ELSEVIER

Journal of Nuclear Materials 283–287 (2000) 404–408

journal of nuclear materials

www.elsevier.nl/locate/jnucmat

Effect of neutron dose and irradiation temperature on the mechanical properties and structure of dispersion strengthened copper alloys

A.S. Pokrovsky [a], S.A. Fabritsiev [b], D.J. Edwards [c], S.J. Zinkle [d], A.F. Rowcliffe [d,*]

[a] *Scientific Research Institute of Atomic Reactors, 433510 Dimitrovgrad, Russian Federation*
[b] *D.V.Efremov Scientific Research Institute, 189631 St. Petersburg, Russian Federation*
[c] *Pacific Northwest Laboratory, P.O. Box 999, Richland, WA 99352, USA*
[d] *Oak Ridge National Laboratory, P.O. Box 2008, Bldg. 5500, Oak Ridge, TN 37831-6376, USA*

Abstract

This report is the first to present the data on the effect of neutron irradiation doses of 0.2 and 0.4 dpa at $T_{\mathrm{irr}} \sim 150°\mathrm{C}$ and $T_{\mathrm{irr}} \sim 300°\mathrm{C}$ on the mechanical properties of the GlidCopAl25IG alloy in different metallurgical conditions (CR + annealed, HIP, as-extruded). The dose dependence of radiation strengthening for GlidCopAl25IG alloy at $T_{\mathrm{test}} = T_{\mathrm{irr}} = 150°\mathrm{C}$ shows that strengthening increases with dose, up to $\Delta\sigma_y \sim 100$ MPa at 0.4 dpa. Simultaneously, the uniform elongation decreases with increasing dose. An important point is that all investigations on ODS copper alloys, regardless of the metallurgical state (CR + ann, HIP, as-extruded), revealed similar dose dependencies of strengthening and loss of ductility. Irradiation up to 0.4 dpa at 300°C results in a slight strengthening of about 30 MPa of the ODS copper alloys. In this case the tendency to embrittlement depends on the metallurgical state and orientation of the specimens. © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

Oxide dispersion strengthened (ODS) copper alloys are leading candidates for the ITER high heat flux components, such as the divertor and first wall [1,2]. These materials are advantageous in terms of high thermal conductivity and high stability of properties at high temperatures. The disadvantage is relatively low ductility at test temperatures in the range 150–400°C. In an attempt to produce an improved GlidCopAl25 ODS Cu alloy the OMG Americas Company developed a modified fabrication procedure (deoxygenation by boron) of GlidCopAl25 IGO, which produces improved uniformity and higher ductility in the longitudinal and transverse directions while retaining a sufficiently high level of the strength and thermal conductivity [3]. However, the first investigations of the irradiation performance of GlidCopAl25 IGO revealed that its properties after neutron irradiation strongly depend on the initial alloy treatment [4].

Below are presented the results of a systematic investigation of how the manufacturing technology and treatment of GlidCopAl25 IGO alloy influence its radiation resistance.

2. Experimental procedure

The impact of the full range of fabrication procedures currently being employed in the development and applications of GlidCopAl25 was investigated. Four different thermomechanical alloy conditions were studied. Specimens were fabricated from plate material prepared by hot isostatic pressing (HIPing) of powder; this material is designated as GlidCopAl25 (HIP). Specimens prepared from as-extruded 25 mm diameter rod are designated as GlidCopAl25 (EXTR). Another

---

[*] Corresponding author. Tel.: +1-423 574 5057; fax: +1-423 574 0641.

E-mail address: af8@ornl.gov (A.F. Rowcliffe).

PII: S0022-3115(00)00226-9

set of specimens was prepared in both the longitudinal and transverse directions of a 25 mm thick plate prepared by the modified procedure at OMG Americas and identified by the designation GlidCopAl25 IGO [3]. Finally, a fourth set of specimens was cut from GlidCopAl25/316SS joints made by HIPping solid plates in Europe (EU); these specimens are designated as GlidCopAl25 (JOINT). Full details of the manufacturing technologies for these materials may be found in references elsewhere [5,6].

