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Journal of Nuclear Materials 307–311 (2002) 431–438

journal of nuclear materials

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Effect of the bake-out regime on the recovery of properties of copper-based alloys and copper/steel joints

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Abstract

Copper alloys for ITER are characterized by rather strong radiation hardening and embrittlement at low irradiation temperatures of 80–200 °C. To reduce this effect it was proposed, with a certain dose attained, to use a special series annealing conditions at temperatures exceeding the operational temperatures. The report presents the results of an investigation into the effect of different annealing conditions, i.e. 300 °C – 10 h, 350 °C – 10 h and 400 °C – 10 h, on the recovery of properties (tensile and electrical conductivity) of copper-based alloys and copper/steel joints irradiated up to doses of 0.4 and 2 dpa at 150 °C. The results demonstrated that annealing helps to recover the mechanical properties of GlidCopAl25 IG alloy and joints. By comparison, the electric conductivity of the irradiated samples is only partially recovered. The conclusion is made that the bake-out regime allows for an effective recovery of ductile properties of copper alloys irradiated at low (150 °C) temperatures and, to somewhat lesser degree, the copper/steel joints.

1. Introduction

Copper alloys applied in the ITER divertor and first-wall are characterized by rather strong radiation hardening and embrittlement at low irradiation temperatures of 80–200 °C even at low damage doses (0.2 dpa). To reduce this effect it was proposed, with a certain dose attained, to use a special regimen of components annealing (divertor) at temperatures exceeding the operational irradiation temperatures. This annealing treatment in effect takes advantage of the periodic in-vessel bake-outs to improve the vacuum in plasma chamber.

The problem of choosing the optimal bake-out regime is highly complicated due to a large number of parameters that control the final result of annealing. The dose and irradiation temperature are of particular importance. At lower irradiation temperatures of about 80 °C small complexes of point defects (black dots) dominate the structure of irradiated copper alloys [1]. At higher irradiation temperatures of about 300 °C dislocation loops and cavities dominate the microstructure [2]. These objects obviously possess a different resistance to annealing compared to the defects produced at lower irradiation temperatures. The situation in DS and PH copper alloys is further complicated by the presence of small (about 2–8 nm) oxide particles or precipitates that act as sinks for the point defects, therefore actively participating in the defect structure evolution. Such a complicated situation apparently cannot be submitted to a direct theoretical simulation. It is clear that to substantiate the practicality of a bake-out annealing it is necessary to carry out a number of experiments at different annealing temperatures to assess their effect on microstructure created by neutron irradiation. Up to now only a very small number of studies on the bake-out annealing simulation have been performed [3], and data on the minimum possible temperature and maximum allowable dose at which the bake-out procedure is still efficient remain unknown.

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PII: S0022-3115(02)00942-X

The report presents the results of investigation into the effect of different annealing regimes, i.e. 300 °C – 10 h, 350 °C – 10 h and 400 °C – 10 h, on the recovery of properties (tensile and electrical conductivity) of copper-based alloys and copper/steel joints irradiated up to doses of 0.4 and 2 dpa at 150 and 300 °C.

2. Experimental

Specimens of GlidCopAl25 IG and Cu–Cr–Zr IG alloys and of GlidCopAl25/316LN and Cu–Cr–Zr/316LN joints (about 20) intended for the bake-out procedure were irradiated in the SPP-1 and SPP-2 facility at 160 °C up to the dose of about 0.4 dpa in the SM-2 reactor. The joints were made by hot isostatic pressing (HIP). The procedure of joints manufacturing and the preparation and geometry of specimens are described in detail in [4,5].

As a rule, two specimens were taken from a batch of four specimens located at the same level of the irradiation capsule, i.e. each accumulated the same dose, to assess the dose dependence. The two remaining specimens were given the bake-out procedure to allow a precise estimate of the effect of annealing on the changes in the properties.

