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Evaluation of hot isostatic pressing for joining of fusion reactor structural components

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Abstract

Hot isostatic pressing (HIP) is a promising technology to fabricate the blanket structure of fusion reactors. HIP joining of solid materials has been selected as a reference fabrication method for the shielding blanket/first wall of the international thermonuclear experimental reactor (ITER). On the basis of experimental results obtained in Europe, Japan and Russia, an evaluation of HIP joining for fusion reactor structural components has been carried out. The parameters of HIP fabrication for copper alloys and stainless steels are given. The results of microscopic observations, X-ray microanalysis, tensile, impact toughness, fracture toughness and fatigue tests are presented. Material science criteria for an estimation of quality for joints fabricated by HIP are discussed.

1. Introduction

Hot isostatic pressing (HIP) is a process for the formation of powder or solid materials under the action of inert gas pressure (up to 200 MPa) at high temperatures (up to 2000°C). HIP is widely used in powder metallurgy for manufacturing near net-shape components. HIP joining was chosen as the basic method for manufacturing Cu/stainless sheet (SS) layer structures applied to the first wall and blanket for the international thermonuclear experimental reactor (ITER) [1]. An extensive research and development (R&D) program was carried out on SS/SS, Cu/Cu and Cu/SS joints, especially the latter. Development of a HIP joining technique included improvement in the metallurgical quality of the materials used and optimization of the process parameters. Results of microscopic observations and mechanical tests of HIP bonded joints have been reported [2–17].

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2. Hip joining technique

The materials used included: precipitation-hardened copper alloy CuCrZr, dispersion-strengthened copper alloys CuAl-15 and CuAl-25 (DSCu), and austenitic stainless steels 316L and 316LN. During the R&D programs, improved material specifications were developed; these are designated as 'IG' grades.

Cu/SS joints were fabricated by HIP with the following sequence of operations:

- production of steel and copper plates,
- fabrication of a steel capsule,
- preparation of the surfaces,
- evacuation and sealing of the capsules by argon-arc or electron-beam welding,
- inspection of capsule weld tightness,
- HIP process,
- dismantling of the capsules.

Surface preparation is extremely important for the fabrication of good joints. Usually it includes polishing (maximum roughness of ~0.8 μm), degreasing, rinsing and drying under Ar. Some DSCu alloys are treated at 1000°C for 1 h under secondary vacuum in order to relieve the residual stresses generated during the cross rolling of the plate and to reduce the oxides present at

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Table 1
HIP parameters of SS/SS and Cu/SS joints

Joint	HIP parameters			Intermediate layer	Ref.
	Temperature (°C)	Pressure (MPa)	Exposure (h)		
316LN/316LN	1100	150	2	–	[1]
DSCu/316LN	920–1040	120–140	2–4	–	[2–9]
DSCu/316L	1050	150	2	–	[10–16]
CuCrZr/316LN	920	120	3	Fe–42%Ni	[2,3]
	1000	130	1	Ni	[17]

the surface. The Cu and steel plates are placed in a mild steel can and degassed 12 h at 120°C before sealing [2–9]. Evacuation at 400°C for 2 h was used for the CuCrZr alloy [17]. Properties of bonded joints depend on the method of surface preparation and the metallurgical quality of materials, especially the morphology of inclusions.

HIP parameters of SS/SS and Cu/SS joints are summarized in Table 1. The HIPing temperature of SS to SS joining is about 1100°C with an exposure of 2 h at 150 MPa [1]. Good quality HIPed joints with tensile properties within the scatter band of the wrought materials can be achieved. Recent experimental data obtained in Europe [2–9] have shown that high-quality DSCu/316LN joints can be made over a wide range of HIP temperatures from 920 to 1050°C. Japanese investigators have concluded that a HIP temperature of 1050°C is an optimal condition for the HIP bonding of DSCu/SS [10–16]. Temperatures above 1050°C result in a rapid degradation of the mechanical properties for DSCu alloys. Therefore considering an uncertainty of 10°C for industrial HIP furnace temperatures, a maximum nominal value of 1040°C can be recommended.