STS-type (sheet tensile) specimens of 10 mm gauge length and 1-mm-thick were used for the investigation of tensile properties [5]. The specimens were irradiated in Channel N5 of the SM-2 reactor at 150°C and 300°C to fluences of $\sim 3.5 \times 10^{20}$ n/cm^2 ($E > 0.1$ MeV) and $\sim 7.5 \times 10^{20}$ n/cm^2 ($E > 0.1$ MeV); this corresponds to doses of ~0.2 and 0.4 dpa, respectively (determined by the NRT standard [2]).

Irradiation was performed in a loop device (0.2 dpa) in two irradiation facilities at 150°C and 300°C, respectively, and in the special ampoules with boiling water (0.4 dpa) in irradiation facilities at 150°C and 300°C. With these facilities it was possible to control specimen temperature during irradiation. According to thermocouple readings, the irradiation temperatures were 150–170°C for STS in the low-temperature irradiation, and 280–300°C for STS in the high-temperature irradiation. Irradiated and reference specimens were tested in tension in the temperature range of 20–350°C at a strain rate of 1.6×10^{-3} s^{-1}. SEM investigations of the fracture surfaces of tested specimens were also performed.

3. Results

3.1. Stress–strain behavior

Fig. 1 shows typical engineering stress–strain curves of GlidCopAl25 IGO alloy at $T_{\text{test}} = 150$°C and 300°C prior to and after irradiation up to 0.4 dpa ($T_{\text{test}} \sim T_{\text{irr}}$). It is apparent that irradiation at $T_{\text{irr}} = 150$°C involves hardening of the alloy, and a severe reduction of uniform elongation. Irradiation at 300°C up to 0.4 dpa involves slight (~50 MPa) strengthening. The uniform elongation of specimens falls within the range of unirradiated values; the total elongation is about 70% of the unirradiated value. Fig. 2 summarizes the test results of unirradiated and irradiated specimens of various GlidCopAl25 alloy modifications investigated in this study.

In the unirradiated condition the GlidCopAl25 IGO exhibits a high level of ductility at room temperature (RT) that decreases with increasing temperature up to 300°C. The GlidCopAl25 (EXTR) has the highest strength of all the variants, but both the uniform and total elongations are significantly lower than for the IGO material. The as-HIPped material, GlidCopAl25 (HIP), has the lowest strength and highest ductility of all the variants. Further HIPping of the IGO material during the manufacture of joints results in a reduction of both strength and ductility. The magnitude of this reduction decreases with increasing test temperature (Fig. 2).

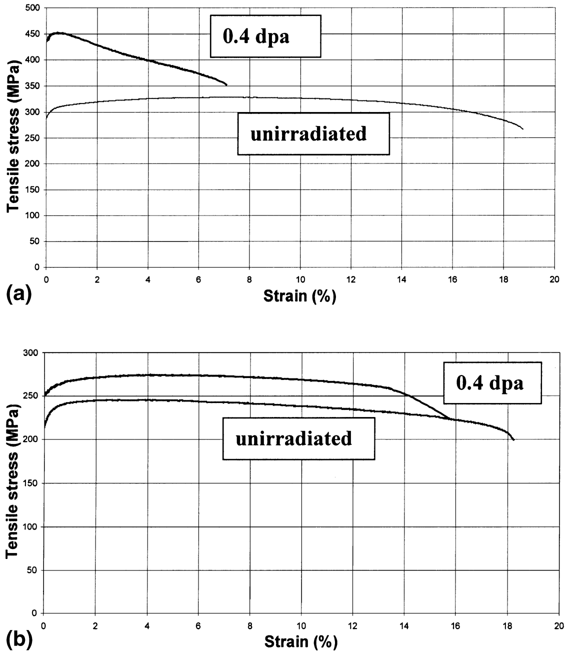

Fig. 1. Engineering stress–strain curves of GlidCopAl25 IGO alloy before and after irradiation up to 0.4 dpa at (a) 150°C and at (b) 300°C.

Irradiation at 150°C resulted in strengthening and reduced ductility for all of the materials investigated. The yield strength increase for specimens irradiated up to 0.2 dpa ranges from 40 to 120 MPa, and for 0.4 dpa specimens the increase ranges from 110 to 150 MPa, (Fig. 2). The strongest material, GlidCopAl25 (EXTR), exhibits the lowest increment of radiation strengthening. Although at a test temperature of about 150°C the different GlidCopAl25 grades in the unirradiated state have a spread in yield strength from $\sigma_Y = 250$–325 MPa, after irradiation at 150°C to 0.4 dpa all the grades have practically the same value of yield strength of 430 ± 10 MPa.

