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Be–Cu joints based on amorphous alloy brazing for divertor and first wall application

B. Kalin [a,*], V. Fedotov [a], O. Sevryukov [a], A. Plyuschev [a], I. Mazul [b], A. Gervash [b], R. Giniatulin [b]

[a] *Moscow State Engineering Physics Institute, Technical University, Kashirskoe sh. 31, Moscow 115409, Russian Federation*
[b] *D.V. Efremov Institute of Electrophysical Apparatus, St. Petersburg 189631, Russian Federation*

Abstract

The limitation on using silver-based alloys for brazing ITER in-vessel components created a development problem for new brazing materials. An application of Be tiles joined to a Cu-based heat sink will be discussed in this paper. For this purpose, rapidly solidified ribbon-type filler metal STEMET 1108 [1] (Cu–Sn–In–Ni system) with a melting temperature of 750°C and a thickness up to 50 μm was developed. A new temperature regime and heating method were applied during this brazing procedure. This improves the properties of joints significantly, in comparison with conventional brazing in a resistance furnace. These treatments can increase the operational threshold for Be/Cu joints under cyclic surface heat loading up to 12 MW/m^2. Metallographic examinations demonstrated the high quality of brazed joints. The brazed seam has a uniform structure along its entire length. Defects of seam filling, pores, intermetallic compounds and other inclusions are not seen. The prospects of these joints for fusion reactor applications are discussed.

1. Introduction

Brazing is a process used to join materials by introducing an interfacing metal that has a melting point higher than 450°C, but lower than the melting point of materials to be brazed [1]. Brazing is the most-used joining technique of ITER materials (CFC-composites, metal-ceramic assembly, SiC-composites, dispersion-strengthened alloys, beryllium, refractory metals etc.). Most analyzed references are devoted to joining of beryllium: Be–Cu [2–7,17–21], Be–Be [2,8], Be–SS [8]; and to joining of copper: Cu–CFC [9–11], Cu–Cu [7,12,13], Cu–SS [14]. Sometimes, diffusion barriers are used in a brazing process to eliminate the formation of intermetallic compounds between the filler metal (FM) and the base materials and a spacer to regulate differences in thermal expansion coefficients. The general requirements for FMs in brazing of ITER materials are as follows:

- ability to wet the substrate materials;
- compatibility with base materials and resistance to operating conditions;
- providing reliable mechanical and heat contact;
- ability to alloy or react with base materials without forming brittle intermetallic compounds;
- good flow characteristics at the brazing temperature.

In selecting FM, it is necessary to minimize the high temperature brazing cycle; FM must have narrow melting and solidification ranges. Analysis of references on the brazing technique for ITER materials has shown that FMs used are: Ag-alloys, Cu-alloys, Ni-alloys, elements (Al, Mg, Ti, Si). The exeption is investigations of the feasibility of rapidly quenched amorphous FM for ITER materials [6,7,15]. New FMs in the form of ductile thin foil may be of interest. These foils are manufactured by rapid solidification and they posses some useful properties, for example:

- extremely high chemical and phase homogeneity;
- higher diffusion [16] and capillary [1,17] activity than crystalline analogues;

* Corresponding author. Fax: +7-095 324 3165; e-mail: kalin@ameto.mephi.ru

[1] STEMET® is a registered trade name of MIFI-AMETO, Ltd. It is a company which specialises in producing of ribbon-type brazing filler metals by rapid solidification.

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- narrow melting and solidification ranges;
- "instantaneous" melting across the whole width and thickness of foil [1];
- outstanding ductility: amorphous foils bend 180° without fracture to comply with complex joint geometries for easy fixturing. They are ductile enough to be mechanically formed and shaped to three-dimensional configurations.

Wetting may be increased by doping of amorphous FMs with active elements: Mn, Ti, Zr, Nb etc. By now some experience has been gained in the application of rapidly quenched FMs STEMET for joining of dissimilar PFC-materials: Cu-graphite, Mo-graphite, V-graphite, Be–Cu, V–Be, SS–Be [2,6,7,15,17].