Intermediate layers (Fe–42%Ni or Ni) have been used for joining CuCrZr alloy to SS (Table 1). Good CuCrZr/316LN joints were fabricated by using HIP schedules of 920°C/120 MPa/3 h [2] and 1000°C/130 MPa/1 h [17]. Cu/Cu joints with good mechanical properties may be made with a wide range of HIP parameters including those indicated for the Cu/SS joints in Table 1.

3. Performance of joints

3.1. Structure

Using microscopic and SEM observations, no defects were observed at the HIPped interfaces of all DSCu/SS joints studied. Fig. 1 shows typical microstructures of the CuAl-25/316L joint [14]. Typical images obtained by electron probe microanalysis (EPMA) are shown in Figs. 2 and 3 [11,17]. Fe and Cr were observed in the DSCu alloys up to ~100 μm from the interface. An intermediate layer of less than 5–10 μm thickness was formed at the interface.

Fabrication of good CuCrZr/SS joints is possible only using an Fe–42%Ni or Ni interlayer (Table 1), which inhibits the precipitation of brittle Zr carbide phases [2]. The SEM examination using the characteristic radiation of elements has shown [17] that there is an increase in zirconium content near the interface in a CuCrZr/SS joint (Fig. 4). The width of the zirconium-enriched area is 1–2 μm. Fig. 5 shows the quantitative analysis of the distribution of chemical elements across interface of the joint. The zirconium content in the interface area is ~12% which is more, by a factor of 100, than in the base CuCrZr alloy (0.1% Zr). There is detectable diffusion of Ni into the Cu alloy over a distance of ~45 μm. The Cu diffuses into the steel to a depth of ~25 μm. Judging from the maximum penetration of the elements, the joints under examination have a diffusion zone with a width of ~70 μm.

3.2. Tensile tests

The tensile properties at room temperature (RT) of the DSCu/SS joints bonded by HIP at 1050°C [10–16] were follows:

- ultimate tensile strength (UTS) of 350–400 MPa,
- yield strength (YS) of 255–280 MPa,
- total elongation (TE) of 6.5–16%.

Tensile strength of the joints was close to that of DSCu base metal. Ductility of the joints was slightly lower in comparison with the DSCu base metal. High ductility at RT can be correlated with necking located about 4–5 μm from the bond. The deformation of the DSCu side of the joint is similar to that of the base metal in this case. Numerical calculations simulating tensile tests on DSCu/SS specimens are in good agreement with these results [3]. No stress concentration near the interface occurred.

Strength of DSCu/SS joints decreases with increasing test temperature from RT to 400°C (Fig. 6). At 300°C failure occurs in the DSCu closer to the interface, and

