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Thermophysical and mechanical properties of W-Cu laminates produced by FAST joining

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W-laminates are multi layered composites realized from alternately stacked W and a second metal foils. Such materials are promising candidates for W-based structural materials for fusion reactors like DEMO or beyond concepts, due to the fact that cold-rolled ultrafine-grained thin W foils show exceptional properties in terms of ductility, toughness and ductile to brittle transition (DBT), in contrast to classic bulk W materials. Therefore, different routes to transfer the W foils properties to bulk materials have been investigated. In this work we present the results obtained for W-Cu laminates produced via a FAST (Field Assisted Sintering Technique) joining route. The main advantages of FAST resides in the short processing time, with subsequent lower recrystallization detrimental effects. Structural, thermophysical and mechanical properties of W-Cu laminate samples produced in this way are investigated and compared with those available for diffusion bonded similar samples.

Keywords: Field assisted sintering technology; W-laminates; mechanical properties; thermo-physical properties.

1. Introduction

W has the highest melting point of all metals, good high temperature strength, high creep resistance and a high thermal conductivity. These properties made W a first choice for armor materials in fusion energy reactors. But W has also low values for tensile ductility, toughness K_{IC} coefficient (crack growth resistance), and thermal expansion coefficient and also a high value of brittle to ductile transition temperature (DBTT). The later can be however decreased by a finer (under micrometer) grain size structure [1]. A large amount of work was devoted to understand and improve W ductility, by alloying with various elements like e.g. Re [2-4], or by creating a ultra-fine grain structure by severe plastic deformation [1, 5] or mechanical alloying [6, 7]. Recently, it was shown that W foils are ductile even at room temperature [8] and this particular and paradoxical feature of W can be used to develop possible new Tungsten based structural materials [9-11].

Attempts to transfer these properties from W foils to W-based bulk materials resulted in the so-called "W-laminates" concept, i.e. multi layered composites from alternate W and other metal foils. Different approaches were already successfully used to create such composites, joining the foils by brazing (e.g. with Cu or with Cu-Ag based brazing alloys) or by diffusion bonding using Ti and Zr interlayers [12]. Following these routes large components like pipes have been produced [13]. Several shortcomings were observed at high temperature exposure [14] or during neutron irradiation [15]. Possible solutions to improve the W irradiation resistance of W have been considered [16-18] and further improvements might be realized by using improved K-doped W foils [19]. A recent comprehensive review on the W-laminates behavior and application potential is performed in reference [20].

In this context, our work was focused in producing W laminates by FAST (field assisted sintering technique). In general, the obvious advantage of this route resides in the short processing time (only a few minutes at temperatures higher than 1000 °C, even using high melting point metals like Ti, V, Zr or Mo), resulting in lower recrystallization detrimental effects. Meanwhile it is possible to reach temperatures close to the metal melting points and thus making the joining process similar to brazing. FAST implies sending trains of short (a few ms) and intense (a few kA) electrical dc current pulses through the sample (in this case the alternatively stacked W and the other metal foils). Thus, since the entire sample is metallic, the main heating arises through Joule effect. On the other side, at the interfaces between foils, due to imperfect electrical contacts, electrical discharges produce effects similar to electrical point welding, however on the entire surface at once. However, as the interface tends to improve due to the heating and discharge stimulated mass transport, a diffusion bonding process also starts.

Due to Joule effect, the metal with a higher electrical resistivity is heated more than the better electrical conductor metal. In the case of W-Cu laminates, W is more heated than Cu and at the W-Cu interface the heat flows from W toward Cu. With the possible additional effect of electrical discharges, Cu will start melting at the surface and this can have a detrimental effect on both foils. Therefore, thin Cu layers (of some microns up to 20-30 microns) are not adequate for this method (see figure 2). On the other hand, the method is ideal for metals with electrical resistivity lower than that of W, as e.g. for V and Ti. In this case, the heat flow from the metal toward W and it is possible to adjust the processing temperature

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to obtain a brazing like joining process. It is important to stress that, the short processing time as well as the fact that using FAST joining make possible to produce specimens with different shapes and forms (profiles), including directly pipe formed components (see figure 1 top image) are advantages worth to consider as a potential processing method.

