



# Effect of neutron irradiation on mechanical properties of Cu/SS joints after single and multiple HIP cycles

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## Abstract

The present design of the ITER plasma facing components consists of a copper alloy heat sink layer between plasma facing materials and stainless steel structure. The main option for manufacturing these components is hot isostatic pressing (HIP) method and several HIP thermal cycles are foreseen for manufacturing of the complete blanket module. Mechanical characterisation of HIP joints between dissimilar metals is a complicated issue, where information on mechanical properties of base alloys, metallurgy of the HIP joints and mechanical testing methods will be required. The tensile and three point bend tests produced different fracture modes, depending on test temperature, applied HIP thermal cycles and neutron irradiation. The fracture mode was either ductile fracture of copper alloy or joint interface fracture. The mechanical properties of the HIP joint specimens were dominated by strength mismatch of the base alloys which was affected by HIP thermal cycles and neutron irradiation. © 2000 Elsevier Science B.V. All rights reserved.

## 1. Introduction

The present design of the ITER plasma facing components consists of a copper alloy heat sink layer between plasma facing materials and stainless steel structure. Hot isostatic pressing (HIP) is applied for bonding copper alloys to stainless steel. In the present design several HIP thermal cycles is foreseen in manufacturing the complete divertor or blanket modules. Limited amount of experimental results exist on the effects of successive HIP thermal cycles on the properties of the base alloys and the joints. In this study metallurgy, tensile and fracture toughness properties of HIP joints between CuAl25 IG0 and CuCrZr alloys and 316 L(N) stainless steel have been investigated in unirradiated and neutron irradiated condition.

Tensile [1–5], shear [6], impact [5] and fracture toughness [6–11] tests have been applied to characterise the mechanical performance of the HIP joint interface between copper alloys and austenitic stainless steel. The

common observation of all these experiments have been that the failure does not occur at the joint interface between copper alloys and stainless steel but in the copper alloys. In the present paper, tensile and fracture toughness test results on HIP joint specimens of CuAl25 IG0 and CuCrZr alloys with stainless steel 316 L(N) are presented and the effects of multiple HIP thermal cycles, neutron irradiation and testing methods are briefly discussed.

## 2. Experimental

Two types of copper alloys, dispersion strengthened CuAl25 IG0 (OMG Americas) and precipitation hardened CuCrZr (Outokumpu Oy), and their joints with austenitic stainless steel 316L(N) IG were studied. The joints were produced by HIP at 960°C for 3 h at a pressure of 120 MPa followed by 1 h furnace cooling and subsequent heat treatment at 460°C for 2 h for CuCrZr alloy HIP joint. Multiple HIP thermal cycles were simulated by giving two additional heat treatments at 960°C for 3 h followed by furnace cooling and subsequent heat treatment at 460°C for 2 h for CuCrZr HIP joint specimens. Test specimens were machined by using

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electric discharge machining from the plates of the above described copper alloys and from the joints. The tensile and the three point bend specimens of the joints were taken across the joint interface so that the tensile axis occurred across the joint interface in tensile tests and crack propagation occurred along the joint interface during fracture resistance tests. The thickness and the gauge length of the tensile specimens were 0.3 and 20 mm, respectively. Tensile testing was performed at a strain rate of  $1.3 \times 10^{-3} \text{ s}^{-1}$  and at the elevated temperatures tests were carried out in a vacuum of about  $10^{-5}$  torr. Three point bend test specimens of dimensions  $3 \times 4 \times 27 \text{ mm}^3$  were used for fracture toughness testing. All specimens were precracked to the initial crack length to specimen width ratio of about 0.5 and 20% side grooved. The fracture resistance curves were determined at temperatures in the range of 22–350°C in a silicon oil bath following the ASTM E1737-96 standard procedure.

The tensile and fracture toughness specimens were irradiated with fission neutrons in the DR-3 reactor at Risø National Laboratory at temperatures in the range of 50–350°C to a neutron fluence of  $1.5 \times 10^{24} \text{ n/m}^2$  ( $E > 1 \text{ MeV}$ ) corresponding to a displacement dose of 0.3 dpa (NRT). Irradiations were carried out with a neutron flux of  $2.5 \times 10^{17} \text{ n/m}^2 \text{ s}$  corresponding to a damage rate of  $5 \times 10^{-8} \text{ dpa (NRT)/s}$ . Irradiations were performed in the atmosphere of helium or a mixture of helium and argon. Irradiations at 200 and 350°C were carried out in temperature controlled rigs where the irradiation temperature was monitored, controlled and recorded continuously throughout the whole irradiation period.