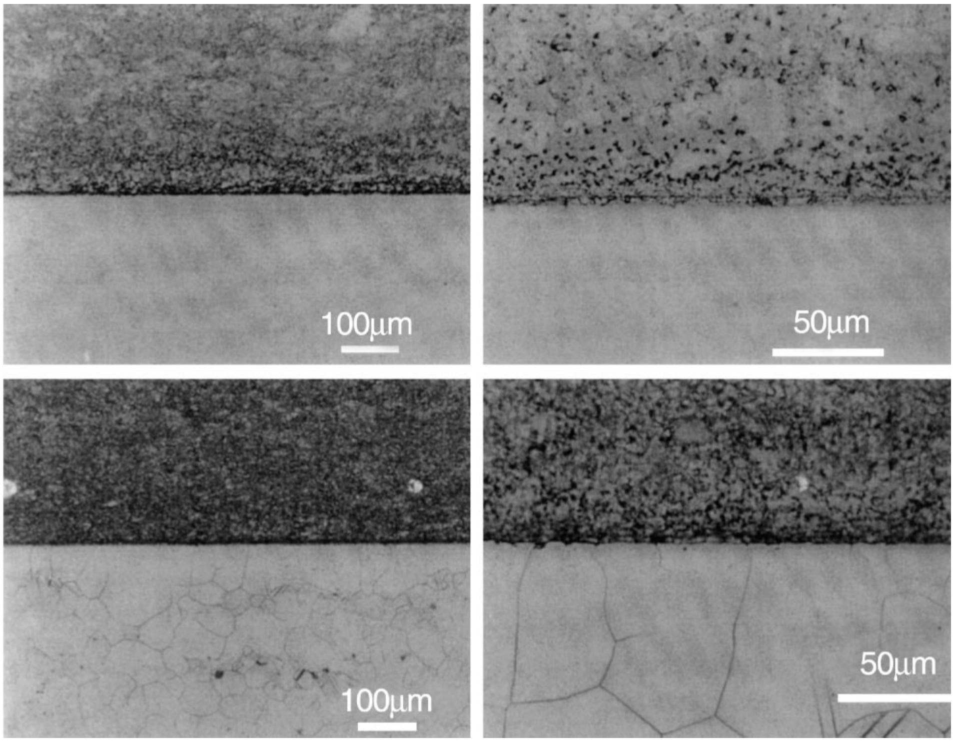


Fig. 1. Microstructure of CuAl-25/316L joint fabricated by HIP at 1050°C. Etched for Cu (a) and 316L (b) part of joint.

with a DSCu strain smaller than the total elongation of DSCu base metal. Numerical calculations carried out in Europe [3] show a high concentration of axial stress in the vicinity of the interface, on the DSCu side. This high stress could explain the brittle fracture of the DSCu alloy near the interface of the DSCu/SS joint. Small values of ductility were observed at test temperatures over 300°C.

The influence of nickel on the tensile properties of DSCu/SS joints is not yet clearly understood. Available experimental data [2,3] show that strength and ductility are reduced in this case compared with the joints fabricated without Ni. Therefore the use of a Ni interlayer is not recommended.

Analysis of experimental data on Cu/SS joints [2,17] has shown that all tensile specimens fractured in the Cu alloy part of the CuCrZr/316LN joints. The plastic deformation took place at the working part section of the Cu alloy. The strength of the joints decreases with increasing test temperature: the UTS decreased slightly from 204–233 MPa at RT to 179–210 MPa at 300°C [17]. These values can be increased to $\sim$385 and $\sim$290 MPa, respectively, if the joint is given full heat treatment for the CuCrZr alloy after the HIP cycle (990°C for 1 h, water quenched and aged at 480°C for 4 h) [2]. However bimetallic joints for fusion reactor applications have large thickness and it is not possible to achieve a sufficiently rapid cooling rate during a post-HIP heat treatment to prevent the rapid decomposition of supersaturated solid solution in the solutionized CuCrZr alloy.

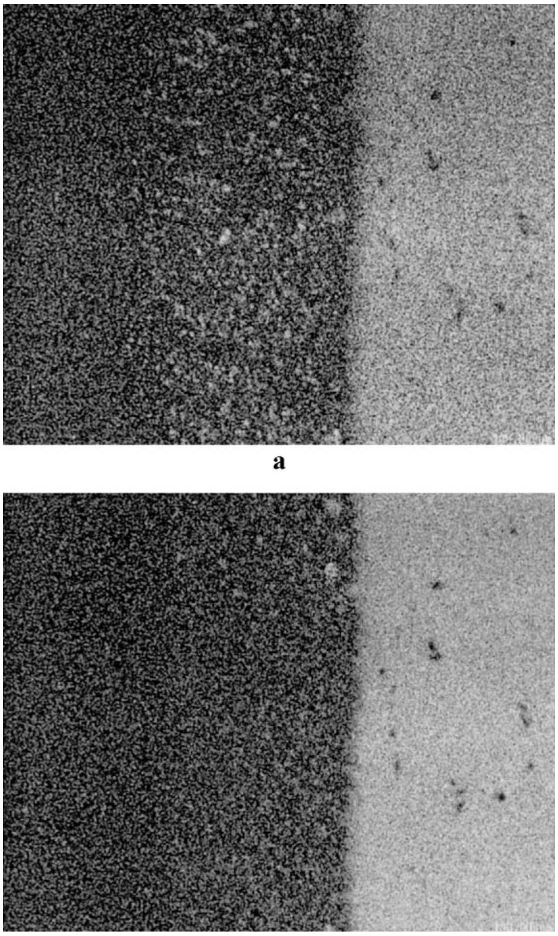
3.3. Impact toughness

Results of Charpy impact tests [10–16] are shown in Fig. 7 for the HIP bonded 316L/316L, CuAl-25/316L and CuAl-15/316L joints. The toughness increases as the HIP temperature increases. Impact toughness is 200–600 J/cm^2 at RT for the joints fabricated at 1050°C/150 MPa/2 h. The fracture was observed in the DSCu base metal for the CuAl-15/316L joint HIPped at 1050°C. The specimens for other joints fractured at the HIPped interface.