The use of thin brazing ribbons allows saving FMs and attaining acceptable appearance of the brazed joint. For example, the interaction zone of FM STEMET 1108 with beryllium was demonstrated to have a width of no more than 3 μm. This zone was free of large intermetallic grains and cracks. The tensile strength of Cu/Be/Cu brazed joints made by STEMET 1108 was 125 ± 12 MPa and the failure of specimens took place in beryllium close to the brazed seam [15].

This paper discusses the problems connected with brazing of Be with CuCrZr copper alloy using the new ribbon-type rapidly solidified Cu–Sn–In–Ni FM (STEMET 1108) in combination with a fast heating at brazing.

2. Rapidly solidified FM

Based on the results of previous investigations [15] the work on improvement of rapid solidification process of STEMET 1108 was continued. The main focus was directed towards production of continuous homogeneous ribbon with controlled thickness (50 μm) and minimal thickness deviation (up to 3 μm). For this goal, the following techniques were used: (i) the micro-doping of alloy with some elements to strength and to improve casting properties; (ii) an enhanced method of smelting ingots for rapid solidification process; (iii) specially developed regimes for time–heat treatment of melting before rapid solidification process; (iv) a precision control system of dynamic parameters of rapid solidification process. The characteristics of rapidly solidified ribbon and cast alloy of STEMET 1108 composition are shown in Table 1. The same FM presented itself in a good light for brazing of Cu with SS [16]. This fact has demonstrated a possibility of manufacturing of the Be–Cu–SS three-metal structure during one brazing cycle.

It is necessary to point to the traditional brazing technique of ITER components, which would change by increasing heat and cool rates of brazing assembly. It will be useful for preservation of base material properties (for example, dispersion-strength Cu-alloys).

3. Brazing method selection

Metallographic analysis of all previous Be/Cu samples tested [21] clearly revealed a very thick (~100 μm) brittle intermetallic layer in the bonding seam. We believe that one of the main reasons for the relatively low number of thermal cycles before failure during the preceding testing campaigns is the formation of a thick intermetallic phases. It was shown [21] that all previous joining technologies considered use significantly long brazing (heating–soaking–cooling) cycle. Such a long cycles take place because of using the traditional 'slow' resistor furnace. At the same time, the best results of thermal cycle tests were achieved when the fast heating obtained by an induction furnace was used for brazing [20]. This is the reason why we decided to use the TSEFEY's electron-beam heating for the fast brazing procedure.

To provide an ITER relevant cyclic thermal load, the TSEFEY electron-beam facility (St. Petersburg, Russian Federation) was developed. This facility is capable of creating cyclic heat fluxes with the desired parameters of cycle time and energy density. The cycle time can be varied in the range from 0.1 s to steady state. The energy

Table 1
The properties of STEMET 1108 rapidly solidified ribbon-type filler metal and conventionally cast alloy of STEMET 1108 composition

Property	STEMET 1108 (rapidly solidified ribbon)	Conventionally cast alloy of STEMET 1108 composition
Chemical composition (wt%)	Cu(bal.)-12Sn-9In-2Ni-1.5(Mn, Cr, P, Si, Fe)	
Solidus temperature (°C)	750	760
Liquidus temperature (°C)	800	820
Dimensions of ribbon		Bars
Thickness (μm)	50 ± 2	
Width (mm)	10 ± 1, 20 ± 1	
Length (m)	Up to 100	
Ductility	Bends 180°C without fracture	Fails easily in bending

density can be varied in the range from 0.1 to 15 MW/m^2. We refer to the literature [20] for detailed description and features of the TSEFEY facility, but even from the above mentioned parameters it is easily recognized that the facility is suitable for fast-heating brazing. A unique brazing process was developed, which uses advanced STEMET 1108 in combination with a fast heating provided by TSEFEY's electron-beam.

