

Journal of Nuclear Materials 307-311 (2002) 1547-1553



www.elsevier.com/locate/jnucmat

# Properties of copper-stainless steel HIP joints before and after neutron irradiation

S. Tähtinen<sup>a,\*</sup>, A. Laukkanen<sup>a</sup>, B.N. Singh<sup>b</sup>, P. Toft<sup>b</sup>

<sup>a</sup> VTT Industrial Systems, P.O. Box 1704, FIN-02044 VTT, Finland <sup>b</sup> Materials Research Department, Risø National Laboratory, DK-4000 Roskilde, Denmark

# Abstract

The tensile and fracture behaviour of CuCrZr and CuAl25 IG0 alloys joint to 316L(N) stainless steel by hot isostatic pressing (HIP) have been determined in unirradiated and neutron-irradiated conditions. The tensile and fracture behaviour of copper alloy HIP joint specimens are dominated by the properties of the copper alloys, and particularly, by the strength mismatch and mismatch in strain hardening capacities between copper alloys and stainless steel. The test temperature, neutron irradiation and thermal cycles primarily affect the copper alloy HIP joint properties through changing the strength mismatch between the base alloys. Changes in the loading conditions i.e. tensile, bend ( $J_1$ ) and mixed-mode bend ( $J_1/J_{II}$ ) lead to different fracture modes in the copper alloy HIP joint specimens. © 2002 Elsevier Science B.V. All rights reserved.

#### 1. Introduction

The first wall and divertor structures of the International Thermonuclear Experimental Reactor (ITER) are multilayer components consisting of austenitic stainless steel, copper alloys and plasma facing armour materials. The copper to stainless steel joints have to withstand the thermal and mechanical loads in the environment of fusion neutrons during the normal operation of ITER. To evaluate the properties of these joints several types of mechanical and fracture tests have been applied. There is, however, no commonly accepted criterion for qualification or structural design for dissimilar metal joint or components [1–4]. This study describes the work carried out to characterise the copper alloy to stainless steel hot isostatic pressing (HIP) joints in the ITER relevant conditions.

# 2. Experimental details

The joints between CuCrZr and CuAl25 IG0 alloys and austenitic stainless steel type 316L(N) IG0 were

produced by the HIP method at 960 °C for 3 h at a pressure of 120 MPa followed by slow cooling  $\simeq 20$ °C min<sup>-1</sup>. The CuCrZr HIP joint specimens were additionally heat treated at 460 °C for 2 h followed by air cooling. The multiple HIP thermal cycles were simulated by repeating the above heat treatment without any applied pressure. Additionally, a long term annealing at 960 °C for 50 h in vacuum was performed for the both copper alloy HIP joint specimens. The applied heat treatments are summarised in Table 1. The tensile  $(0.3 \times 3 \text{ mm}^2)$ , gauge length 7 mm), the threepoint bend  $(3 \times 4 \times 27 \text{ mm}^3)$  and the four-point bend  $(10 \times 10 \times 55 \text{ mm}^3)$  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 following the ASTM E1737-96 standard procedure. Mixed-mode loading tests were carried out using an asymmetric four-point bend test. Test 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  $1.5 \times 10^{24}$  n/m<sup>2</sup> (E > 1 MeV) corresponding to a displacement dose of 0.3 dpa (NRT) [5,6].

<sup>&</sup>lt;sup>\*</sup>Corresponding author. Tel.: +358-9 456 6859; fax: +358-9 456 7002.

E-mail address: seppo.tahtinen@vtt.fi (S. Tähtinen).

Material	Heat treatment							
CuCrZr	Prime aged	960 °C, 2 h, sc <sup>a</sup> , 460 °C, 2 h						
CuCrZr, 316L(N)	HIP cycle	960 °C, 3 h, 120 MPa, sc <sup>a</sup> , 460 °C, 2 h						
CuCrZr, 316L(N)	Double HIP cycle	HIP cycle + 960 °C, 3 h, sc <sup>a</sup> , 460 °C, 2 h						
CuAl-25	Prime aged	980 °C, 2 h						
CuA125 IG0, 316L(N)	HIP cycle	960 °C, 3 h, 120 MPa, sc <sup>a</sup>						
CuAl25 IG0, 316L(N)	Double HIP cycle	HIP cycle + 960 °C, 3 h, sc <sup>a</sup>						

Table 1Materials and applied heat treatments

<sup>a</sup> Slow cooling  $\simeq 20$  °C min<sup>-1</sup>.