The results of Menage impact tests at RT for CuCrZr/SS specimens showed that the maximum values of impact toughness are 158–179 J/cm^2 [17]. In this case fracture of specimens initiated in the Cu part near the interface, but the interface was not ruptured.

3.4. Fatigue

The results of fatigue tests [10–16] at room temperature and elevated temperatures of 316L/316L, CuAl-15/316L and CuAl-25/316L joints are shown in Figs. 8



a

b

Fig. 2. Map of Cr (a) and Fe (b) distribution in CuAl-25/316LN joint fabricated by HIP at 1000°C/130 MPa/1 h.

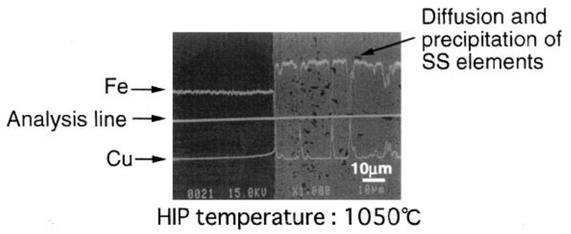


Fig. 3. EPMA line analyses of Fe and Cu observed for a CuAl-25/316L joint.

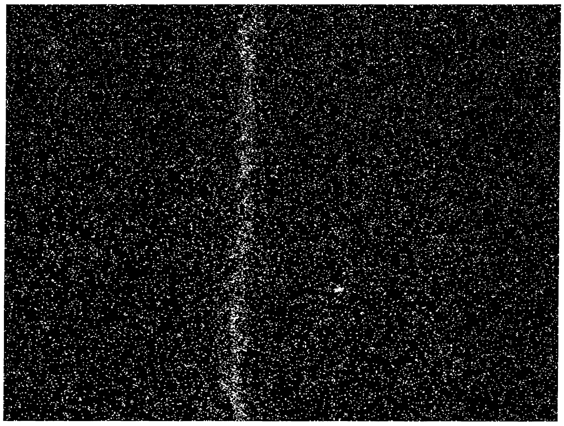


Fig. 4. Map of Zr distribution in a CuCrZr/316LN joint.

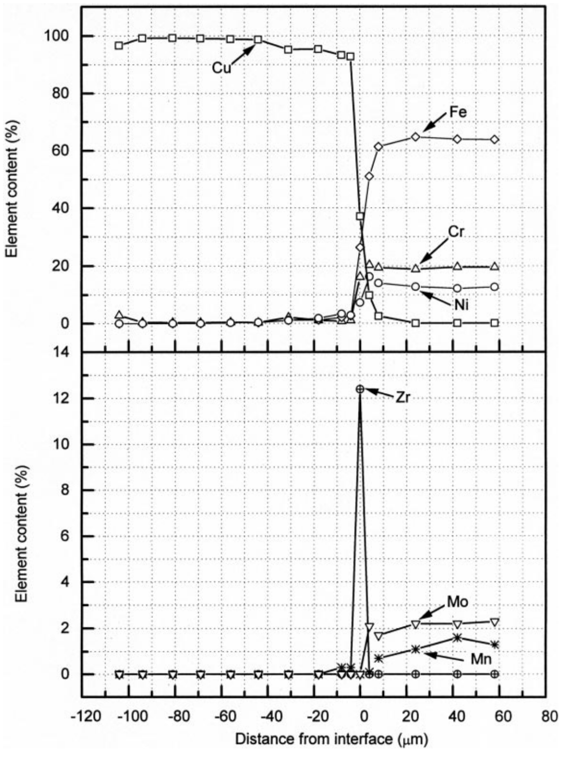


Fig. 5. Concentration profiles of elements across a CuCrZr/316LN interface.

and 9. Data for the HIP bonded joints fabricated at 1050°C and base metals are plotted in the form of the total strain range versus the number of cycles to failure. Fatigue strength of the HIP bonded 316LN/316LN joints were close to those of the base metal, while most of the data of the HIP bonded DSCu/316LN joints showed a slight degradation compared with those of the DSCu base metal.