Strengthening of the specimens is accompanied by reduction of the uniform elongation (Fig. 2(b)). Total elongation is also reduced but not as greatly as the uniform elongation. It is interesting that all the irradiated specimens, regardless of the initial level of uniform elongation (from 13.2% to 5.7%), had the same uniform elongation of 2.4 ± 0.3% when tested at 150°C

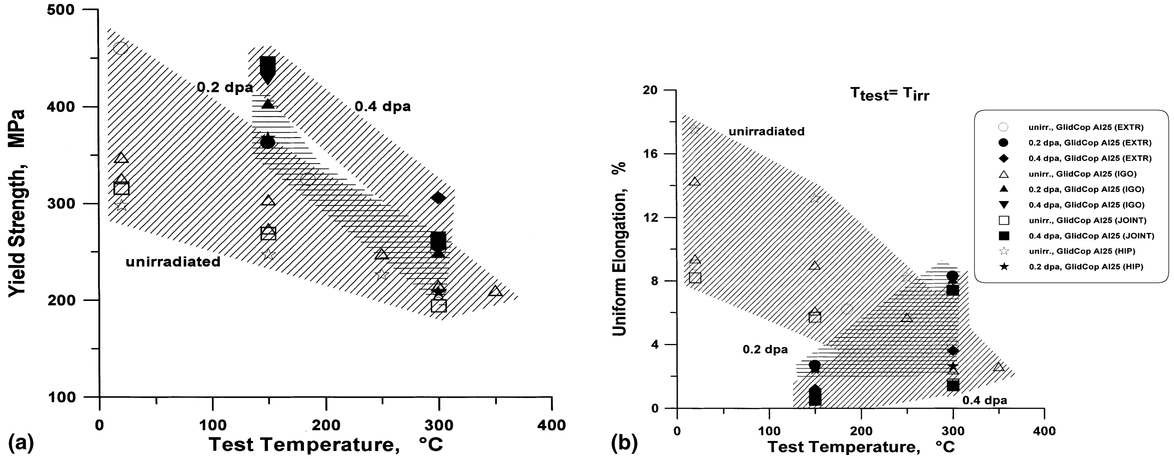

Fig. 2. (a) Yield strength and (b) uniform elongation versus testing temperature of the different GlidCopAl25 alloys grades (Extruded; Cross-rolled and annealed; HIP solid; HIP consolidated), when unirradiated and after irradiation to 0.2 and 0.4 dpa at $T_{\mathrm{irr}} = 150$°C and $T_{\mathrm{irr}} = 300$°C.

after irradiation up to 0.2 dpa at 150°C. At a dose of 0.4 dpa the uniform elongation was still lower but also the same for different fabrication methods, i.e., 0.9 ± 0.4%.

In contrast, the total elongation of irradiated specimens depends highly on the initial ductility and on the irradiation dose. Thus, for relatively low-ductility variants GlidCopAl25 (EXTR and JOINT), the total elongation was 1.6–3.2% after irradiation to 0.4 dpa (about 1/6 of the initial value), for high-ductility IGO specimens it was about 5.5% (about 1/3 of the initial value).

Irradiation at 300°C up to 0.2 dpa did not produce any significant hardening; the unirradiated and irradiated data fields for all variants overlap (Fig. 2). Further irradiation to 0.4 dpa produces a slight strengthening (~50 MPa). Correspondingly, the uniform elongation of all variants is significantly higher than at 150°C. The lowest uniform elongation (~1.5%) occurred for the GlidCopAl25 (JOINT), while the highest uniform elongation (~80%) occurred for the (EXTR) material. Total elongation values for all variants irradiated at 300°C were fairly high, about 50–80% of the initial value.

