



# Irradiation examination of CuNiCrSi alloy for ITER applications

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## Abstract

Precipitation-hardened CuNiCrSi alloy was studied after the following treatments: (1) solution annealing, cold working and aging; (2) solution annealing and aging; (3) thermal simulation of diffusion bonding and annealing. In the first and second conditions, specimens were irradiated in the IVV-2M reactor to a dose of  $\sim 0.35$  dpa at 300°C. In the third condition, specimens were exposed to a dose of  $\sim 0.2$  dpa at 80°C, 200°C and 300°C. Results of irradiation tests performed at 300°C show that irradiation to a dose of  $\sim 0.35$  dpa cause loss of strength and embrittlement of the CuNiCrSi alloy in a high-strength state. The alloy in the third state becomes highly resistant to radiation embrittlement. Exposure at 200°C and 300°C to a dose of  $\sim 0.2$  dpa results in a substantial increase in the electrical conductivity of this material. © 1999 Elsevier Science B.V. All rights reserved.

## 1. Introduction

GlidCop A125 and CuCrZr copper alloys are considered presently as candidate materials for first wall and divertor of ITER [1]. These alloys have rather high yield strength ( $YS = 400\text{--}500$  MPa at 20°C) and thermal conductivity (80–90% from pure copper). However, for some components of ITER, copper alloys of improved strength are required. In this connection, particular attention has been given to CuNiBe [2] and CuNiCrSi [3] alloys.

Developed in Russia, the CuNiCrSi alloy is commonly used after thermomechanical treatment consisting of solution annealing, cold work and aging. It has high strength ( $YS = 600\text{--}700$  MPa at 20°C) and moderate thermal conductivity (50–60% from pure copper). Solution annealing and aging without cold work result in 10–15% lower strength than thermomechanical treatment. However, the ductility of this alloy in both conditions is low at 250–300°C [3].

In manufacturing ITER components by hot isostatic pressing, materials can be subjected to thermal cycles of the following type: heating, holding at 930–1050°C for 1–2 h and slow cooling. After similar heat treatment, the CuNiCrSi alloy has low strength and thermal conductivity. Additional heat treatment is required to improve the properties of this alloy. An optimum level of strength, ductility and thermal conductivity can be produced by annealing at 600°C.

Rather restricted data on radiation behaviour of CuNiCrSi alloy are now available. The objective of this paper is to study the effects of neutron irradiation on the properties of CuNiCrSi alloy treated under various conditions.

## 2. Experimental

The materials studied are CuNiCrSi alloy products (Table 1) produced by JS 'Institute Tsvetmetobrabotka' according to Standard TU 48-21-547-82. The materials were processed as follows (Table 1): solution annealing (SA), cold working (CW) and aging (AG); solution annealing (SA) and aging (AG); thermal simulation of diffusion bonding (DB) and annealing (A). The chemical composition of studied materials are given in Table 2.

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Table 1  
Characteristics of CuNiCrSi alloy products in this study

Thickness (mm)	Treatment	Legend
1	Solution annealing at 980–1000°C for 1 h, water cooling, 70% cold work, aging at 460°C for 4 h, air cooling	SA + CW + AG
10	Solution annealing at 950°C for 1 h, water cooling, 40% cold work, aging at 460°C for 4 h, air cooling	SA + CW + AG
30	Solution annealing at 950°C, water cooling, aging at 480°C for 4 h, air cooling	SA + AG
30	Holding at 1000°C for 1 h, furnace cooling, annealing at 600°C for 1 h, air cooling	DB + A

Flat (1 mm thick and 10 mm in gage length) and cylindrical (gage part of 3 mm in diameter and 18 mm in length) specimens were used for tensile tests. The specimens were cut perpendicular to the rolling direction.