3.5. Fracture toughness

The DSCu/SS joints bonded by HIP at 1050°C had higher fracture toughness compared with joints bonded

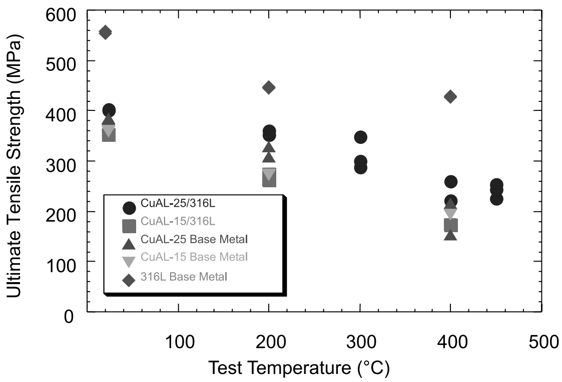


Fig. 6. Ultimate tensile strength of DSCu/316L joints and base metals.

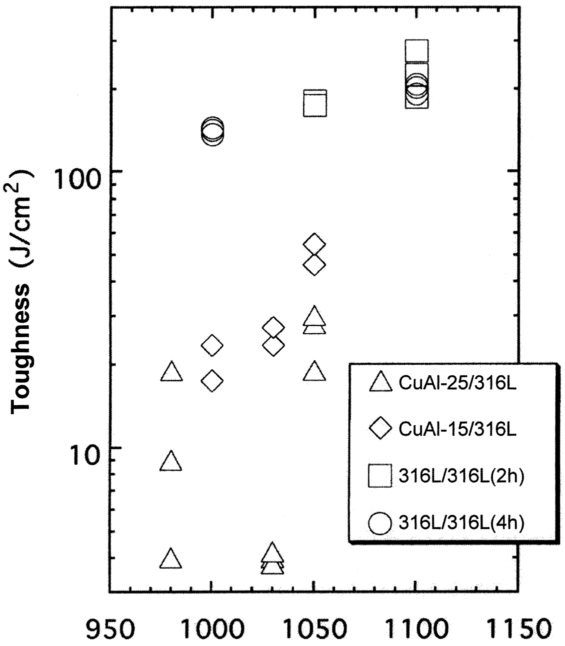


Fig. 7. Impact toughness of HIP bonded joints.

at 980°C or 1030°C, but it was lower by a factor of 2 in comparison with DSCu base metal [15,16]. Fracture tests have confirmed that cracks propagate along the HIP interface either within the intermediate layer formed by HIP process between DSCu and SS or in the interface between the intermediate layer and the DSCu region, 5–10 μm away from the original HIP interface.

Japanese investigators studied crack propagation behavior in a DSCu/SS joint in various orientations [10,15]. A crack, which initiated along the bonded interface propagated faster than a crack propagating