Irradiated specimens were annealed in vacuum with the annealing temperature controlled to within ±10 °C. These specimens were annealed in vacuum at 350 °C for 10 h (1st annealing condition) and also at 400 °C, 10 h (2nd annealing condition). Heating time amounted to 0.5 h, and cooling time to about 0.6 h. Mechanical

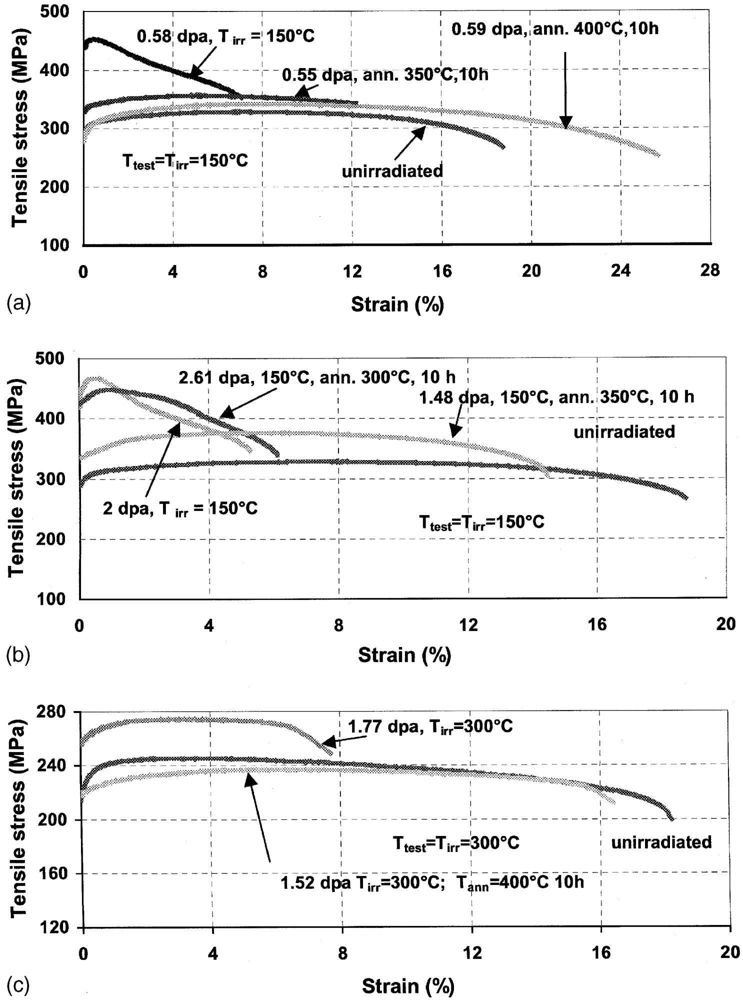

Fig. 1. Effect of bake-out annealing at 300–400 °C (10 h) on the engineering stress–strain curves of GlidCop Al25 (IG) (CR + ann) alloy (a) irradiated to 0.55–0.59 dpa at $T_{test} = T_{irr} = 150$ °C, (b) irradiated to 1.48–2.61 dpa at $T_{test} = T_{irr} = 150$ °C and (c) irradiated to 1.52–1.77 dpa at $T_{test} = T_{irr} = 300$ °C.

properties and electrical resistivity of the irradiated and annealed specimens were measured.

Irradiated and reference tensile specimens (gauge length 10 mm) were tested for tension at $T_{\text{test}} = T_{\text{irr}}$ at a deformation rate of 1.6×10^{-3} s^{-1}. SEM investigations of the fractured surface of tested specimens were also performed.

Additionally to the first experiment another 20 specimens were irradiated in the SPP-7 facility at 150 °C up the dose of about 2 dpa. These specimens were annealed at 350 °C, 10 h and at 300 °C, 10 h (3rd annealing condition). Additional samples irradiated at 300 °C to 2 dpa were also given an annealing treatment at 400 °C for 10 h in order to estimate the annealing influence on the properties of materials.