The chemical compositions of Be (TGP-56) and Cu-alloy (CuCrZr) used are shown in Table 2. The main physical and mechanical properties of the base alloys are shown in Table 3. The properties of CuCrZr are strongly dependent on previous heat treatment. In our case, the heat treatment was as follows:

- solution annealing (980°C, 1 h) + water quench;
- cold working (~40%);
- aging (470°C, 4 h);

The following two mock-ups were fabricated using the STEMET 1108 brazing FM.

1. The mock-up for moderate heat loaded PFC – two $44 \times 44 \times 10$ mm^3 Be tiles were brazed to CuCrZr heat sink block.
2. The mock-up for high loaded PFC – 32 tiles of dimensions $5 \times 5 \times 5$ mm^3 were brazed to CuCrZr heat sink blocks.

Before brazing, the joining surfaces of Be and Cu-alloy were machined and etched. Beryllium tiles were fixed on the CuCrZr surfaces using light steel clamps. One layer (~50 μm thick) of a previously etched STEMET 1108 FM was laid between Be and CuCrZr. These light steel clamps were used mainly to fix the Be tiles and brazing the FM on the CuCrZr surface. The brazing pressure is estimated to be in the order of 0.1 MPa. Then assemblies were installed into the TSEFEY facility and heated by an electron-beam up to 780°C. The temperature of the brazing zone was controlled with a thermocouple. The heating–cooling down curve for the brazing cycle is presented in Fig. 1. The heating rate during the brazing cycle was ~160–180°C/min and the cool-down rate ~20°C/min. It is seen that the brazing time is significantly less in comparison with the brazing time in resistor furnace.

Table 2
The chemical compositions of the base alloys

Base metals	Chemical composition (wt%)
CuCrZr	Cu – base
	Cr – 0.6
	Zr – 0.08
	Total others – 0.1 max
TGP-56 beryllium	Be(min) – 97.8
	BeO(max) – 2.5
	Al(max) – 0.03
	C – 0.12
	Fe – 0.1–0.2
	Si – 0.04
	Ti – 0.05
	Cr – 0.08
	F – 0.005
	Others – 0.08

Table 3
The main physical and mechanical properties of the base alloys

Property	CuCrZr	TGP-56 beryllium
Electrical conductivity	45 m/Ω mm^2	–
Thermal conductivity	320 W/m K	180 W/m K
Young's modulus	130 GPa	297 GPa
Expansion coefficient	17.6×10^{-6}/K	11×10^{-6}/K
Density	8.92 g/cm^3	1.85 g/cm^3
Ult. tensile strength	480 MPa	380 MPa
0.2% Yield strength	350 MPa	360 MPa
Total elongation	18%	1.5%
Hardness	140 HB	–

4. Brazing zone microstructure

The microstructure of the brazed joint was investigated by optical microscopy. In the first stage of investigation, the Be/CuCrZr joint sample (Sample 1) brazed in a traditional resistor furnace was compared with the Be/CuCrZr sample (Sample 2) brazed by fast electron-beam heating in TSEFEY. The photos of the brazed seams of samples are shown in Figs. 2 and 3.

One can see at least two main differences.

1. The width of the brazing seam for Sample 1, which was brazed in the resistor furnace is ~420 μm but is only 20–30 μm for Sample 2, which was brazed by fast electron-beam heating. This difference can be attributed to the difference in the heating period in the brazing processes for Sample 1.
2. A thin (~10 mm) layer with a number of pores in Be near the pure Be/brazing FM boundary was formed in Sample 1 but no pores were seen in Sample 2. This can be explained by the fact that a high rate of diffusion migration of Be into the brazing FM has caused an elevated temperature and a long time of brazing.

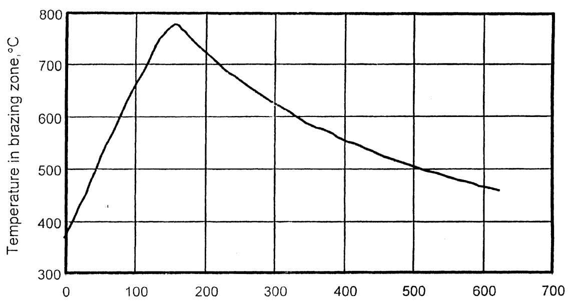

Fig. 1. Time–temperature brazing cycle in TSEFEY electron-beam facility.