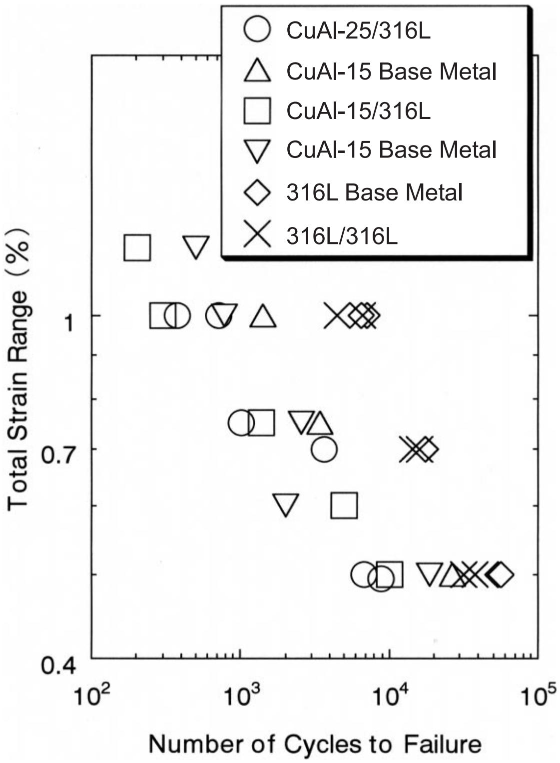


Fig. 8. Fatigue properties of HIP bonded joints and base metals at RT.

across the DSCu interface. The crack in the latter case stopped at the interface and then propagated along the DSCu/SS bonded interface.

Recent fracture toughness tests have been carried out in Europe at RT and 200°C on CT-SL specimens according to the standard ASTM 1737-96, both for the base DSCu alloy and the joints containing side-grooves. Results are given in Table 2.

For the fatigue pre-cracked CT specimens, despite the side grooves the pre-crack grows in the DSCu side at 80–150 μm from the joint, close to the zone enriched by precipitates. In the specimen pre-cracked by an oxide flaw, the fractography analysis shows that the crack immediately deviates in the DSCu side to reach the precipitate line (Cr, Fe) few microns from the interface and follows the weakest delaminated planes. Some small cracks appear perpendicular to the propagation direction and are stopped by the interface. In order to derive a rupture criterion, numerical simulations of the tests have been carried out. Results are focused on the DSCu side, where the rupture occurs. Values needed for damage analysis calculated at the moment corresponding to unstable crack propagation at the crack tip are listed in Table 3. The calculated J_q values are in good agreement with the experiment. At 20°C and 200°C, the local val-

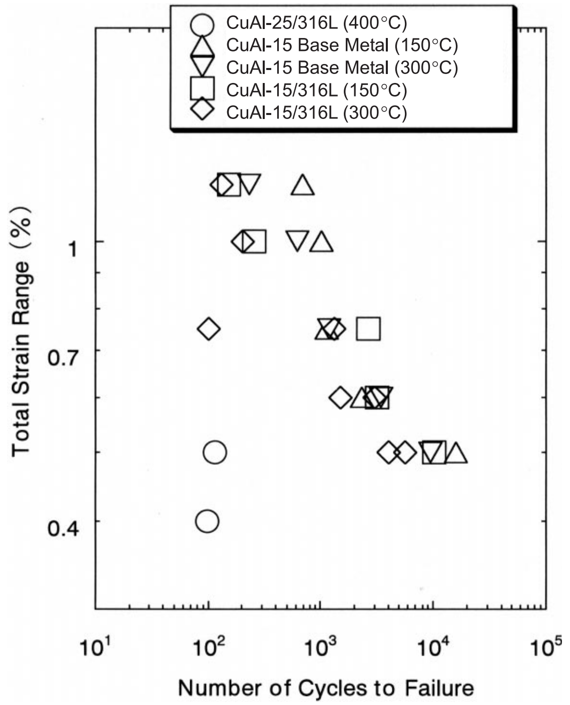


Fig. 9. Fatigue properties of HIP bonded joints and base metals at elevated temperature.

ues are always found lower in the bi-layer case. A clear understanding of these lower properties requires additional analysis.

During the growth of fatigue cracks in CuCrZr/SS joints [17], it was found that irrespective of where the notch tip was located, the fatigue crack developed in the Cu alloy only. All specimens failed in the alloy during further tests for three-point bending. This datum indicates that the joint's interface is more resistant to crack propagation than the base metal of the CuCrZr alloy.

4. Discussion

Experimental research, including microscopic observation and mechanical tests, have shown that DSCu/316LN joints have a defect-free interface, and the ultimate tensile and fatigue strengths are close to the respective characteristics of the Cu alloy base metal. Choice of HIP schedule for fabrication of multi-layer components (Cu/Cu/SS/SS) should be founded on the following points:

- an optimal HIP temperature of 1100°C for SS/SS joints,
- the melting temperature of ~1083°C for Cu alloys,
- an uncertainty of industrial HIP furnace temperature (usually ±10°C).