## 3. Results and discussion

## 3.1. Tensile tests

Fig. 1 illustrates the tensile stress-strain curves for the unirradiated copper base alloys and stainless steel after different HIP thermal cycles and heat treatments. The strength of the prime aged CuCrZr alloy is significantly reduced due to HIP thermal cycle with slow cooling rate of  $\simeq 20 \,^{\circ}\text{Cmin}^{-1}$  and further reduction in strength is observed after double HIP thermal cycle. In contrast to CuCrZr, the heat treatments simulating HIP



Fig. 1. Tensile stress strain curves of (a) CuCrZr and 316L(N) stainless steel (b) CuAl25 IG0 and 316L(N) stainless steel together with corresponding HIP joint specimens after different heat treatments. The applied heat treatments are summarised in Table 1.

thermal cycle given to the CuAl25 IG0 alloy had no significant effects on the tensile properties. The tensile behaviour of the HIP joint specimens is dominated by the tensile properties of the corresponding base alloys. Accordingly, the tensile strength of the HIP joint specimen is comparable with that of corresponding copper alloy and can be used as a first assessment of the HIP joint quality [1,2].

The tensile stress-strain curves of the CuCrZr and CuAl25 IG0 HIP joint specimens after single and double HIP thermal cycles in the temperature range of 22–350 °C in unirradiated and neutron-irradiated conditions are shown in Fig. 2. Both copper alloy HIP joint specimens showed significant work hardening capability in the above temperature range in the unirradiated condition. It is noted that the observed work hardening of the CuAl25 IG0 HIP joint specimens arise due to lower yield strength of stainless steel compared to that of the base CuAl25 IG0 alloy (see Fig. 1). The double HIP thermal cycle increased the ductility of both copper alloys HIP joint specimens similar to copper and stainless steel base alloys (see Fig. 1). The neutron irradiation induced a significant increase in the yield strength and a decrease in the work hardening capability particularly at temperatures below 200 °C in single and double HIP joint specimens of both copper alloys. This is consistent with the



Fig. 2. Tensile stress-strain curves for single and double HIP joints between (a) CuCrZr and 316L(N) and (b) CuAl25 IG0 and 316L(N) stainless steel in unirradiated and neutron-irradiated conditions at different temperatures. Note significant work hardening capacity in HIP joint specimens in unirradiated condition.

effect of irradiation on mechanical properties of the base copper alloys [7,8]. In tensile tests conducted at 350 °C, initial hardening and decrease in ductility in both copper alloy HIP joint specimens were observed after neutron irradiation. It is noted that softening and recovery of ductility particularly in the CuCrZr base alloy has been reported in tensile tests conducted at 350 °C under neutron-irradiated conditions [7,8]. The observed decrease in ductility of the HIP joint specimens indicates an intrinsic weakening of the joint interface structure after neutron irradiation to a dose level of 0.3 dpa at 350 °C.

#### 3.2. Fracture resistance tests

The effect of temperature and neutron irradiation on the initiation fracture toughness of CuCrZr and CuAl25 IG0 HIP joint specimens after single and multiple HIP thermal cycles are shown in Fig. 3. In unirradiated conditions, the multiple HIP thermal cycle slightly increased the fracture toughness of both copper alloy HIP joint specimens at temperatures below 200 °C when compared to the fracture toughness of the single HIP thermal cycle specimens. This is in accordance with the



Fig. 3. Initiation fracture toughness of single and multiple HIP joint specimens of (a) CuCrZr/316L(N) and (b) CuAl25 IG0/316L(N) stainless steel in unirradiated and neutron-irradiated condition at different temperatures. Note that fracture propagated within copper alloy except in CuCrZr/316L(N) single and multiple HIP joints at 350 °C and in CuAl25 IG0/316L(N) multiple HIP joints at 200 and 350 °C.