Irradiation tests have been carried out in the IVV-2M reactor. Specimens in SA + CW + AG and SA + AG states were irradiated to a fluence of  $3.6 \times 10^{20}$  n/cm<sup>2</sup> ( $E \geq 0.1$  MeV) at 300°C in a helium environment (pressure of  $\sim 0.1$  MPa). The calculated damage level for the CuNiCrSi alloy was  $\sim 0.35$  dpa. The alloy in the DB + A state was irradiated to a fluence of  $1 \times 10^{20}$  n/cm<sup>2</sup>,  $E \geq 0.1$  MeV (calculated damage level of  $\sim 0.2$  dpa) at 80°C in water, at 200°C in helium and at 300°C in nitrogen–helium environment. The experimental uncertainty in irradiation temperature measurements was  $\pm 5^\circ\text{C}$ . Tensile tests were carried out in air at 20°C and in a vacuum ( $\sim 0.013$  Pa) at 100–550°C. The accuracy of temperature measurements was  $\pm 3^\circ\text{C}$ . The speed of moveable clamps was  $1 \pm 0.025$  mm/min (test after a dose of  $\sim 0.35$  dpa) and  $3 \pm 0.075$  mm/min (test after a dose of  $\sim 0.2$  dpa). Load measurements were accurate to within  $\pm 1\%$ . The fracture surfaces were investigated by scanning electron microscopy (SEM) with X-ray micro analyser. The electrical conductivity was measured at room temperature by standard four-point probe technique. The experimental uncertainty in the conductivity measurements was  $\pm 1\%$ . The electrical conductivity was determined before and after irradiation on tensile test specimens. In addition, the electrical conductivity of CuNiCrSi alloy was measured at 300°C after long term holdings.

### 3. Results

Tensile tests of specimens unirradiated (Fig. 1) showed that the highest values of strength and ductility (UTS = 717 MPa, YS = 703 MPa, elongation (E) = 11.5%, reduction in area (RA) = 33% at 20°C) are observed for the 1 mm thick sheets in SA + CW + AG state. The 30 mm thick plates in SA + AG state possessed the following properties at 20°C: UTS = 661 MPa, YS = 553 MPa, E = 16.5%, RA = 40.5%. The 10 mm thick sheets demonstrated the least strength and ductility in comparison with all products considered. It is probably related to the fabrication of this product from an ingot with a low silicon content (Table 2).

Fig. 1 shows that the irradiation to a dose of  $\sim 0.35$  dpa at 300°C causes a decrease of strength in the CuNiCrSi alloy (SA + CW + AG and SA + AG). Compared to an initial state, the strength after irradiation decreased by 5–9% for 1 mm thick sheet, and by 35–40% for 30 mm thick plate. The total elongation and reduction in area, decreased in all products after irradiation.

At 300°C, specimens before and after irradiation failed in a brittle manner, without necking and the fracture was predominantly intergranular. An uniform distribution of nickel and silicon over the fracture surface was observed. Local chromium-enriched areas about 4  $\mu\text{m}$  in size were also detected.

Tensile test results of the alloy in the DB + A state irradiated to a damage level of  $\sim 0.2$  dpa are given in Fig. 2. It is seen that irradiation increases the strength of the alloy at all test temperatures. The maximum strength change is observed at 80°C and the minimum at 300°C.

Table 2  
Chemical composition of CuNiCrSi alloy products in this study (wt.%)

Thickness of product (mm)	Alloying elements			Impurities					
	Cr	Ni	Si	Bi	As	Fe	Pb	Zn	Al
1	0.58	2.46	0.64	<0.004	<0.01	0.05	<0.01	<0.05	<0.04
10	0.49	2.03	0.29	<0.004	<0.01	0.07	<0.01	0.047	<0.04
30	0.44	2.09	0.48	<0.004	<0.01	0.05	<0.01	0.036	0.027

