Investigation of a brazed joint EK-181/V/W obtained by Cu-Sn and Cu-Ti amorphous foils

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Abstract. The work presents the results of high temperature brazing of reduced activated ferritic martensitic steel EK-181 with pure tungsten, which is essential for DEMO fusion reactor. Vanadium interlayer was used to reduce thermal stresses. Brazing alloys to be used were rapidly quenched into ribbons Cu-12Sn, Cu-20Sn, Cu-12Sn-0.4P for EK-181/V, Cu-50Ti for V/W. Microstructure investigations, mechanical and thermocycling test were carried out.

1. Introduction
The DEMO thermonuclear reactor is the next step towards the creation of an energy thermonuclear reactor; its conceptual projects are currently being developed by all the leading countries of the world. DEMO construction is possible in the 30s of the current centuries, depending on the success of the ITER program. In recent years, there has been intensive development of projects for demonstration and energy fusion reactors: DEMO-C in Russia, ARIES in the USA, SEAEP and PPCS in Europe, SSTR in Japan, etc. [1–4]. Each project is individual in its own way and assumes the presence of the first wall, which is a stressed structural element.

A reduced activation ferritic-martensitic steel (EK-181 [5], Eurofer [6], F82H [7]) is used as a structural material, and tungsten is used as a plasma facing material [8]. Numerous problems limit the direct connection of tungsten and steel due to significant differences in their physical properties, in particular, various coefficients of coefficient of thermal expansion: (10.5-12.3) * 10⁻⁶ K⁻¹ in steel and (4.3-6, 0) * 10⁻⁶ K⁻¹ at tungsten. This difference causes large residual stresses during cooling, and as a result leads to the destruction of the connection [9,10]. Therefore, firstly, it is necessary to choose the right technology for joining these materials, in this case, the use of brazing technology will ensure the formation of high-quality compounds. Secondly, the use of a buffer layer of pure vanadium will compensate for the differences between steel and tungsten [11,12].

First of all, filler alloys for brazing must meet the requirement of reduced activation. Another important limitation when while joining is the maximum process temperature - it should not exceed 1100°C, because in this case, an active grain growth occurs in a ferritic-martensitic steel.

It follows from the foregoing that the problem of joining dissimilar materials of the DEMO thermonuclear reactor and, in particular, the buffer layer of vanadium alloy and ferritic-martensitic steel is really relevant and has not yet been resolved.
2. Materials and Experimental Methods

In this work the following materials were used:

- Reduced-activated steel of ferritic-martensitic grade EK-181 (Fe – 12Cr – 2W – V – Ta – B, wt.%).
- In the annealed state, CTE = (10.5-12.3) * 10^-6 K^-1. Dimensions for mechanical tests: 20x7x7 mm, size for thermocyclic tests: cylinder D = 13 mm, h = 6 mm;
- Tungsten 99.96% purity, CTE = (4.3-6.0) * 10^-6 K^-1. Dimensions for mechanical tests: 1 x 7 x 7 mm; size for thermocyclic tests D = 10 mm, h = 1 mm;
- Pure vanadium, CTE = (8.3-10.9) * 10^-6 K^-1. Dimensions for mechanical tests 10x10x0.2 mm; size for thermocyclic tests h = 0.2 μm in the form of a square with a side of ≈10 mm.

Rapidly quenched into ribbon filler alloys STEMET™ were used: Cu-12Sn, Cu-12Sn-0.4P, Cu-20 Sn, Cu-50 Ti, wt.% (the melting temperature: 900 °C, <900 °C, 755 °C, 955-980 °C respectively). STEMET™ brazing alloys are used for production of ITER components [13, 14].

In this work, we obtained compounds EK-181 / W (with the presence of spacers from V). In all cases, the V / W joint was obtained using Cu-50Ti brazed, and for EK-181 / V – Cu-Sn. Brazing was carried out in a vacuum furnace at a vacuum not lower than 10^-3 mm Hg. Comparative analysis of the compounds was carried out on samples that were brazed with the mode №1 1100 °C, 20 min. A metallographic analysis was carried out, thermocyclic and shear tests were performed for samples:

- EK-181 / Cu-12Sn / V / Cu-50Ti / W,
- EK-181 / Cu-12Sn-0.4P / V / Cu-50 Ti / W,
- EK-181 / Cu-20 Sn / V / Cu-50 Ti / W.