### 3. Results

#### 3.1. Microstructure and composition of joints

The microstructures of the joints between copper alloys and 316 L(N) austenitic stainless steel developed during the HIP thermal cycle were characteristic for the copper alloys in question. In the CuCrZr alloy joint, zirconium carbonitrides precipitated readily at the joint interface with 316 L(N) stainless steel. Due to the subsequent decrease in the stability of the austenite – decrease in nitrogen, carbon and nickel contents – a ferrite layer about 3  $\mu\text{m}$  thick formed within the austenitic stainless steel side of the joint. The multiple HIP thermal cycles further enhanced diffusion of alloying elements and as a result, for example, zirconium carbonitride formed almost a continuous layer, ferrite layer extended to about 7  $\mu\text{m}$  and iron–chromium-rich precipitates appeared within copper alloy side of the joint. The corresponding microstructure of the CuAl25 IG0 joint was characterised by the formation of iron–chromium-rich precipitates in copper alloy within a distance of about 30–40  $\mu\text{m}$  from the joint interface after the single

HIP thermal cycle. The repeated HIP thermal cycles seemed to increase the size and density of the precipitates and after triple HIP cycles the zone containing precipitates extended to about 50–60  $\mu\text{m}$  from the interface. The multiple HIP cycles also led to the formation of fine scale porosity along and close to the joint interface. In both copper alloys there was a clear indication that after HIP thermal cycles the joint interface shifted, less than 1  $\mu\text{m}$ , into the stainless steel side of the joint interface.

#### 3.2. Tensile tests

Figs. 1 and 2 illustrate the tensile behaviour of single and double HIP specimens of CuCrZr and CuAl25 IG0 alloys with stainless steel both in unirradiated and neutron irradiated conditions. Double HIP joint specimens showed consistently lower tensile strength values in both copper alloys HIP joints when compared with single HIP joint specimens. In both copper alloys HIP joint specimens the neutron irradiation induced a significant increase in yield strength and decrease in work hardening ability at temperatures below 200°C. In tensile tests conducted at 350°C some hardening and a clear recovery in work hardening was observed in the CuCrZr alloy HIP joints whereas almost no ductility was observed in CuAl25 IG0 alloy HIP joint specimens after neutron irradiation.

#### 3.3. Three point bend tests

Figs. 3 and 4 illustrate the normalised load and load line displacement curves of single and triple HIP joint specimens of CuCrZr and CuAl25 IG0 alloys with stainless steel in unirradiated and neutron irradiated conditions at different test temperatures. The observed displacement values at maximum loads were about an

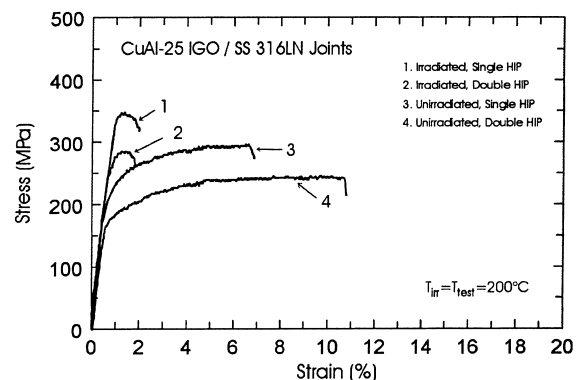


Fig. 1. Tensile stress-strain curves for single and double HIP joint specimens of CuAl25 IG0 alloy with stainless steel 316 L(N) in unirradiated and neutron irradiated conditions at 200°C.

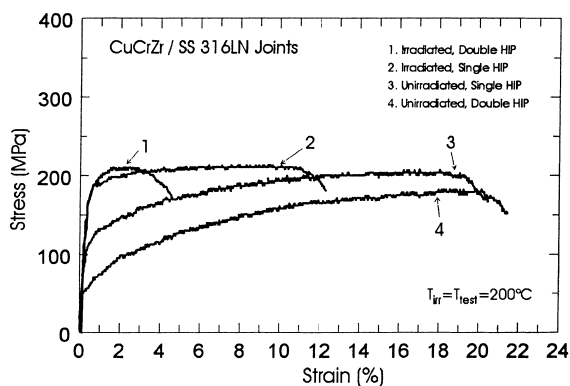


Fig. 2. Tensile stress-strain curves for single and double HIP joint specimens of CuCrZr alloy with stainless steel 316 L(N) in unirradiated and neutron irradiated conditions at  $200^\circ\text{C}$ .