3.2. Fracture mode

Optical metallography and SEM fractography showed that unirradiated samples of the GlidCopAl25 IGO tested at 150°C and at 300°C were characterized by ductile transgranular fracture with no evidence of intergranular fracture. The amount of intergranular fracture in GlidCopAl25 (HIP) was a bit larger than in other grades. At the same time, investigation of the side surfaces showed that the IGO alloy demonstrated noticeable deformation due to grain-boundary sliding. This process was not observed in the (HIP) specimens, possibly because the grain size is larger and more equiaxed. At 300°C some evidence of intergranular fracture appeared in the unirradiated GlidCopAl25 (JOINT) specimens.

Optical metallography and SEM fractography of specimens tensile tested at 150° following irradiation at 150° to both 0.2 and 0.4 dpa showed that the GlidCopAl25 (IGO) fractured in a ductile transgranular mode. The GlidCopAl25 (HIP) material exhibited some intergranular cracking. The (JOINT) material failed in the copper with a mixed transgranular/intergranular mode. Following irradiation and testing at 300°C, the (IGO) material failed primarily in a ductile transgranular mode but with some evidence of intergranular failure; surface steps related to grain boundary sliding were also observed. On the other hand, the fracture surfaces of (HIP) material exhibited an exclusively intergranular character with no evidence of surface steps due to grain boundary sliding. In the (JOINT) material fracture occurred in copper alloy predominantly along the grain boundaries at 300°C.

4. Discussion

The investigations performed showed that as a whole, regardless of substantial differences in the GlidCopAl25 processing technology, the main radiation effects, such as strengthening and reduction in ductility, are very similar in each alloy condition.

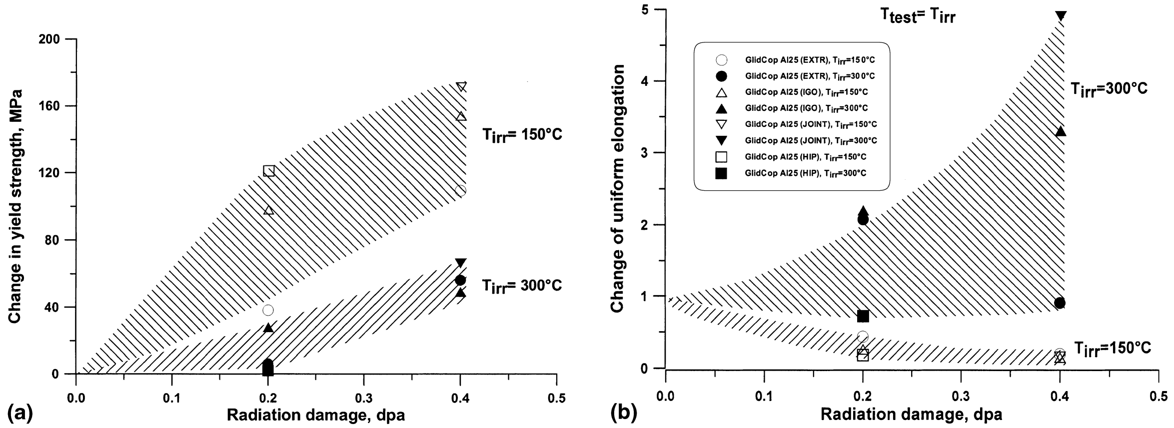

Fig. 3. (a) Radiation damage effect on the yield strength change $\Delta\sigma = \sigma_{\mathrm{Y\,irr}} - \sigma_{\mathrm{Y\,unirr}}$ of GlidCopAl25 (Extruded), GlidCopAl25 IGO (CR + ann), GlidCopAl25 IGO (HIP consolidated) and GlidCopAl25 IGO (CR + ann + HIP solid) copper alloys, irradiated in SM-2 reactor up to 0.2–0.4 dpa and tested at $T_{\mathrm{test}} = T_{\mathrm{irr}}$. (b) Radiation damage effect on the uniform elongation fractional change $S = \delta_{\mathrm{uniform\,irr}}/\delta_{\mathrm{uniform\,unirr}}$ GlidCopAl25 (Extruded), GlidCopAl25 IGO (CR + ann), GlidCopAl25 IGO (HIP consolidated) and GlidCopAl25 IGO (CR + ann + HIP solid) copper alloys, irradiated in the SM-2 reactor up to 0.2–0.4 dpa and tested at $T_{\mathrm{test}} = T_{\mathrm{irr}}$.