3. Results

3.1. Tensile properties

3.1.1. Base alloys

3.1.1.1. GlidCopAl25 IG. Fig. 1(a) shows the strain–stress curves for GlidCopAl25IG after irradiation to 0.6 dpa at 150 °C and annealing (350 °C, 10 h, and 400 °C, 10 h). It is obvious that at these doses both annealing conditions efficiently recover strength and plasticity of the alloy. The annealing at 400 °C, 10 h restores the mechanical properties of the irradiated material to the unirradiated level.

At higher irradiation doses of about 2 dpa (Fig. 1(b)) annealing at 350 °C for 10 h also restores a large degree of the ductility of the irradiated alloy, whereas annealing at 300 °C for 10 h only slightly (by about 5%) increases the irradiated alloy ductility. For the samples irradiated at 300 °C, annealing at 400 °C for 10 h considerably increases the ductility and decreases the strength of GlidCopAl25 IG (Fig. 1(c)).

3.1.1.2. Cu–Cr–Zr IG. Fig. 2(a) shows the stress–strain curve of the Cu–Cr–Zr JET (IG) (SA + aged) alloy after irradiation to 2 dpa and after annealing at 300 °C, 10 h and 350 °C, 10 h. Annealing at 300 °C, 10 h is ineffective at restoring the ductility of the alloy; even appearing to somewhat strengthening the material further beyond the irradiation-induced levels.

After annealing at 350 °C, 10 h, the specimens irradiated up to 1.4 dpa at 150 °C exhibit an increase in

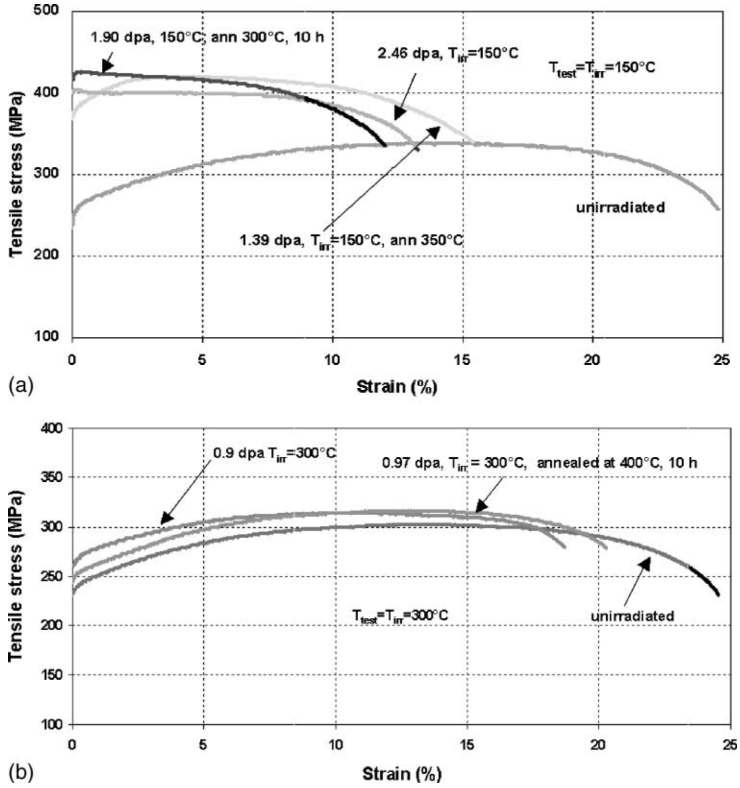

Fig. 2. Effect of bake-out annealing at 300–400 °C (10 h) on the engineering stress–strain curves of CuCrZr (IG) (SA + aged) alloy (a) irradiated to 1.39–2.46 dpa at $T_{\text{test}} = T_{\text{irr}} = 150$ °C and (b) irradiated to 0.9–0.97 dpa at $T_{\text{test}} = T_{\text{irr}} = 300$ °C.

