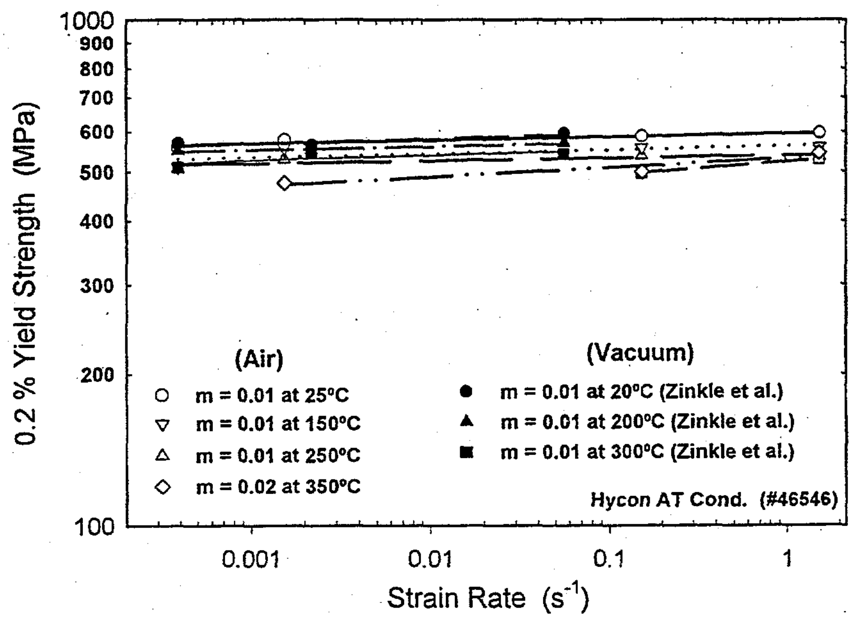

Figure 2. Strain rate dependence of Hycon 3HP™ (AT temper) tested in air and vacuum at different temperatures.

FUTURE WORK

Further measurements on the reduction in area are planned to be made. Specimens from CuCrZr are also planned for testing to compare the results with those obtained from the dispersion strengthened copper and Hycon 3HP™ alloys. Auger analysis will be performed on specimens of all three alloys to provide additional insight into the behavior of the alloys.

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