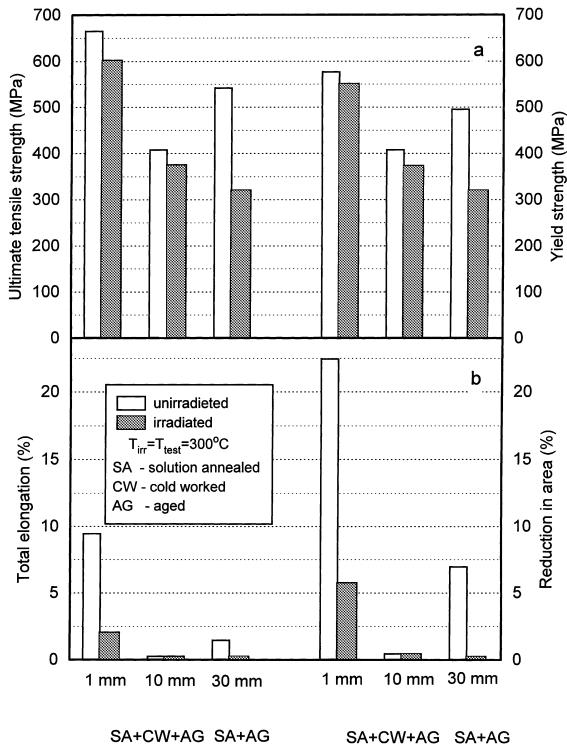


Fig. 1. Effect of irradiation to a dose of  $\sim 0.35$  dpa at  $300^\circ\text{C}$  on strength (a) and ductility (b) of CuNiCrSi alloy after different treatments.

In the range of ITER operating temperatures  $150\text{--}200^\circ\text{C}$ , the yield strength of the irradiated alloy is  $350\text{--}400$  MPa.

Fig. 2(b) shows that irradiation decreases the ductility of the alloy at  $80^\circ\text{C}$  and  $200^\circ\text{C}$ . However, at  $200^\circ\text{C}$  the ductility is rather high: UE = 3.6%, TE = 10% and RA = 55%. At  $300^\circ\text{C}$ , the ductility of the alloy is practically unaffected by irradiation.

The fracture of unirradiated specimens tested at  $400^\circ\text{C}$  is mainly transgranular (Fig. 3(a)) and mixed (transgranular and intergranular) after holding at a temperature of  $300^\circ\text{C}$  for 331 h (Fig. 3(b)). The fracture of both types has a ductile nature. Irradiation at  $300^\circ\text{C}$  does not change the mode of fracture (Fig. 3(c)).

X-ray micro analysis shows that the fracture surface has isolated areas enriched with chromium of  $\sim 4$   $\mu\text{m}$  in size. Numerous nickel-enriched areas with  $0.3\text{--}0.5$   $\mu\text{m}$  in diameter are shown at the fracture surface of irradiated specimens (Fig. 4).

The electrical conductivity of the alloy remains unchanged after irradiation at  $80^\circ\text{C}$ , but increases by  $\sim 10\%$  at  $200^\circ\text{C}$  and  $\sim 50\%$  at  $300^\circ\text{C}$  (Fig. 5). The effect of long-term holding at  $300^\circ\text{C}$  up to 331 h on the electrical conductivity of the unirradiated alloy is shown in Fig. 6. After 331 h (time of irradiation to a dose  $\sim 0.2$

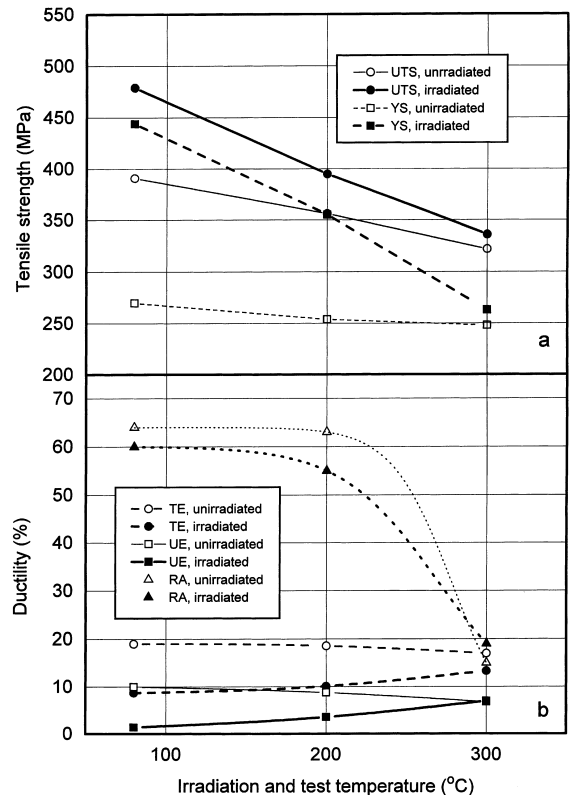


Fig. 2. Effect of irradiation to a dose of  $\sim 0.2$  dpa on strength (a) and ductility (b) of CuNiCrSi alloy after holding at  $1000^\circ\text{C}$  for 1 h, furnace cooling, annealing at  $600^\circ\text{C}$  for 1 h, air cooling.

dpa) maximum increase in electrical conductivity was 6.4%. Therefore, irradiation at  $300^\circ\text{C}$  results in the main contribution to the increase of the electrical conductivity of the CuNiCrSi alloy.