ductility from 1.6% to 4%, with some decrease in the strength. The work hardening ability of the material is restored, but not to the same levels as the unirradiated material. The apparent yield drop exhibited by the as-irradiated material has disappeared after annealing at 350 °C for 10 h (Fig. 2(a)). Annealing at 300 °C for 10 h only slightly (by about 5%) increases the irradiated alloy plasticity. Comparison of the work hardening coefficients of irradiated and irradiated and annealed specimens shows that annealing at 350 °C during 10 h recovers the material ability for strain hardening. Despite the fact that the yield strength of annealed specimens remains essentially higher (by 100 MPa) than that of the unirradiated ones, the annealing suppresses (as seen from the stress–strain curves) the processes resulting in yield drop, the dislocation sources therein work and dislocations propagate rather freely.

Annealing at 400 °C, 10 h of samples irradiated at 300 °C does little to affect the Cu–Cr–Zr IG properties (Fig. 2(b)).

3.1.2. Joints

First tests of the GlidCopAl25/316SS (friction welding) and MAGT0.2/316 LN HIP joints irradiated to 0.2 dpa at 150 °C showed the effectiveness of annealing at 350 °C, 10 h [4]. At higher irradiation doses 0.5 dpa annealing at 350 °C for 10 h and 400 °C for 10 h increased the uniform and total elongation of the GlidCopAl25/316 LN EU joint (Figs. 3 and 4).

At high irradiation doses (1–2.5 dpa) annealing at 300 °C for 10 h caused little change in the uniform elongation, but does increase the total elongation of the GlidCopAl25/316 LN JA joint. The yield and ultimate strength of the joint decrease by 40 MPa. Annealing at 350 °C for 10 h decreases the strength by 80 MPa, has little effect on the uniform elongation and doubles the total elongation. The same behaviors were exhibited by the GlidCopAl25/316 RF HIP joints. The annealing therefore causes some improvement in the ductility of the as-irradiated joints.

The CuCrZr/316 RF HIP joint is more ductile after irradiation to 2 dpa at 150 °C compared to the GlidCopAl25 joints (Fig. 4). The level of uniform elongation is about 6%, and total elongation about 15%. Annealing has little effect on the yield and ultimate strength, but it does lower the uniform and total elongation after annealing at 350 °C for 10 h.

Annealing at 400 °C for 10 h of specimens of GlidCopAl25/316 JA and CuCrZr/316 RF irradiated to