In order to combine the process of brazing and heat treatment of the steel mode №2 was applied 1100 °C, 60 min (to analyze the influence of the holding time during brazing on the microstructure) and mode №3 1100 °C, 60 min (cooling at a rate of 20 °C/min) + 720 °C 180 min, the latter corresponds to the traditional heat treatment of steel EK-181 [15].

The microstructure was investigated on cross sections of brazed cylindrical samples. The microstructure was investigated on an MTI MM500T metallographic microscope, a JEOL JSM-6610LV scanning electron microscope. To identify the microstructure of steel for metallographic studies, a solution was used: 20% HNO₃ + 20% HF + 60% glycerol, etching time 20–40 s.

For thermocyclic tests the samples were placed into a quartz ampoule with a gettier from iodide zirconium and evacuated to 10⁻² mm Hg. Test mode: heating in a tube furnace with resistive heating up to 700 °C followed by quenching in water (30 and 50 cycles). The mode was selected taking into account the operating conditions of the tungsten / steel joint in the “finger” of the HEMJ divertor [16], however, this concept has recently lost relevance, therefore, the task of connecting steel to tungsten is primarily to create the first wall, the operation mode of which is much softer [17]. However, we applied this mode to provide degradation of the joints and proceed fast comparison of different filler alloys as it often used.

We studied the distribution of microhardness HV₀.₁ (load 100 g, holding time 15 s) in the cross section of a brazed seam on a Vickers FM-800 (Future-Tech) microhardness tester. The mechanical shear tests were carried out on an Instron 5569 installation (crosshead speed 1 mm / min) of the samples. Tests were carried out on samples before and after 50 thermal cycles. The test design is presented elsewhere [12].

3. Results and Discussion

The vigorous investigation of V/W microstructure was presented earlier [12]. The microstructure of brazed joints EK-181/V obtained using Cu-Sn filler alloy (Figure 1) consists of the following areas: 1 - zone of ferritic-martensitic steel; 2 - ferrite zone, divided into two areas: a - ferrite formed as a result of carbon depletion, b - ferrite formed as a result of crystallization; 3 - σ (Fe-V); 4 - VC; 5 - pure vanadium. The same microstructures were formed while using Cu-Ge alloys [12]. In the brazed joint EK-181 // Cu-12Sn-0.4P // V (“12Sn + P”), a phosphide is also supposedly formed, near which microcracks form (highlighted by an orange rectangle in Figure 1).
Both in the brazed joint “12Sn” and “12Sn + P”, the microhardness changes in a similar way: zone 1 $\approx 400$ HV0.1; zone 2 $\approx 200$ HV0.1; zones 3 + 4 $\approx 600$ HV0.1.

A distinctive feature of the “20Sn” brazed joint is the absence of $\sigma$ (Fe-V) in the predominant volume of the joint. This phase can only be observed near fillets, which leads to lower microhardness $\approx 400$ HV0.1, in comparison with samples in which the sigma phase is detected [12]. When using “12Sn + P” filler alloy, we observe more copper precipitations along grain boundaries.

**Figure 1.** The microstructure of the cross-section of the joint EK-181 / V obtained by mode 1 using filler alloy a) Cu-12Sn; b) Cu-20Sn; c) Cu-12Sn-0.4P.

According to the results of mechanical tests for shear before thermocyclic tests, the tensile strength of the joints is EK-181 / Cu-12Sn / V / Cu-50Ti / W (“12Sn”), EK-181 / Cu-12Sn-0.4P / / V / Cu-50Ti / W (“12Sn + P”), EK-181 / Cu-20Sn / V / Cu-50Ti / W (“20Sn”) is 140 MPa, 84 MPa, 160 MPa, respectively (mode 1). The 12Sn and 20Sn compounds are destroyed on the tungsten side (Figure 2 a, b), the 12Sn + P compound (Figure 2 c) is mainly destroyed on the steel side, which is probably associated with the formation of the phosphide phase.