Irradiation to 0.4 dpa at 150°C results in strengthening (~150 MPa) and reduction of the uniform elongation of GlidCopAl25 (IGO), but the total elongation remains at a reasonably high level. After irradiation at 300°C strengthening of GlidCopAl25 (IGO) is low and a high level of total elongation is retained even at 0.4 dpa. Thus, when irradiated to 0.4 dpa at 300°C, the tensile properties of the GlidCopAl25 (IGO) are not severely degraded and an adequate level of ductility is retained.

The GlidCopAl25 (JOINT) material demonstrates an obvious tendency towards intergranular fracture with increasing temperature even in the unirradiated state. After irradiation at 300°C this tendency is enhanced, hence the alloy demonstrates practically zero uniform elongation and fractures at low levels of total elongation of about 2–4%. Obviously, the HIP treatment during joining affects the radiation performance of the GlidCopAl25.

When radiation strengthening, $\Delta\sigma_y$, and uniform elongation change $S = \delta_{\mathrm{unif\,irr}}/\delta_{\mathrm{unif\,unirr}}$ are plotted (Fig. 3), it becomes evident that all GlidCop modifications irradiated and tested at 150°C are strengthened by about 110–150 MPa and embrittled to $S \sim 0.2$ (dose 0.4 dpa). At an irradiation temperature of 300°C, strengthening is also realized for all grades but the magnitude is rather small, $\Delta\sigma_y \sim 30$–50 MPa (Fig. 3). When irradiated and tested at 300°C, the ductility depends on the material initial state. The GlidCop (JOINT) alloy demonstrates embrittlement due to an increasing tendency to intergranular fracture in the irradiated specimens. In contrast, the GlidCopAl25 (IGO) alloy has an increase in ductility after irradiation, and the tendency to intergranular fracture is suppressed.

5. Conclusion

The investigations performed indicate that the new GlidCopAl25 IGO modification exhibits good tensile properties in the unirradiated state, and a satisfactory resistance to radiation damage. Low-temperature in radiation embrittlement in this modification is less pronounced than in the earlier investigated ODS-type alloys. Total elongation of specimens irradiated up to 0.2–0.4 dpa at 150°C is maintained at a sufficiently high level of about 6–10%. The GlidCopAl25 (HIP) manufactured by means of the HIP consolidation of powder demonstrates a tendency to intergranular fracture at elevated test temperatures. This tendency is enhanced in the irradiated state and results in a substantial brittleness of irradiated material, when irradiated up to 0.4 dpa at $T_{\mathrm{irr}} = T_{\mathrm{test}} = 300$°C.

References

[1] S.J. Zinkle, S.A. Fabritsiev, Nucl. Fus. 5 (Suppl.) (1994) 163.

[2] S.A. Fabritsiev, S.J. Zinkle, B.N. Singh, J. Nucl. Mater. 233–237 (1996) 127.

[3] R.R. Solomon, J.D. Troxell, V.V. Nadkarni, J. Nucl. Mater. 233–237 (1996) 542.

[4] S.A. Fabritsiev, A.S. Pokrovsky, D.J. Edwards, S.J. Zinkle, A.F. Rowcliffe, R.R. Solomon, The effect of copper–steel joining technology, with the HIP method used, on the radiation resistance of ITER copper alloys, presented at Eighth International Conference on Fusion Reactor Materials, Sendai, Japan, 1997.

[5] D.J. Edwards, S.A. Fabritsiev, A.S. Pokrovsky, S.J. Zinkle, R.F. Mattas, R.D. Watson, Status of ITER Task

T213 colloborative screening experiment on Cu/SS joints in the Russian Federation SM-2 reactor, Fusion Materials, Semiannual Progress Report, DOE/ER-0313/19, December 1995, p. 165.

[6] S.A. Fabritsiev, A.S. Pokrovsky, D.J. Edwards, S.J. Zinkle, A.F. Rowcliffe, Effect of irradiation temperature on the mechanical properties and structure of Cu/SS joints irradiated at low doses, presented at Ninth International Conference on Fusion Reactor Materials, Colorado Springs, CO, Oct. 1999.