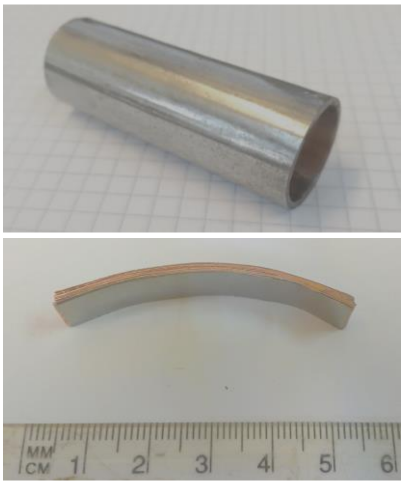

Fig. 1. W-Cu laminates produced by FAST joining: top image a pipe with 60 mm length processed directly as pipe in one step Example a one-column figure; bottom image a 50×10×2 mm laminate produced in the hybrid FAST-hot-rolling prototype.

As shown in the bottom image of figure 1, it is also possible to create a hybrid FAST-hot rolling equipment able to join materials in a dynamic regime. The W-Cu laminate shown in the image was produced in this way by a laboratory scale prototype inserted in our FAST equipment vessel (40×30× 30 cm^3) and the process can be up-scaled to an industrial production.

In this work we present microstructural, mechanical and thermo-physical properties results obtained for W-Cu laminates produced by this method.

2. Experimental

100 μm thick W foils produced by Plansee and commercially available Cu foils with different 7, 25, 50 and 100 μm thickness have been joined by FAST at about 900 °C (please note that this is the average temperature read by pyrometer at the top graphite piston surface). The joining was performed in Ar atmosphere at about 1 Pa applying different pressing forces perpendicular to the stacked foils.

As already mentioned in introduction, due to the complex phenomenology occurring in the FAST joining process of metals, there is a strong dependence of the produce specimens' quality on the material, number and thickness of foils as well as on temperature and applied pressure. Figure 2 illustrates the differences for W-Cu laminates with 20 pairs of 100 μm W and 25 μm Cu foils with the same parameters used for similar laminates with 100 μm Cu foils (top) and with optimized parameters for the reduced Cu thickness.

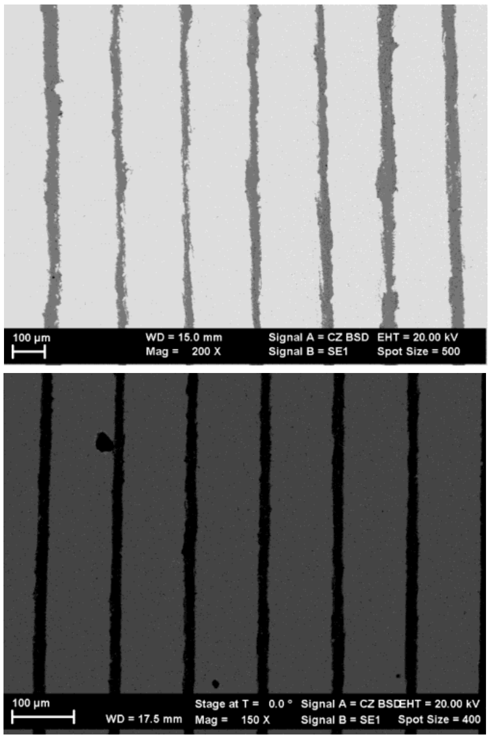

Fig. 2. Comparison of W-Cu laminates produced by FAST using un-optimized (top) and optimized (bottom) parameters (see text for details). The SEM images are obtained with the back-scattered electrons.

Generally, the process parameters temperature and pressure are around 860 °C and 35 MPa respectively for 20 pairs of 100 μm W and Cu foils (e.g. the samples for Charpy tests) and should be decreased with decreasing number of foils and with decreasing Cu foil thickness. For very thin Cu foils (7 μm) it was in fact not possible to obtain uniform layers after the FAST joining, as shown in figure 3.

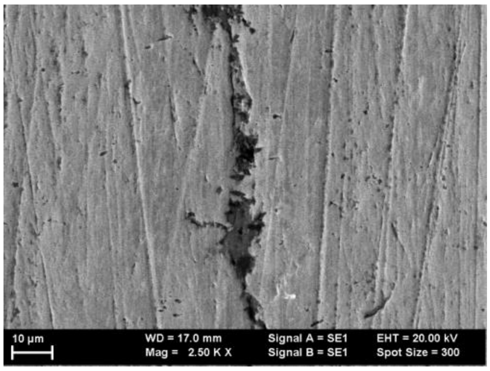

Fig. 3. SEM secondary electrons image of a W-Cu laminates produced by FAST using 7 μm thick Cu foils.