There were no changes in the structure of the brazed of these compounds after 30 thermal cycles (Figure 3 a, c; there is no image for “12Sn + P”). After 50 cycles a structural change in copper precipitates is observed near the fillets which can also indicate a phase change due to the diffusion redistribution of components during heating. In addition, as in the case of compounds obtained using Cu-Ge filler alloy, discontinuities and delamination are formed near the fillets. There is a change in structure from the side of tungsten (Figure 3 b, g). Mechanical tests after 50 cycles give the following result: “12Sn” - 35 MPa, “12Sn + P” - 28 MPa, “20Sn” - 46 MPa.
Figure 2. Samples after mechanical tests before thermal cycling:
a) EK-181 / Cu-12Sn / V / Cu-50Ti / W; b) EK-181 / Cu-20Sn / V / Cu-50Ti / W;
c) EK-181 / Cu-12Sn-0,4P / V / Cu-50Ti / W.

Figure 3. The microstructure of the cross section of the seam of brazed joints:
EK-181/Cu-12Sn / V/ Cu-50Ti / W a– after 30 thermal cycles; b– after 50 thermal cycles; 
EK-181 / Cu-20Sn / V / Cu-50Ti / W c– after 30 thermal cycles; g - after 50 thermal cycles.

Based on the described results due to the higher value of shear strength as well as a reduced amount of 
sigma phase, Cu-20Sn filler alloy was considered for further studies.

To combine the process of brazing and heat treatment of steel an analysis was made of the 
dependence of the microstructure on the temperature-time regime as well as a change in mechanical 
properties during the transition to the heat treatment of steel. Comparing the microstructure of the
brazed EK-181 / V (figure 4) of the samples obtained in modes 2, 3 with the sample (mode 1), zones with and without sigma phase are observed. No grain growth of ferritic zinc nor growth of other phases were observed.

![Figure 4](image)

**Figure 4.** Brazed seam EK-181 / V obtained using Cu-20Sn according to the modes:

a) mode 2; b) mode 3.

Final tensile strength of the brazed joint obtained by mode 3 - 93 ± 24 MPa. The destruction of the samples occurs on the side of tungsten.

Figure 5 presents the summary results of the mechanical tests carried out in the work. It is noted that thermocyclic tests lead to a decrease in strength by more than a factor of two, which is associated with delamination and chipping of the phases on the V / W side. The heat treatment of steel leads to a slight decrease in strength, However, the achieved values are higher than in some works [18,19], as well as while using Cu-Ge alloys [10,12].

![Figure 5](image)

**Figure 5.** Results of mechanical shear tests
4. Conclusion

High-temperature brazing of the EK-181 / V / W joint was carried out with rapidly quenched into ribbon filler alloys: Cu-50Ti for V / W and Cu-12Sn, Cu-20Sn, Cu-12Sn-0.4P for EK-181 / V in three modes:

№ 1. 1100 °C, 20 min (for comparative analysis);
№ 2. 1100 °C, 60 min (to analyze the effect of the holding time during brazing on the microstructure);
№ 3. 1100 °C, 60 min + 720 °C 180 min (corresponds to the heat treatment mode EK-181).

The brazed seam EK-181 / V consists of a zone of ferritic-martensitic steel, a zone of ferrite, divided into two areas: a - ferrite formed as a result of carbon depletion, b - ferrite formed as a result of crystallization, vanadium carbide, σ(Fe-V), pure vanadium. However, the amount of σ (Fe-V) in the brazed joint obtained by Cu-20Sn is much lower compared to other brazing alloys. The absence of this phase results in a higher shear strength of the joint.

It was established that an increase in the holding time during brazing doesn’t lead to the microstructural changes. Subsequent annealing at 720 °C for 180 min also does not affect.