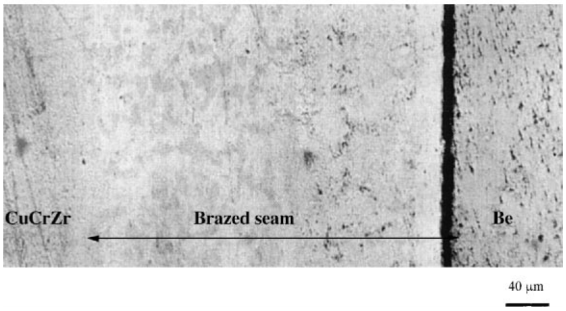

Fig. 2. Brazed joint cross-section for the joint produced by brazing in resistor furnace (Sample 1).

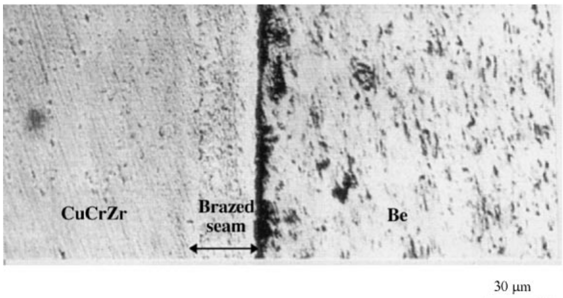

Fig. 3. Brazed joint cross-section for the joint produced by fast electron-beam heating (Sample 2).

The significantly low shear strength of Sample 1 (22 MPa) is due to cracking of the brazed joint through Be close to the brazing boundary. This can be considered as a confirmation of the presence of pores in Be resulting from the Kirkandall diffusion effect. At the same time, the shear strength of Sample 2 produced by fast electron-beam heating was found to be 137 MPa.

To examine the brazing technique, HHF tests were carried out with both mock-ups at the TSEFEY electron beam facility. The main purpose of the test was to check the reliability of Be/Cu joints under cyclic heat loading. The tests were performed under the conditions: for the first mock-up 2000 and 3000 cycles (15/15 – load/pause time per cycle) were applied at the heat flux of 2 and 1.5 MW/m^2, respectively. For the second mock-up 4500 cycles (10/10 s) were performed at the heat flux of 12 MW/m^2. The brazed seam cross-section of Sample 2 after the thermal cycling (3000 cycles, 1.5 MW/m^2) is presented in Fig. 4. In compare with the initial state (Sample 2) the width of brazing seam has been insignificantly increased up to $\sim$40 μm but pores or cracks were not found. During thermocyclic tests, no changes of surface and bulk temperatures were detected from cycle to cycle for these mock-ups, as it was demonstrated earlier for a heat flux of 8 MW/m^2 [22]. After the tests,

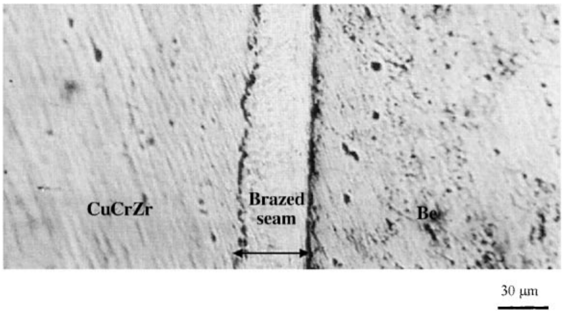

Fig. 4. Brazed joint cross-section after the thermal cyclic test for the joint produced by fast electron-beam heating.

no visible damage was observed in the Be/Cu joints of both mock-ups.

The performed HHF tests with Be-armored mock-ups have demonstrated the acceptance of the current brazing technology for PFCs, and can work under a wide range of heat loads.