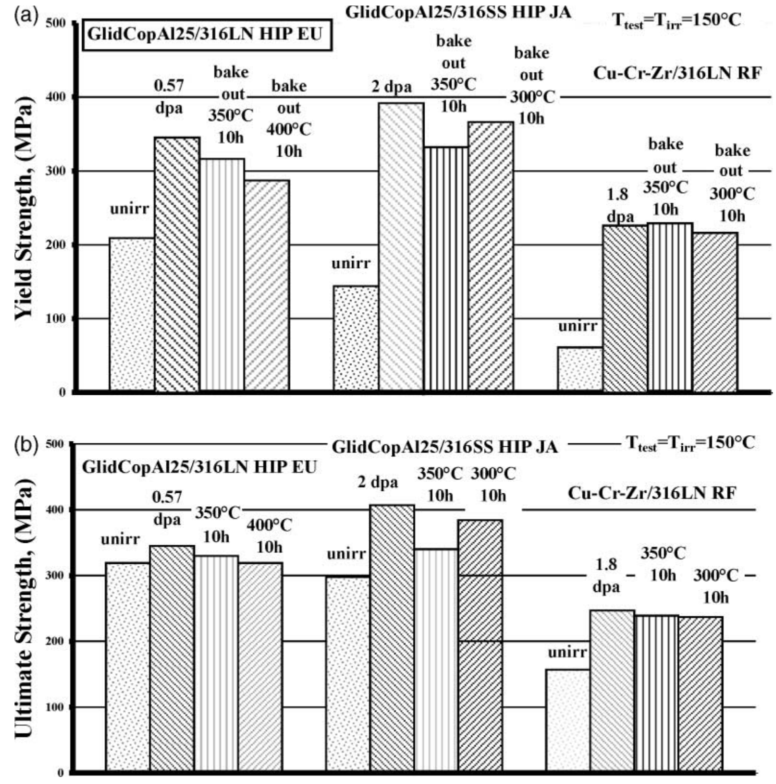

Fig. 3. Effect of bake-out annealing at 300–400 °C (10 h) on yield strength (a) and ultimate strength (b) of GlidCopAl25/316LN and CuCrZr/316LN joints irradiated to 0.5–2 dpa at $T_{test} = T_{irr} = 150$ °C.

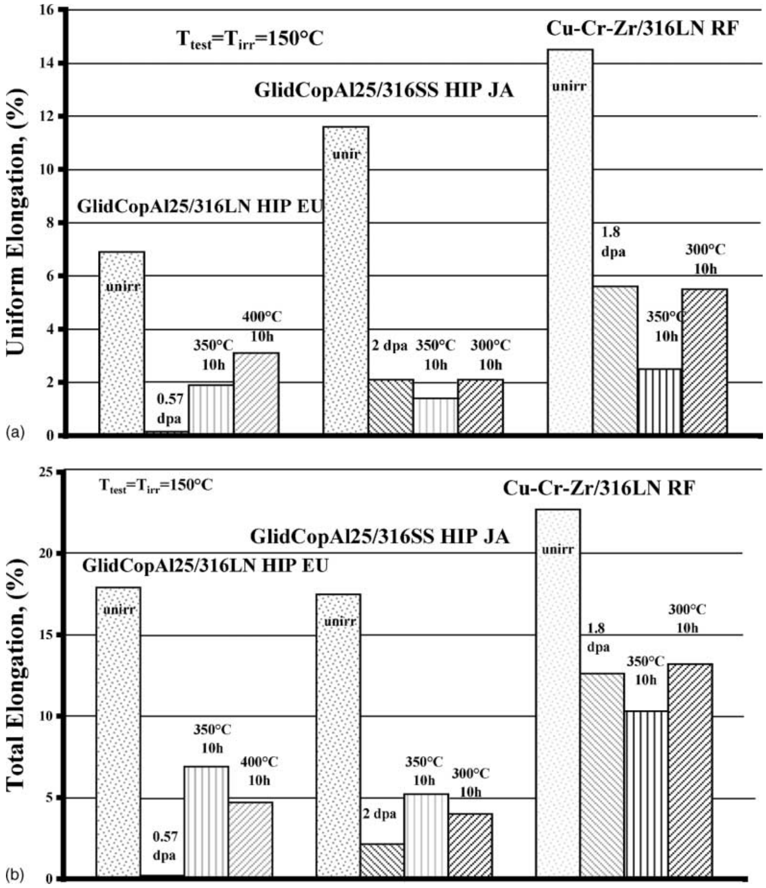

Fig. 4. Effect of bake-out annealing at 300–400 °C (10 h) on uniform elongation (a) and total elongation (b) of GlidCopAl25/316LN and CuCrZr/316LN joints irradiated to 0.5–2 dpa at $T_{test} = T_{irr} = 150$ °C.

2 dpa at 300 °C did not result in changes in joint properties. GlidCopAl25/316 remained highly brittle (uniform elongation is about 1%, total elongation about 1.3%), and the CuCrZr/316 joint plasticity even showed some increase (uniform elongation about 10%, total elongation about 20%). However, the yield strength of GlidCopAl25/316 is lower by 80 MPa than that of CuCrZr/316.

3.2. Fracture character

The SEM investigations of the fracture mode of irradiated specimens of GlidCopAl25IG and Cu–Cr–Zr JET (IG) alloys reveal that both alloys fractured mostly in the ductile transgranular mode. But in this case in GlidCopAl25IG alloy one can observe the areas on the fracture surface, where small dimples of about 0.5 mkm merge to form microcracks.