In this connection, to prevent overheating the copper parts of a component during the HIP process, a maximum temperature of 1040°C can be recommended. Very high accuracy of maintenance of the HIP temperature is required for large-size components, especially in the vertical direction of the HIP furnace.

Table 2
Fracture toughness of DSCu/316LN joints and DSCu alloy at 20°C and 200°C

HIP cycle	Temperature (°C)	Length (mm)		J_q (kJ/m^2)	$(dJ/da)_q$	Max load (N)
		Pre-crack	Final crack			
DSCu-IG1 (CT-SL)	20	24.37	26.06	30.7	181.6	14 200
		23.14	24.04	42.8	216.5	17 460
	200	23.37	25.35	12.3	63.5	11 200
		23.13	25.00	12.3	61.6	11 150
DSCu-IG1/316 (CT-SL)	20	23.05	26.41	7.8	15.8	8900
	200	22.75	35.85	4.9	17.1	8216
		22.46	22.76	5.5	18.9	9399
		22.43	25.12	5.1	13.3	7897
DSCu-IG1/316 (CT-SL), oxide flaw	20	18.36	19.37	8.9	16.0	13 325
		18.04	19.32	4.2	17.6	12 498
DSCu-IG1/316 (SENB[a])	20			12.2, 11		
	200			4.4, 4.2		
DSCu-IG0/316 (SENB[a])	20			7		
	200			3		

[a] Single-edge notch bending.

Table 3
Numerical results at F (J_q)

Specimen	T (°C)	J_q (kJ/m^2)		σ_{yy} [a] (MPa)	σ_I [b] (MPa)	ζ [c]
		Experimental	Numerical			
DSCu-IG1	20	30.7	32	1043	1060	2.5
DSCu-IG1/316LN	20	7.8	6.5	790	800	2.1
DSCu-IG1	200	12.3	11	725	730	2.4
DSCu-IG1/316LN	200	5.5	5	615	620	2.1

[a] Stress normal to the crack propagation.
[b] Maximum stress.
[c] Stress triaxiality.

The UTS is the determining characteristic of Cu/SS joints, as it is used in design and stress analysis. HIP provides joints with a UTS, that is not less than 0.9 UTS of the base metal for both DSCu and CuCrZr alloys. The characteristics of ductility rather indirectly reflect the quality of the bimetallic components and have no direct physical meaning.

The fracture toughness of DSCu/SS joints in the SL direction is still very low. CT specimens have been tested with an oxide flaw introduced to simulate crack propagation from a more relevant defect; no difference was found in comparison with fatigue pre-cracked CT specimens. Numerical analysis has been carried out to determine the main local parameters important for the damage analysis; the classical criterion applied to these bimetal specimens is not able to predict their rupture. The local values are always found to be lower in the bimetal case. A clear understanding of these lower properties requires more analysis.

To account for the metallurgical quality of Cu/SS joint manufacturing, it is necessary to specify the basic methods of tests and the examination criteria. The UTS of joints should be not less than 0.9–0.95 of the strength of the base metal of a copper alloy. Rupture location and the nature of the fracture surface provide an initial evaluation. Rupture in the Cu part of good Cu/SS specimens takes place near the joint interface in tensile tests. Specimens, which have failure along an interface and have a brittle fracture appearance, characterize poor quality HIP manufacturing of joints. Microscopic investigation could provide information on the presence of defects near an interface. Defects in the diffusion zone of Cu/SS joints are not allowed. In comparison with fracture toughness tests, the impact test is a rather simple and sensitive method for estimation of joint quality. The tests discussed above provide an acceptable means of evaluating the quality of Cu/SS joints made by HIPping.

5. Conclusions

HIP joining of copper alloys and stainless steel was developed and applied to fabrication of multi-layer components for fusion reactors. Analysis of available experimental data has demonstrated that Cu/SS joints that have a defect-free interface have rather high tensile and fatigue strengths but low impact and fracture toughness.

The following criteria for the estimation of the metallurgical quality for Cu/SS joints can be used: presence of defects in an interface, value of the UTS and the level of impact toughness.

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