5. Conclusions

The main conclusions of this work are:

1. The thick layer of brittle intermetallic compounds formed after Be/Cu joining using 'slow' heating in a resistor furnace is one of the main reasons of failure under cyclic thermal loading.
2. At the expense of reduction of a melting interval and small thickness, the STEMET 1108 rapidly solidified brazing FM permits carrying out the brazing process by fast heating without defects of seam filling and other defects of brazed joints.
3. The combination of fast brazing techniques (like TSEFEY used) with microcrystalline type of brazing alloys reduces significantly the width of the intermetallic zone.
4. Mechanical properties (shear strength) of Be/Cu joints produced by fast heating are significantly higher than heating in a resistor furnace.
5. Fast heating can avoid full annealing of the Cu alloy (in case of using CuCrZr).

Recent thermal fatigue results obtained on the TSEFEY facility are evidence of the above mentioned logic.

References

[1] N.J. De Christofaro, A. Datta, Rapidly Quenched Metals 1985, pp. 1715–1721.
[2] B.C. Odegard, B.A. Kalin, J. Nucl. Mater. 233–237 (1996) 44.
[3] T.M. Gitoeva, V.T. Pronyakin, J. Nucl. Mater. 233–237 (1996) 918.

[4] M. Araki, D.L. Youchison, M. Akiba et al., J. Nucl. Mater. 233–237 (1996) 632.
[5] A.A. Gervash, R.N. Giniatouline, I.V. Mazul et al., J. Nucl. Mater. 233–237 (1996) 626.
[6] B.A. Kalin, V.T. Fedotov, A.E. Grygoriev et al., Fusion Eng. Design 28 (1995) 119.
[7] B.A. Kalin, V.T. Fedotov, O.N. Sevryukov et al., J. Nucl. Mater. 233–237 (1996) 945.
[8] V.R. Barabash, L.S. Gitarsky, G.S. Ignatovskaya et al., J. Nucl. Mater. 212–215 (1994) 1604.
[9] G. Chaumat, P. Le Gallo, G. Le Marois et al., J. Nucl. Mater. 233–237 (1996) 932.
[10] P.W. Trester, P.G. Valentine, W.R. Johnson et al., J. Nucl. Mater. 233–237 (1996) 906.
[11] R.E. Nygren, C.A. Walker, T.J. Lutz et al., J. Nucl. Mater. 212–215 (1994) 1621.
[12] F. Saint-Antonin, M. Suery, P. Meneses et al., J. Nucl. Mater. 233–237 (1996) 935.
[13] S. Chen, J.Y. Liu, B.A. Chin, J. Nucl. Mater. 212–215 (1994) 1600.
[14] S. Chen, T. Bao, B.A. Chin, J. Nucl. Mater. 233–237 (1996) 902.
[15] B.A. Kalin, V.T. Fedotov, O.N. Sevryukov et al., Plasma Devices and Operations 4 (1996) 245.
[16] B.A. Kalin, V.T. Fedotov, O.N. Sevryukov et al., Svarochnoe Proizvodstvo 1 (1996) 15 (in Russian).
[17] Yu. G. Prokofiev, V.R. Barabash, V.F. Khorunov et al., J. Nucl. Mater. 191–194 (1992) 483.
[18] C.K. Lee, B.A. Chin, S. Zinkle et al., J. Nucl. Mater. 191–194 (1992) 488.
[19] E. Franconi, G.S. Ceccotti, L. Magnoni, J. Nucl. Mater. 191–194 (1992) 493.
[20] V. Gagen-Torn, I. Kirillov, V. Komarov et al., Proc. 18 SOFT, Karlsruhe, 1994, pp. 363–366.
[21] A. Gervash, R. Giniatouline, I. Mazul et al., in: Proceedings of the Second IEA International Workshop on Beryllium Technology for Fusion, Wyoming, USA, 1995, pp. 364–380.
[22] R. Giniatulin, I. Mazul, V. Komarov et al., Proc. ISFNT-4, Tokyo, Japan, 1997.