On the whole it is worth noting that the comparison showed no essential difference in the fracture character of irradiated and irradiated and annealed specimens. Evidently it is attributed to the fact that the level of local deformation preceding the fracture is rather close for materials in the as-irradiated and as-irradiated and annealed states.

3.3. Influence of bake-out regime on electrical resistivity

As follows from Fig. 5(a), at 0.2 dpa the annealing at 350 °C for 10 h leads to reduction in the ρ_{irr}, but does not lower the resistivity to the levels in the unirradiated samples. Furthermore, for the extruded GlidCopAl25 specimens irradiated to ~0.47 dpa the annealing at 350 °C leads to even smaller reduction of ρ_{irr}.

A comparison of the annealing efficiency at 350 and 400 °C leads to the conclusion that at 400 °C the annealing of radiation-induced defect complexes is more effective than at 350 °C (Fig. 5(a) and (b)). This is further illustrated for the GlidCopAl25 IGO irradiated to

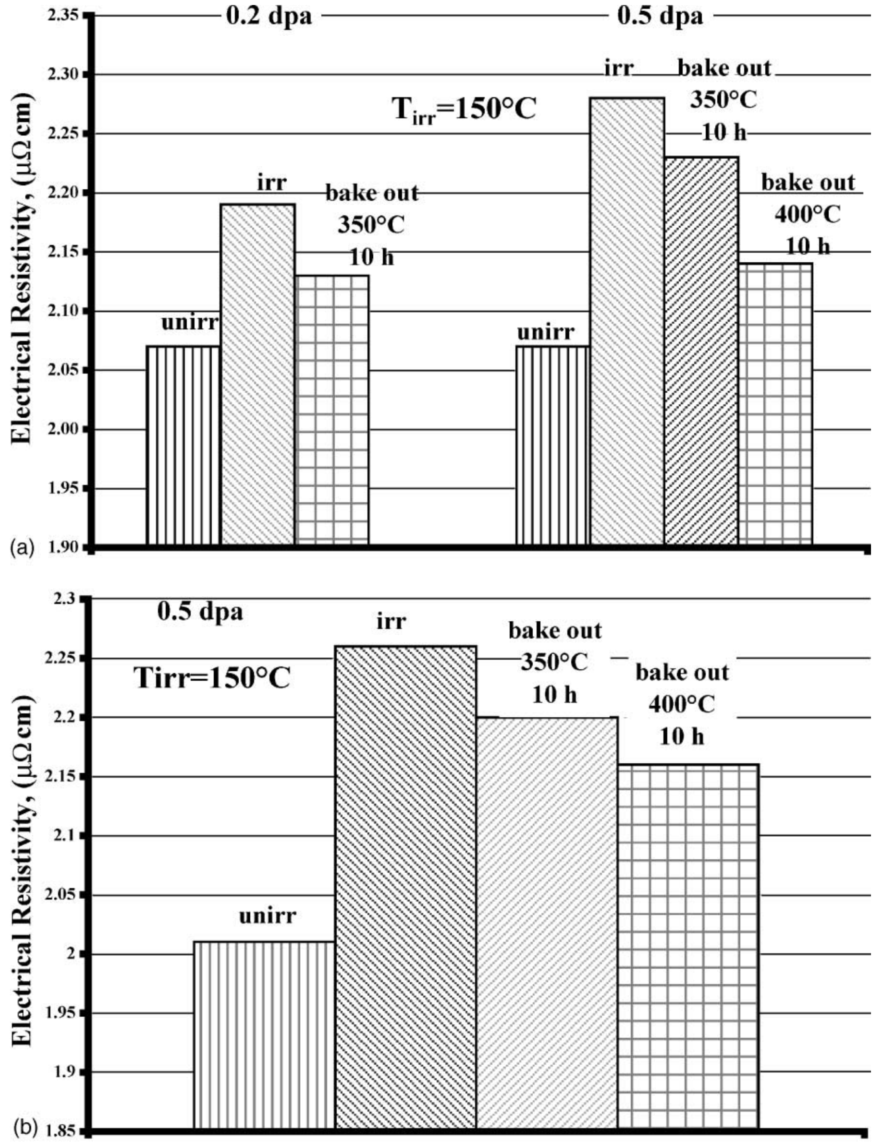

Fig. 5. Effect of bake-out annealing at 350–400 °C (10 h) on recovery of electrical resistivity of GlidCopAl25 extruded (a) and GlidCopAl25 IG (CR + ann) (b) alloys irradiated to 0.2–0.5 dpa at $T_{test} = T_{irr} = 150$ °C.