#### 4. Discussion

Irradiation at  $80^\circ\text{C}$  results in pronounced hardening of CuNiCrSi alloy in the DB + A state. The increase in the yield strength of this alloy reaches  $\sim 170$  MPa after a dose of  $\sim 0.2$  dpa. The uniform elongation decreases from 9% in the initial state down to 2% after irradiation. Our experimental results on low-temperature radiation embrittlement of CuNiCrSi alloy agree well with the available data [4]. At irradiation to a dose of  $\sim 0.2$  dpa near  $100^\circ\text{C}$ , the alloy CuNiCrSi has uniform elongation not lower than GlidCop A125 and CuCrZr alloys, which are considered as candidate materials for ITER.

Comparison of the mechanical properties of the CuNiCrSi alloy after various heat treatments and, correspondingly, with various strength levels after irradiation at  $300^\circ\text{C}$  is given in Fig. 7. This comparison is

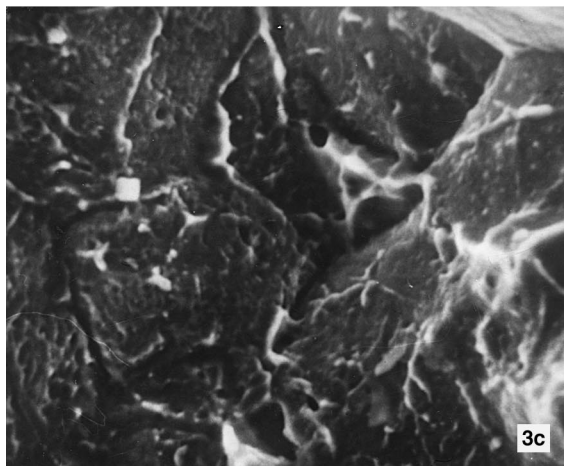
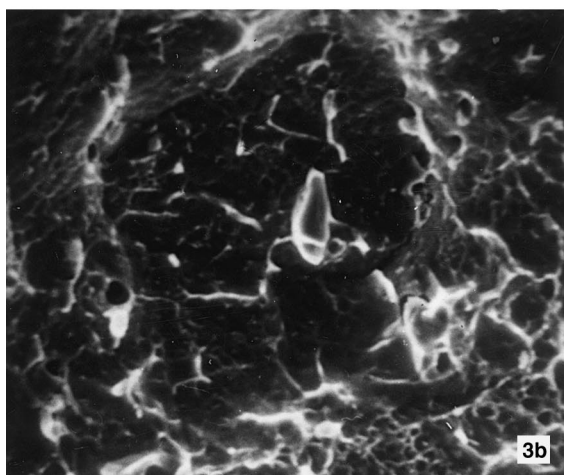
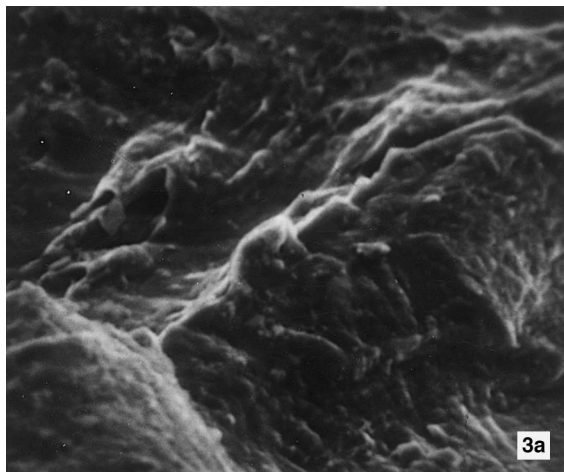


Fig. 3. Fracture surface of CuNiCrSi alloy tested at 400°C (×1000): unirradiated (a), unirradiated and exposed at 300°C for 331 h (b), irradiated to a dose of ~0.2 dpa at 300°C.