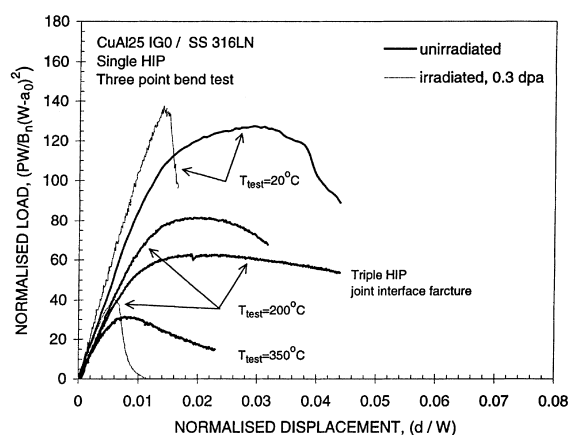


Fig. 3. Normalised load and load line displacement curves for single HIP joint specimens of CuAl25 IG0 alloy with stainless steel 316 L(N) in unirradiated and neutron irradiated conditions at different temperatures.

order of magnitude smaller in CuAl25 IG0 alloy compared to that in CuCrZr alloy HIP joint specimens. In CuCrZr alloy HIP joints the crack deviated from the joint interface and propagated in the CuCrZr matrix at temperatures below  $200^\circ\text{C}$ . The fracture mode changed to joint interface fracture, along the zirconium carbonitride layer, in both single and triple HIP CuCrZr alloy joint specimens at  $350^\circ\text{C}$ . In single HIP CuAl25 IG0 alloy joints crack propagated in copper matrix close and parallel to HIP joint interface at all test temperatures. After triple HIP thermal cycle, the fracture mode changed to joint interface fracture. The multiple HIP joint specimens showed consistently lower load values in both copper alloys HIP joints when compared with single HIP joint specimens.

The effect of neutron irradiation on the normalised load and load line displacement curves of single HIP

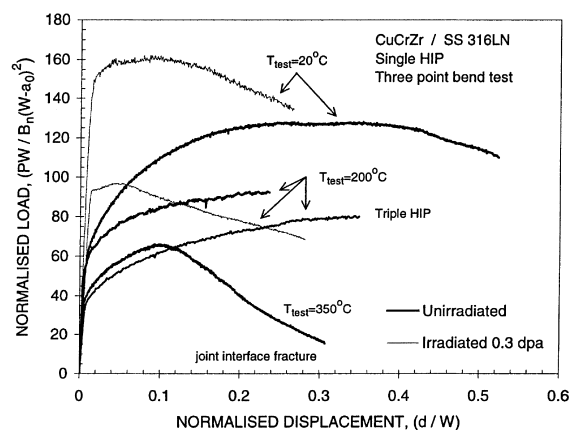


Fig. 4. Normalised load and load line displacement curves for single HIP joint specimens of CuCrZr alloy with stainless steel 316 L(N) in unirradiated and neutron irradiated conditions at different temperatures.

CuCrZr alloy joints showed significant hardening and lower displacement values at maximum load at temperatures below  $200^\circ\text{C}$  when compared with those in unirradiated condition. In the CuAl25 IG0 alloy HIP joints, the measured loads and displacements after neutron irradiation were lower than those in the unirradiated condition. The corresponding fracture toughness of CuAl25 IG0 alloy HIP joints were an order of magnitude lower than that of CuCrZr alloy HIP joints both in unirradiated and neutron irradiated conditions.

#### 4. Discussion

During the HIP thermal cycles different elements diffused across the joint interface and caused phase transformations and migration of the joint interface. The apparently different metallurgy of CuCrZr and CuAl25 IG0 alloy HIP joints with stainless steel originates from the differences in the activity or chemical potentials of elements on both sides of the joint interface. During the HIP thermal cycle at  $960^\circ\text{C}$ , the precipitates in CuCrZr alloy are partly dissolved. Furthermore, the precipitation of zirconium nitrocarbides at the joint interface destabilises the austenite phase in stainless steel and causes ferrite formation. In the CuAl25 IG0 alloy, the alumina particles are stable during the HIP thermal cycle. However, diffusion of iron, chromium and nickel in copper leads to precipitation of iron–chromium rich-precipitates in copper close to the joint interface. Diffusion of elements, phase transformations and migration of boundaries change the local properties of the joint interfaces and further complicates the interpretation of the mechanical test results.