increase on the tensile ductility due to double HIP thermal cycle. When tested at 350 °C no marked differences in fracture toughness values between single and multiple HIP joint specimens were observed. It is noted that in the single HIP joint specimens the fracture occurred on the copper alloy side of the joint in all cases except in the CuCrZr alloy HIP joint specimens when tested at 350 °C where fracture occurred along the HIP joint interface. The change in fracture path was associated with a dramatic decrease in fracture toughness of CuCrZr HIP joint specimens particularly after neutron irradiation. As no major changes in the strength mismatch between CuCrZr alloy and 316L(N) stainless steel were observed in the studied temperature range, the microstructure of the joint interface is expected to become inherently weak at 350 °C [9–13]. After multiple HIP thermal cycles, fracture occurred along the HIP joint interface also in CuAl25 IG0 alloy HIP joint specimens at temperatures above 200 °C. Neutron irradiation to a dose level of 0.3 dpa induced a clear decrease in the fracture toughness of both copper alloy HIP joint specimens and no marked differences in fracture toughness between single and multiple HIP joint specimens were observed. Fracture toughness of CuCrZr HIP joints were clearly higher that of CuAl25 IG0 HIP joints in neutron-irradiated condition.

#### 3.3. Mixed-mode loading tests

By virtue of the strength mismatch of the base alloys (i.e. copper alloys and stainless steel) the intrinsic loading condition in the vicinity of the crack tip is of a mixed-mode type even in the mode I type three-point bend fracture resistance tests. It has been shown that mismatch in strain hardening capabilities promote localisation of plastic strain in the copper alloy side of the joint and near the joint interface [1,8,14]. When subjected to an external mixed-mode loading, the effects of strength mismatch to the fracture resistance are highlighted via even higher concentrations of deformation in copper near the interface locations. The load-displacement curves for HIP joint specimens of the CuAl25 IG0 and CuCrZr alloys with different loading modes are shown in Fig. 4. The increase in load carrying capacity with increasing mode II type of loading was observed for both copper alloy HIP joint specimens. It is noted that in the CuAl25 IG0 HIP joint specimens the mixed-mode loading condition resulted in a drastic change of the fracture type, the stable ductile crack propagation changed to low toughness ductile fracture of the joint. In the CuCrZr HIP joint specimens crack propagation retains its stability particularly due to larger strain hardening capability compared to that of the CuAl25 IG0 alloy [1,8,14].

#### 3.4. Microstructure

The microstructure of the HIP joint interface is characterised by interdiffusion and diffusion induced phase transformations. The HIP joint of the CuCrZr alloy consists of Zr precipitates at the interface and ferrite layer on the stainless steel side of the joint interface. In the case of the CuAl25 IG0 alloy joint with 316L(N) stainless steel (Fe, Cr)-rich precipitates were found on the copper alloy side of the joint interface. The microstructure is relatively stable [15–17] and multiple



Fig. 4. Load-displacement curves of unirradiated HIP joint specimens of CuCrZr/316L(N) and CuAl25 IG0/316L(N) stainless steel under mixed-mode ( $J_{\rm I}/J_{\rm II}$ ) loading at room temperature. Equivalent modal angle  $\Psi$  has a value of 90° in loading mode II and 0° in loading mode I.



1	65.85	28.01	2.27	3.05	0.02	0.81	
2	5.91	3.27	-	-	0.86	89.96	
3	0.57	0.42	-	-	93.43	5.59	
4	4.13	-	-	-	1.01	94.86	
5	71.13	14.29	0.89	0.42	0.63	12.64	
_							-

Fig. 5. SEM micrograph of single HIP joint specimens between CuCrZr and 316L(N) stainless steel after thermal heat treatment at 960 °C for 50 h in vacuum and corresponding EDX analysis on indicated locations.

HIP thermal cycles or long term annealing at HIP temperature did not change the basic microstructure of the CuAl25 IG0 alloy. It is noted that chromium reprecipitates as (Fe, Cr)-rich precipitate which was also observed in the CuCrZr alloy after multiple HIP thermal cycles. It should be noted that diffusion induced joint interface migration, distance of  $\simeq 1 \,\mu$ m, towards stainless steel occurred during HIP thermal cycle. This is clearly illustrated for CuCrZr alloy HIP joint after long term annealing in Fig. 5.

# 4. Conclusions

The tensile and fracture behaviour of HIP joint specimens of copper alloys were found to be dominated by the properties of the copper alloys, and particularly, by the strength mismatch and mismatch in strain hardening capabilities between copper alloys and stainless steel. The mismatch promote localisation of plastic strain in the copper alloy side of the joint and near the joint interface.