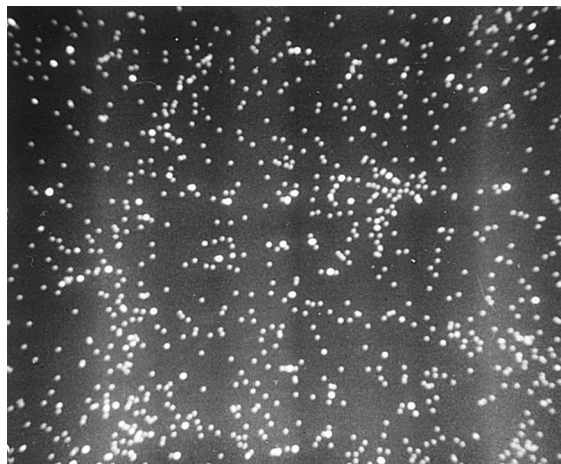


Fig. 4. Micrograph of Ni distribution on fracture surface of CuNiCrSi alloy irradiated to a dose of ~0.2 dpa at 300°C and tested at 400°C (×3000).

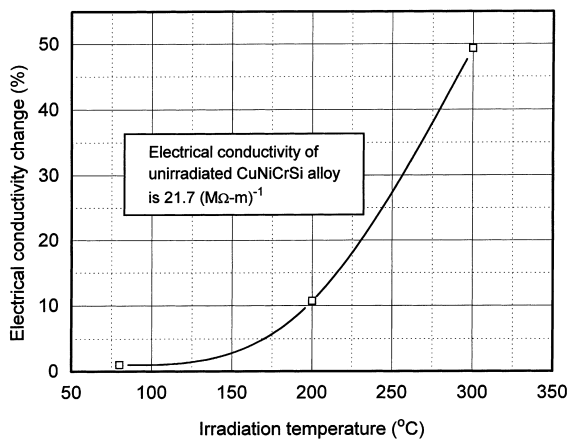


Fig. 5. Electrical conductivity change of CuNiCrSi alloy versus irradiation temperature (damage level of ~0.2 dpa).

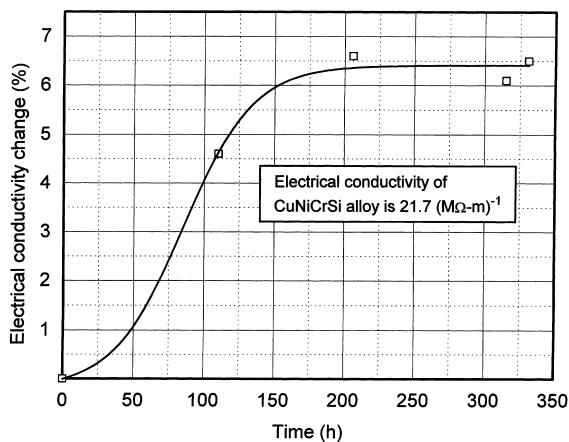


Fig. 6. Effect of holding time at 300°C on electrical conductivity change of CuNiCrSi alloy.

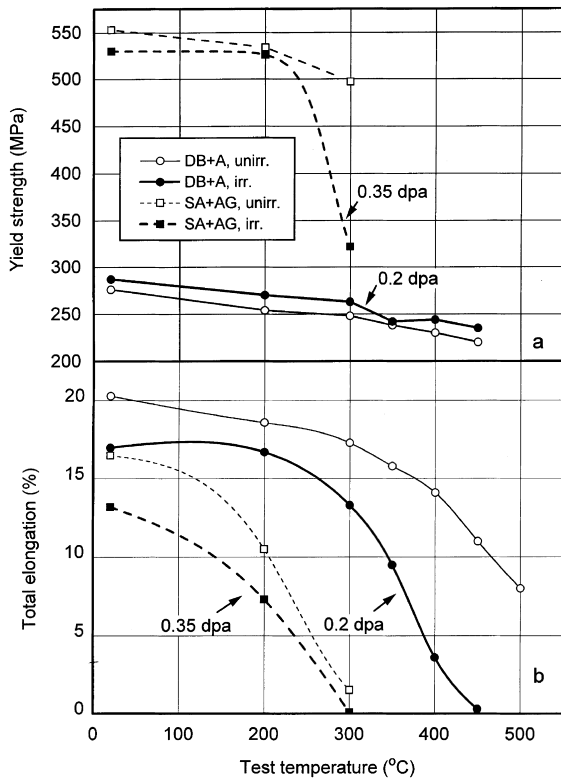


Fig. 7. Irradiation effect at 300°C on yield strength (a) and total elongation (b) of CuNiCrSi alloy.