The mechanical characterisation of the HIP joint interfaces is not a simple straightforward issue and it requires a detailed knowledge of the base alloys, metallurgy of the joint and the applied testing methods. Interfaces between different materials are responsible for local irregularities and strength mismatch which complicates the interpretation of mechanical testing results of the HIP joint specimens. The general procedures to determine the tensile and fracture toughness properties as presented by several testing standards cannot be considered to be strictly valid for the HIP joint specimens. In the case of tensile testing, the initial yielding and final fracture is expected to occur within the softer base material of the HIP joint [3]. The tensile test results can be considered only as a screening test of the joint quality showing that the strength of the actual HIP joint interface is higher than that of the softer material. In the case of fracture mechanical tests the finite element modelling on the effect of strength mismatch on the J-integral solution indicate that the effect of strength mismatch is relatively small on the macroscopic J-integral solution [6,12]. However, the strength mismatch may alter the local conditions at the crack tip which in turn may affect the fracture process. The plastic strain have a tendency to localise on the side of the softer material of the HIP joint due to strength mismatch. With increasing strength mismatch the plastic strain and hydrostatic stress concentrate at the joint interface and at the notch tip as illustrated in Fig. 5 [6].

The strength mismatch of the base alloys and the local properties of the joint interface depend on the testing temperature and are also affected by multiple HIP thermal cycles and by neutron irradiation. Both the relative amount of softening due to multiple HIP ther-

mal cycles and the relative amount of hardening due to neutron irradiation have a tendency to increase the strength mismatch relative to the single HIP condition. The increase in strength mismatch causes stronger localisation of plasticity in the softer alloy and also at the joint interface. In the case of HIP joint specimens of CuCrZr and CuAl25 IG0 alloys with 316 L(N) stainless steel the situation is complicated and in addition to initial strength mismatch – yield strength ratio – a knowledge of work hardening properties of the two alloys (i.e., copper alloys and stainless steel) is also needed.

The applied stress and strain states are higher and more localised at the actual HIP joint interface in three point bend tests of precracked bend specimens than that in tensile test with smooth tensile specimens. This difference in stress and strain states may lead to different fracture behaviour depending on the strength mismatch and the local properties of the HIP joint interface. The HIP joint specimens of CuCrZr alloy showed ductile fracture in the base material CuCrZr in tensile tests at all test temperatures but an interface fracture in three point bend test at 350°C. The transition to joint interface fracture was also observed in the triple HIP joint specimens of CuAl25 IG0 alloy at temperatures above 200°C.

Although the tensile and three point bend test results of the HIP joint specimens are not directly comparable with those of the corresponding base copper alloys, the HIP joint specimens showed consistently lower strength values when compared to strength of the copper alloys in the prime age condition [6–8]. This strength reduction is also consistent with the observed decrease in the shear strength of the copper alloy due to HIP thermal cycles [6]. The applied HIP thermal cycles consisted of slow cooling phase, e.g., furnace cooling, after HIP annealing temperature which is expected to result in a low supersaturation of alloying elements in CuCrZr alloy and low strength after precipitation heat treatment. Multiple HIP thermal cycles further reduced the strength of both copper alloys HIP joint specimens possibly due to coarsening of precipitates and grains in CuCrZr alloy and further recovery of dislocation microstructure in the CuAl25 IG0 alloy.

## 5. Conclusions

Multiple HIP thermal cycles enhanced the diffusion of elements across the HIP joint interface between CuCrZr as well as CuAl25 IG0 alloys with stainless steel 316 L(N). This led to phase transformations, precipitation, porosity formation and migration of the joint interfaces. Multiple HIP joint specimens showed consistently lower strength in both copper alloys when compared to that of single HIP joint specimens.