0.5 dpa at 150 °C, where annealing at 350 and 400 °C for 10 h reduces of ρ_{irr} by only 20% and 40%, respectively (Fig. 5(b)).

4. Discussion

As follows from Fig. 1 and Table 1, the annealing leads to the decrease of yield strength and ultimate strength of irradiated specimens of the GlidCopAl25 IG alloy. Uniform and total elongations of the specimens become 5 to 10 times greater.

The conclusion can be made that the bake-out regime of 350 °C, 10 h is an efficient means to recover ductility of the GlidCopAl25 IG alloy up to doses of about 2 dpa.

By comparison, the Cu–Cr–Zr IG alloy was found to be less sensitive to the bake-out procedure (Fig. 2) as compared with GlidCopAl25 IG and the recovery of its properties is significantly lower.

Note that the higher ductility of the unirradiated and as-irradiated Cu–Cr–Zr IG alloy are at the level close to that of the GlidCopAl25 IG alloy after the bake-out. It is possible to conclude that apparently the presence in

Table 1
Effect of bake-out regime on mechanical properties of GlidCopAl25IG and CuCrZrIG alloys

Materials	Dose (dpa)	T_{irrr} (°C)	T_{test} (°C)	Test results				Bake-out condition
				Uniform elongation δ_{unif} (%)	Total elongation δ_{total} (%)	Yield strength σ_y (MPa)	Ultimate strength σ_u (MPa)	
GlidCopAl25Ydir (CR + ann)	Unirr.		150	5.76 ± 0.38	17.82 ± 0.99	274.64 ± 4.43	324.91 ± 2.90	
GlidCopAl25Ydir (CR + ann)	Unirr.		300	2.22 ± 0.40	20.28 ± 2.85	206.65 ± 11.04	238.41 ± 9.51	
GlidCopAl25Ydir (CR + ann)	0.58	160	150	0.77 ± 0.22	6.37 ± 0.38	431.00 ± 4.00	441.33 ± 2.22	
GlidCopAl25Ydir (CR + ann)	0.55	160	150	5.30	12.20	336.00	357.00	Ann. 350 °C, 10 h
GlidCopAl25Ydir (CR + ann)	0.59	160	150	8.40	25.70	297.00	342.00	Ann. 400 °C, 10 h
GlidCopAl25Ydir (CR + ann)	1.51	150	150	0.90 ± 0.30	3.40 ± 1.90	422.00 ± 28.00	445.50 ± 21.50	
GlidCopAl25Ydir (CR + ann)	1.48	150	150	6.65 ± 2.25	18.35 ± 2.75	329.50 ± 25.50	375.50 ± 2.50	Ann. 350 °C, 10 h
GlidCopAl25Ydir (CR + ann)	2.71	150	150	0.70 ± 0.30	4.00 ± 1.70	463.50 ± 23.50	473.50 ± 22.50	
GlidCopAl25Ydir (CR + ann)	2.61	150	150	0.7	7.5	442	450	Ann. 300 °C, 10 h
GlidCopAl25Ydir (CR + ann)	1.52	285	300	3.10 ± 0.10	6.05 ± 1.65	259.00 ± 28.00	268.50 ± 30.50	
GlidCopAl25Ydir (CR + ann)	1.52	285	300	7.5	16.4	216	236	Ann. 400 °C, 10 h
CuCrZr JET (SA + aged)			150	13.60 ± 0.32	24.06 ± 0.34	262.33 ± 1.78	338.15 ± 2.64	
CuCrZr JET (SA + aged)			300	11.73 ± 0.33	25.80 ± 1.82	231.38 ± 0.01	302.14 ± 1.68	
CuCrZr JET (SA + aged)	2.46	150	150	0.38 ± 0.15	16.10 ± 2.80	386.50 ± 11.50	398.50 ± 0.50	
CuCrZr JET (SA + aged)	1.90	150	150	1.00 ± 0.50	13.05 ± 1.05	411.50 ± 7.50	420.50 ± 1.50	Ann. 300 °C, 10 h
CuCrZr JET (SA + aged)	1.39	150	150	3.2	11.3	389	418	Ann. 350 °C, 10 h
CuCrZr JET (SA + aged)	0.90	285	300	11.13 ± 0.31	17.50 ± 0.80	272.67 ± 2.89	326.00 ± 3.33	
CuCrZr JET (SA + aged)	0.97	285	300	13.25 ± 0.35	19.05 ± 1.25	256.00 ± 0.00	327.50 ± 1.50	Ann. 400 °C, 10 h