3. Results and discussion

3.1 Mechanical properties

The mechanical properties of FAST joined laminates produced in NIMP have been investigated at IAM-KIT. The tensile tests were performed at different temperatures using standard specimens as depicted in figure 4. The specimens were processed by EDM from 10×40×0.5 mm^3 samples produced with 3 foils of W and 12 foils of Cu, both type with 100 μm thickness.

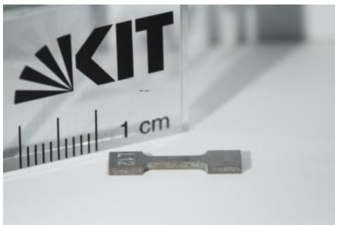

Fig. 4. W-Cu laminate specimen for tensile tests.

In figure 5 the stress-strain curves obtained at different temperatures are summarized for W-Cu laminates produced by FAST (black symbols), diffusion bonded (red symbols) and Ar soldered (pink symbol) together with the results obtained for W-CuCrZr laminates produced by diffusion bonding, with and without a heat treatment (2 hours at 470 °C in vacuum). From start it can be seen that at room temperature none of the samples is able to bear elongations around 15%, as shown for instance in figure 24 from ref. [20]. In fact for all samples included here the maximal elongation at room temperature is around 1% or less.

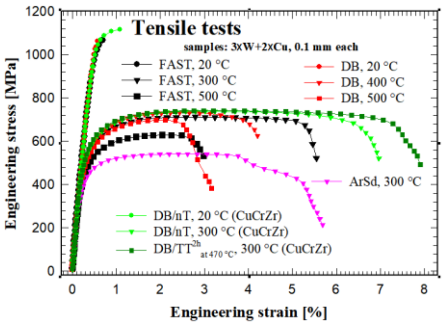

Fig. 5. Comparison of W-Cu laminates' tensile behavior for specimens produced by different methods (FAST in NIMP), (DB-diffusion bonding in KIT), (ArSd- soldered in Ar atmosphere in KIT) with W-CuCrZr laminates produced by diffusion bonding at KIT with and without a subsequent annealing at 470 °C.

These results suggest that at room temperature at least the W-Cu laminates break quickly after leaving the stage where W is partially elastic and Cu is partially plastic, thus making Cu failure responsible for the fracture. This can be explained in the FAST joined specimens by the violent heating process, which is susceptible of voids formations in Cu and as a result increase the risk of brittle fracture. On the other side, at high temperatures, both the diffusion bonded and FAST bonded materials outperform the W foil and also the reference materials from ref. [20] in terms of yield stress. If we compare the present results with those from figure 24 from ref. [20], we can assume that the heating of the material at moderate temperatures helps Cu to recover its plasticity.

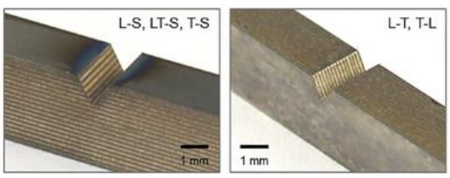

Fig. 6. W-Cu specimen orientation for Charpy tests.

The Charpy impact tests have been performed on KLST notched samples as depicted in figure 6, for the FAST joined samples. The samples have been prepared with 20 pairs of 100 μm W and Cu foils. The results are presented in figure 7 for different temperatures, for both L-S and L-T directions.

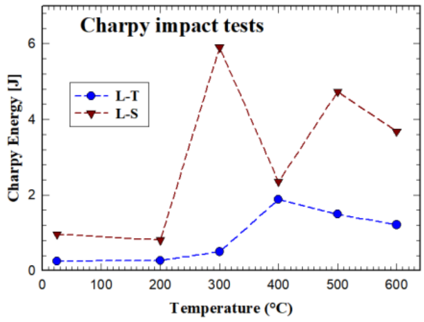

Fig. 7. Charpy impact tests results for W-Cu laminates produced by FAST joining.

Inspection of the data plotted in figure 7 shows that the Charpy energy values for both directions and regardless of the temperature are similar at low temperatures (below 350 °C and much smaller above this temperature than the value reported in the literature for diffusion bonded W-Cu laminates (compare e.g. figure 20 from ref. [20]). This is somehow puzzling, since the tensile properties showed a similar if not better behavior than the W-Cu diffusion bonded laminates. A possible reason for this might be connected with the fact that the rolling direction of the foils was not followed during the specimens processing and the distinction between L and T directions was lost. Indeed, as shown for W-AgCu laminates in figure 5 from ref. [20], different values are obtained for L-S and T-S orientations.