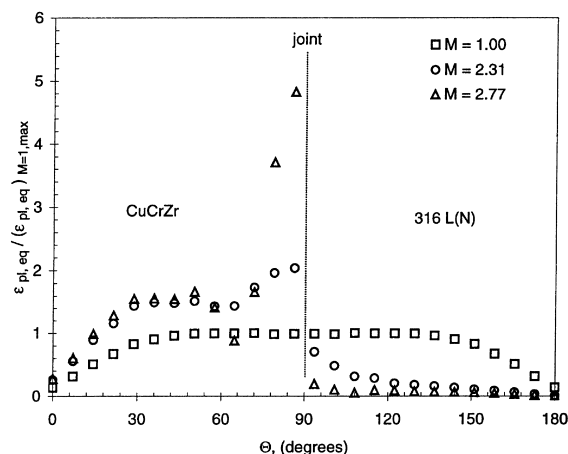


Fig. 5. Distribution of normalised equivalent plastic strain at the crack tip with different values of strength mismatch showing localisation of plasticity in softer alloy of the joint and at the joint interface with increasing strength mismatch [6].

The tensile and three point bend tests led to different type of fracture modes. In tensile tests the fracture mode was ductile fracture of copper alloys at all test temperatures and conditions. In three point bend test with precracked specimens the fracture mode changed to joint interface fracture in the single and triple HIP CuCrZr alloy at 350°C and in the triple HIP CuAl25 IG0 alloy joint specimens at temperatures above 200°C. The fracture behaviour of HIP joint specimens were dominated by the strength mismatch between the base alloys.

After neutron irradiation a substantial amount of hardening and ductility in tensile and three point bend tests was observed in CuCrZr alloy HIP joint specimens compared to that in CuAl25 IG0 alloy HIP joint specimens.

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### References

- [1] S. Sato, T. Kuroda, T. Kurasawa, K. Furuya, I. Togami, H. Takatsu, *J. Nucl. Mater.* 233–237 (1996) 940.
- [2] A. Lind, Studsvik Report M-97-41, Studsvik, 1997.
- [3] G. Le Marois, H. Burlet, J.M. Gentzbittel, L. Briottet, F. Saint-Antonin, Note Technique D.E.M. No32/97, CEA/CEREM, Grenoble, 1997.
- [4] S.A. Fabritsiev, A.S. Pokrovsky, D.J. Edwards, S.J. Zinkle, A.F. Rowcliffe, *J. Nucl. Mater.* 258–263 (1998) 2069.
- [5] S. Sato, T. Hatano, T. Kuroda, S. Hara, M. Enoeda, H. Takatsu, *J. Nucl. Mater.* 258–263 (1998) 265.
- [6] S. Tähtinen, M. Pyykkönen, B.N. Singh, P. Toft, VTT Report VALB282, Espoo, March 1998.
- [7] S. Tähtinen, M. Pyykkönen, B.N. Singh, P. Toft, in: M.L. Hamilton, A.S. Kumar, S.T. Rosiski, M.L. Grossbeck (Eds.), *Effects of Radiation on Materials 19th International Symposium, ASTM-STP 1366*, American Society for Testing and Materials, West Conshocken, PA, 2000, p. 1243.
- [8] S. Tähtinen, M. Pyykkönen, P. Karjalainen-Roikonen, B.N. Singh, P. Toft, *J. Nucl. Mater.* 258–263 (1998) 1010.
- [9] M. Pyykkönen, S. Tähtinen, B.N. Singh, P. Toft, in: B. Beaumont, P. Libeyre, B. de Gentile, G. Tonon (Eds.), *Proceedings of the 20th Symposium on Fusion Technology, Marseille, France, 7–11 September 1998*. Fusion Technology 1998, Association Euratom-CEA, vol. 1, p. 173.
- [10] H. Burlet, L. Guetaz, P. Bucci, J.M. Gentzbittel, I. Chu, N. Scheer, Note Technique DEM 98/75, CEA-CEREM, December 1998.
- [11] T. Hatano, M. Kanari, S. Sato, M. Gotoh, K. Furuya, T. Kuroda, M. Saito, M. Enoeda, H. Takatsu, *J. Nucl. Mater.* 258–263 (1998) 950.
- [12] A. Laukkanen, P. Karjalainen-Roikonen, P. Moilanen, S. Tähtinen, in: *12th European Conference on Fracture, Fracture from Defects, ESIS*, University of Sheffield, UK, 14–18 September 1998, p. 6.