Cu–Cr–Zr of chromium available for diffusion and its binding to radiation defects causes the higher stability of radiation defect complexes in that material as compared with GlidCopAl25 IG.

For doses up to 0.5 dpa, annealing at 350 °C or 400 °C for 10 h increases the uniform elongation slightly and substantially improves the total elongation of the GlidCopAl25/316 LN EU joint. Annealing at 400 °C for 10 h of specimens of GlidCopAl25/316 JA and CuCrZr/316 RF irradiated to 2 dpa at 300 °C did not result in changes in the properties of these joints. Based on these results we conclude that at high irradiation doses annealing of the GlidCopAl25/316 joint allows a slight increase the total elongation of the joints. The bake-out annealing treatment offers little in the case of the Cu–Cr–Zr/316 joint since it has enough ductility after irradiation to 2 dpa at any irradiation temperature (150, 300 °C).

The irradiated specimens revealed that the beneficial effect of annealing decreases as the dose increase.

From the point of view of earlier concepts about the role of radiation defects and transmutants in increasing the electrical resistivity of copper alloys [6–9] it is clear that the bake-out treatment enables the partial annealing of $\Delta\rho_{\mathrm{irr}}$ which depends on the complexes of radiation defects.

It is clear that when increasing the dose this process is inhibited since the complexes become larger and more stable. Increase of the annealing temperature leads to the growth of vacancy flow to the radiation defect complexes, which are mainly complexes of interstitials and their solution.

In general, it is possible to conclude that the bake-out regime may reduce by 30% the increase of electrical resistivity of copper alloy specimens. In general in the ITER the increase of electrical resistivity and corresponding decrease of thermal conductivity at the dose of about 1 dpa is substantially lower, about 10%.

It is possible to conclude that the main benefit of bake-out annealing treatment resides in the recovery of plasticity properties of irradiated copper alloys while offering some recovery of the thermal conductivity of the alloys.

5. Conclusions

On the whole the investigations performed showed that the bake-out annealing treatment at 350 °C, 10 h made it possible to recover rather efficiently the ductility of both the alloys Cu–Cr–Zr IG and GlidCopAl25 IGO alloy irradiated to 2 dpa at 150 °C. Annealing at a lower temperature of 300 °C offered little improvement in the ductility irradiated at 150 °C specimens.

Annealing at 400 °C, 10 h, of samples irradiated at 300 °C considerably increases the ductility and decreases the strength of GlidCopAl25 IGO, but has little effect on the Cu–Cr–Zr IG properties.

Particularly important is the fact that the bake-out effectively recovers the ductility of joints of GlidCop-Al25/316LN and Cu–Cr–Zr/316LN type irradiated to 0.5 dpa at 150 °C.

On the whole it may be concluded that the bake-out procedure is effective for recovery of the ductility of ITER copper alloys and joints.

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