The test temperature, neutron irradiation and thermal cycles related to component manufacturing or operational cycles primarily affects the copper alloy HIP joint properties through changing the strength mismatch between the base alloys. The loading conditions i.e. tensile, bend  $(J_1)$  and mixed-mode bend  $(J_1/J_{II})$  induce in different fracture modes in the Cu/SS HIP joint specimens which indicate that qualification criteria for Cu/SS HIP joint properties should be based on the operational loading conditions of the multilayer components.

## Acknowledgements

This work was performed under the frame work of European Fusion Technology Programme by Associations Euratom-Tekes and Euratom-Risø.

#### References

- S. Tähtinen, A. Laukkanen, B.N. Singh, in: 21st International Symposium on Fusion Technology, 11–15 September 2000, Madrid, Spain, Fus. Eng. Des. (2001).
- [2] S. Tähtinen, B.N. Singh, in: B. Raj, K. Bhanu Sankara Rao, T. Jayakumar, R.K. Dayal (Eds.), Proceedings of the International Symposium on Materials Aging and Life Management ISOMALM 2000, vol. 3, Allied Publishers Limited, Chennai, 2000, p. 1080.
- [3] A.D. Ivanov, S. Sato, G. Le Marois, J. Nucl. Mater. 283– 287 (2000) 35.
- [4] L. Briottet, P. Bucci, H. Burlet, I. Chu, Note Technique DEM No. 99/26, CEA-CEREM, Crenoble, 1999, p. 32.
- [5] S. Tähtinen, M. Pyykkönen, B.N. Singh, P. Toft, in: M.L. Hamilton, A.S. Kumar, S.T. Rosinski, M.L. Grossbeck (Eds.), Effects of Radiation and Materials; 19th International Symposium, STP 1366, ASTM, American Society for Testing and Materials, West Conshohocken, PA, 2000, p. 1241.
- [6] A. Laukkanen, S. Tähtinen, Research report BVAL64-011132, VTT Manufacturing Technology, Espoo, 2001, p. 26.
- [7] S. Tähtinen, M. Pyykkönen, B.N. Singh, P. Toft, Research report VALB-282, VTT Manufacturing Technology, Espoo, 1998, p. 22.
- [8] M. Pyykkönen, S. Tähtinen, B.N. Singh, P. Toft, in: B. Beaumont, P. Libeyre, B. de Gentile, G. Tonon (Eds.), Fusion Technology 1998, Euratom-CEA, Marseille, 1998, p. 173.
- [9] S. Tähtinen, B.N. Singh, Researh report BVAL62-001030, VTT Manufacturing Technology, Espoo, 2000, p. 14.
- [10] S. Tähtinen, B.N. Singh, Research report BVAL62-001062, VTT Manufacturing Technology, Espoo, 2000, p. 12.
- [11] S. Tähtinen, M. Pyykkönen, P. Karjalainen-Roikonen, B.N. Singh, P. Toft, J. Nucl. Mater. 258–263 (1998) 1010.
- [12] S. Tähtinen, B.N. Singh, P. Toft, J. Nucl. Mater. 283–287 (2000) 1238.
- [13] S.A. Fabritsiev, A.S. Pokrovsky, D.J. Edwards, S.J. Zinkle, A.F. Rowcliffe, J. Nucl. Mater. 283–287 (2000) 523.
- [14] A. Laukkanen, S. Tähtinen, in: Proceedings of EURO-MAT 2000, in: D. Miannay, P. Costa, D. Francois, A.

Pineau (Eds.), Advances in Mechanical Behaviour, Plasticity and Damage, vol. 1, Elsevier Science Ltd., Oxford, 2000, p. 103.

- [15] Q. Xu, D.J. Edwards, T. Yoshiie, J. Nucl. Mater. 283–287 (2000) 1229.
- [16] S. Revol, C. Labonne, T. Portra, E. Rigal, Note Technique D.E.M. No. 74/98, CEA-CEREM, Grenoble, 1998, p. 17.
- [17] T. Hatano, M. Kanari, S. Sato, M. Gotoh, F. Furuya, T. Kuroda, M. Saito, M. Enoeda, H. Takatsu, J. Nucl. Mater. 258–263 (1998) 950.