An other factor able to slightly affect the impact behavior is the C contamination of the exposed W foil during the FAST process. The top foil was in direct contact with a graphite foil and therefore at least this foil might have been compromised. Note that the C presence in W is known to make it more brittle. In the case of tensile specimens, additional W foils were placed top and bottom to reduce the sample contamination.

3.2 Thermophysical properties

The thermal transport properties have been investigated using a Netzsch LFA 457 Microflash up to 1000 °C in the transversal mode (i.e. the laser pulse perpendicular on the foil surfaces) and the expansion coefficients have been determined in the same temperature range using a Netzsch 402 C dilatometer with thermal expansion measured along the interface direction. The LFA equipment allows the direct measurement of the thermal diffusivity, while the specific heat of materials, can be determined by a differential method using a reference sample (Molybdenum_SRM781). The thermal conductivity is calculated by $\lambda = \alpha \times \rho \times C_p$, with ρ the density and α the diffusivity of the material.

To asses the interface quality for different number of foils and Cu interlayer thickness values, thermal transport measurements have been performed on samples with 10 or 20 pairs of W and Cu foils, for each Cu thickness (i.e. 7, 25, 50 and 100 μm). The results are plotted in figure 7.

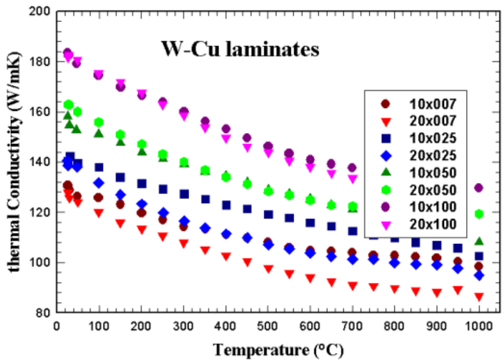

Fig. 7. Thermal conductivity for different W-Cu laminates produced by FAST. The legends designates the umber of W-Cu pairs joined together (10 or 20) followed by the thickness of Cu foils in microns.

As already mentioned, for thinner Cu foils the quality of the joints decreases, and this is reflected in differences obtained between the thermal conductivity values for samples with similar materials but different number of interfaces. Meanwhile the differences for samples with thicker Cu interlayers are reduced below the measurement errors. One should note also that the thermal conductivity of a W foil, perpendicular on the foil is smaller than the bulk W thermal conductivity with about 15% (i.e. ~ 140 W/m/K at room temperature, instead of ~ 170 W/m/K). Thus, even for the 25 mm Cu interlayers the expected thermal conductivity contribution of Cu in the W-Cu laminate is almost compensated by the interface thermal contact resistance.

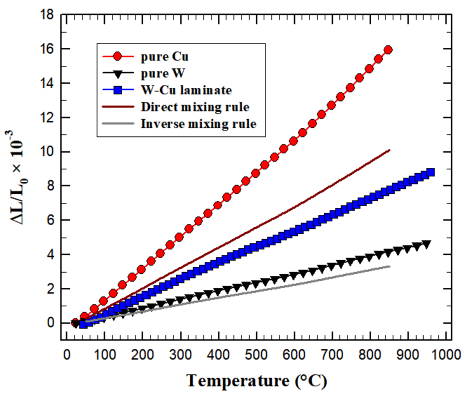

Fig. 8. Thermal expansion measured in the L direction for W-Cu laminate. The solid line are the calculated values derived from the direct and inverse mixing rule.

In figure 8, the thermal expansion of a W-Cu laminate produced with 20 pairs of 100 mm W and Cu foils is compared with the values of pure bulk Cu and W materials and values derived from the direct and inverse mixing rule. The increasing deviation from the direct mixing rule value suggest that above ~ 150 °C the Cu enters in a plastic regime while W remain in the elastic regime.

4. Conclusions

In this work we have demonstrated that FAST can be successfully used to process W-Cu laminates. From the sample morphology and thermal properties point of view the best results are obtained for 0.05 ÷ 0.1 mm thick Cu foils. Tensile tests results show that the FAST processed W-Cu laminates are comparable to those obtained for by a diffusion bonding route. Charpy impact tests show that the FAST joined W-Cu might be comparable with diffusion bonded similar materials but the Charpy energy values are ~ 50% compared to the best materials from literature, i.e. W-CuAg laminates.

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