conventional, as the irradiation has been performed with different damage levels. Irradiated at 300°C and tested in the range of 20–200°C, this alloy in a DB + A state has a yield strength less than half of the SA + AG state (Fig. 7(a)). However, at irradiation and test temperatures of 300°C, its strength in a the DB + AG state is reduced by a factor of 1.2–1.3 as compared with a SA + AG state. The ductility of the irradiated alloy in the DB + A state substantially exceeds that of the alloy in the SA + AG state (Fig. 7(b)). Therefore, the alloy in the DB + A state has higher radiation resistance at 300°C than after SA + AG. It is necessary to emphasise that this material in the DB + A state has (Figs. 2 and 7) satisfactory ductility (UE ~ 7%, TE ~ 12% and RA ~ 19%) and strength (UTS ~ 340 MPa and YS ~ 260 MPa) after irradiation and testing at  $T_{irr.} = T_{test} = 300^{\circ}C$ .

The results of performed investigations show that the irradiation of CuNiCrSi alloy brings about an essential increase of the electrical conductivity at 200–300°C. The data obtained agree well with those of papers [5,6] which points out that irradiation of precipitation-hardened copper alloys with moderate electrical conductivity in an initial unirradiated state resulted in its noticeable increase, for example in solution annealed CuNiBe alloy irradiated to a dose of 0.3 dpa (Fig. 8). High dose ir-

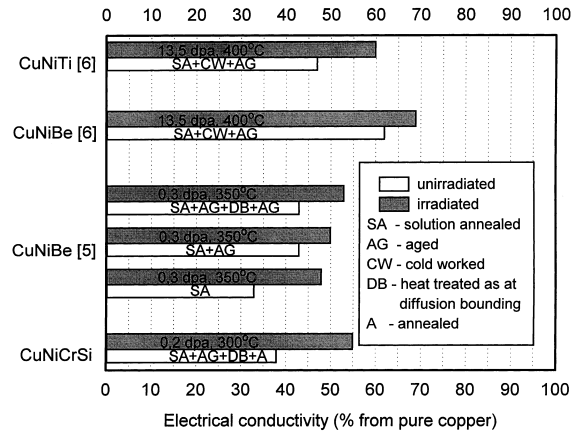


Fig. 8. Irradiation effect on electrical conductivity of precipitation-hardened copper alloys.

radiation (13.5 dpa) at 400°C, as well as low dose irradiation resulted in an increase in the electrical conductivity of CuNiBe and CuNiTi alloys.

X-ray micro analysis data obtained in this investigation on numerous areas with enriched nickel concentration in CuNiCrSi alloy irradiated at 300°C, can be explained by the increase in the hardening phase  $Ni_3Si$ . Taking this into account, the increase in electrical conductivity of the alloys presented in Fig. 8 can be related to the process of their coagulation under irradiation. It is known [2,7] that irradiation of copper alloys with high electrical conductivity in its initial unirradiated state results in its decrease as a result of accumulation of decay products (Ni and Zn mainly) and radiation defects (vacancies and dislocation loops). Hence, it be assumed that aging in precipitation-hardened copper alloys under moderate irradiation dose causes an increase in electrical conductivity. However, such explanation can not be applied to the observed increase in electrical conductivity of CuNiBe and CuNiTi alloys after high dose irradiation when the accumulation of considerable amounts of decay products has taken place and must cause a decrease in the property under consideration. On the whole, it should be noted that in precipitation-hardened copper alloys under irradiation aging occurs at 300–400°C.

5. Conclusions

It is concluded that CuNiCrSi alloy in a high strength state provided by solution annealing and aging with or without cold work has low radiation resistance at a dose of ~0.35 dpa. After thermal simulation of diffusion bonding and annealing, the alloy exhibits high resistance to radiation embrittlement under an irradiation to damage level of ~0.2 dpa at 100–300°C. Irradiation

above 200°C results in an increase in the electrical conductivity of this material.

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