Mechanical shear tests were carried out for brazed joints obtained according to mode 1 (before thermocyclic and after 50 thermal cycles of 700 °C - quenching in water). Also shear strength was investigated for as-joined “20Sn” obtained by mode 3 (before thermocyclic tests).

The strength of the joint EK-181 / Cu-20Sn / V / Cu-50Ti / W is:
Mode 1 (0 thermal cycles) – 160 ± 38 MPa;
Mode 1 (50 thermal cycles) – 46 ± 6 MPa;
Mode 3 (0 thermal cycles) – 93 ± 24 MPa.

The strength of the joint EK-181 / Cu-12Sn / V / Cu-50Ti / W is:
Mode 1 (0 thermal cycles) – 140 ± 32 MPa;
Mode 1 (50 thermal cycles) – 35 ± 3 MPa;

The strength of the joint EK-181 / Cu-12Sn-0.4P / V / Cu-50Ti / W is:
Mode 1 (0 thermal cycles) – 84 ± 9 MPa;
Mode 1 (50 thermal cycles) – 28 ± 8 MPa.

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References

[1] T R Barrett et al 2016 Fusion Eng. Des. 109111 917
[2] M I Subbotin, K D K, and F E A 2010 Quest. Nucl. Sci. Technol. 3 55 (In Russian)
[3] K Tobita et al 2018 Fusion Eng. Des. 136 1024
[4] G Federici et al 2018 Fusion Eng. Des. 136 729
[5] V M Chernov, M V. Leont’e-smirnova, E M Mozhanov, N S Nikolaeva, A N Tyumentsev, N A Polekhina, I Y Litovchenko and E G Astafurova 2016 Tech. Phys. 61 209
[6] A A F Tavassoli et al 2004 Materials design data for reduced activation martensitic steel type EUROFER (North-Holland) p 257
[7] T Hirose, T Kato, H Sakasegawa, H Tanigawa and T Nozawa 2020 Fusion Eng. Des. 160 111823
[8] V Philippis 2011 J. Nucl. Mater. 415 S2
[9] W W Basuki and J Aktaa 2011 J. Nucl. Mater. 417 524
[10] D Bachurina, A Suchkov, B Kalin, O Svirikov, I Fedotov, P Dzhumaev, A Ivannikov, M Leont’e-smirnova and E Mozhanov 2018 Nucl. Mater. Energy 15 135
[11] W W Basuki and J Aktaa 2012 J. Nucl. Mater. 429 335
[12] D Bachurina, A Suchkov, A Filimonov, I Fedotov, M Savelyev, O Sevryukov and B Kalin 2019 Fusion Eng. Des. 146 1343
[13] B A Kalin, A N Suchkov, V T Fedotov, O N Sevryukov, A A Ivanniko and A A Gervash 2016 *Nucl. Mater. Energy* **9** 388

[14] I V. Mazul, V A Belyakov, A A Gervash, R N Giniyatulin, T Guryeva, V E Kuznetsov, A N Makhankov, A A Okunev and O N Sevryukov 2016 *Fusion Eng. Des.* **109–111** 1028

[15] A G Ioltukhovskii, M V Leont’eva-Smirnova, V M Chernov, V V Tsvelev, M I Solonin, V N Golovanov and V K Shamardin 2002 *Termicheskaya Obrab. Met.* **44** 11

[16] P Norajitra, S Antusch, R Giniyatulin, V Kuznetsov, I Mazul, H J Ritzhaupt-Kleissl and L Spatafora 2011 *Fusion Eng. Des.* **86** 1656

[17] Y Huang, F Cismondi, E Diegele, G Federici, A Del, F Moro and N Ghoniem 2018 *Fusion Eng. Des.* **135** 31

[18] J C Wang, J Huang, H Sun, X Gao, W Wang, Q Li, C Xie, X Wang, Z Chen, Q Gao, S Liu, G N Luo and J Li 2019 *Fusion Eng. Des.* **138** 313

[19] J Wang, W Wang, R Wei, X Wang, Z Sun, C Xie, Q Li and G Luo 2017 *J. Nucl. Mater.* **485** 8