4.2 RECENT PROGRESS IN THE FABRICATION AND CHARACTERIZATION OF DUCTILE-PHASE-TOUGHENED TUNGSTEN COMPOSITES FOR PLASMA-FACING MATERIALS –K. H. Cunningham, G. R. Odette, K. Fields, D. Gragg, and F. W. Zok (University of California, Santa Barbara), C. H. Henager, Jr., R. J. Kurtz, T. J. Roosendaal, and B. A. Borlaug (Pacific Northwest National Laboratory)

OBJECTIVE

The objective of this study is to develop ductile-phase-toughened tungsten composites as candidates for plasma-facing components in future fusion reactors.

SUMMARY

A promising approach to increasing fracture toughness and decreasing the DBTT of a W-alloy is by ductile-phase toughening (DPT). In this method, a ductile phase is included in a brittle matrix to prevent crack propagation by bridging. Composite specimens were fabricated by two methods: 1) sintering W powders together with carburized W wires, and 2) hot pressing W and Cu foils together with W wires. These composites were tested in tension and three-point bending.

PROGRESS AND STATUS

Background

Background information and previous progress have been discussed in earlier semi-annual reports [1-2].

Experimental

W-Wires and W and Cu Foils as Reinforcements

We have previously reported the tensile properties of tungsten wire, which motivated an investigation of using W-wire as reinforcement. Table 1 summarizes the tensile properties of W-wire of various diameters. [2] A general trend of increased strength and decreased ductility is observed as the wire diameter decreases. The ultimate strength reaches a remarkable value of ≈ 4.25 GPa in the 15-µm diameter wire, with about 1.0% total elongation. While the strengths are lower for the larger diameter wires the total elongations are about 1.7 and 2.4% respectively.
The 250-µm W wires, used to make the composites described below, were characterized to quantify how their properties evolved after several of the high-temperature processing steps used in composite fabrication. After processing at 1900°C for 5 minutes during the carburization process described below, only modest effects on the grain structure and texture of the wires were observed in EBSD measurements, which require further quantitative analysis. The hardness of the wire decreased about 9% after processing treatments of hot pressing the laminate in vacuum at 900°C for 5 minutes as well as performing an oxide reduction for 360 minutes in H_2 at 900°C, followed by a second hot pressing step under the same conditions as the first for a total of 370 min at temperature.

Table 1. Average values of W wire properties from tensile testing. [2]

Wire Diameter (µm)	Yield Stress (GPa)	Ultimate Tensile Strength (GPa)	Total Elongation (%)	Reduction in Area (%)
15	3.58	4.24	1.0	N/A
250	1.78	2.30	1.7	29.9
500	1.34	2.16	2.4	27.1

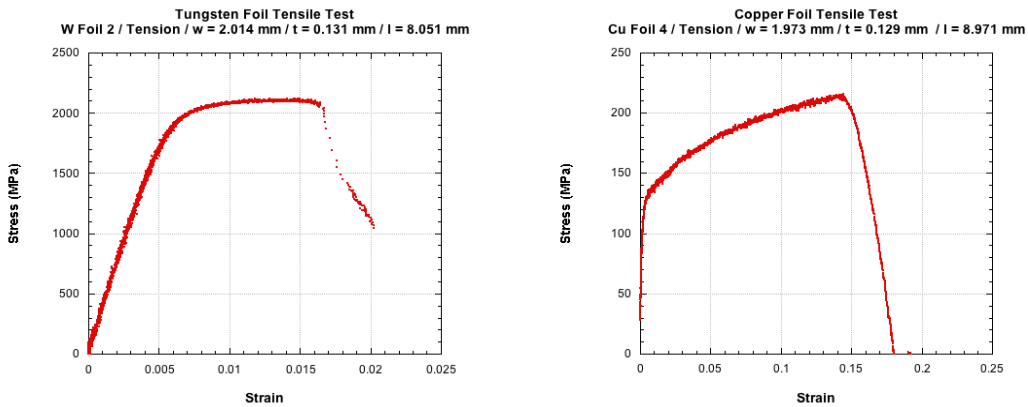


Figure 1. Representative stress-strain curves for tungsten (left) and copper (right) foils. Note the order-of magnitude differences for both the stress and strain axes between graphs.

We also tested pure W and Cu foils with nominal thickness 127 µm. These foils were used to fabricate a laminate composite as discussed below. Tensile tests were performed at a strain rate of 0.011 min^{-1} at ambient temperature in air, with a gauge length and width of 9 mm and 2 mm, respectively. Strain was measured with a laser extensometer. Representative stress-strain curves for the pure tungsten and copper foils are shown in Figure 1. The tensile properties are summarized in Table 2. The W-foil has a high strength and limited ductility. The Cu-foil has low strength, but high ductility.

Table 2. Summary of tensile data for tungsten and copper foils (nominal thickness = 127 µm).

	Yield Stress (MPa)	Ultimate Tensile Strength (MPa)	Total Elongation (%)
W	1997.7 ± 10.5	2119.4 ± 4.5	3.4 ± 1.3
Cu	130.2 ± 4.6	218.7 ± 4.9	16.9 ± 3.3

The W-WC Sintered Composite System

Fiber-reinforced composites were explored with unidirectional coated W wires in a W matrix. Efforts to use a Cu coating on the W wire have been reported previously. [2] Tungsten carbide (WC) was investigated as a potential highly stable debonding layer since it is even more brittle than W and because C is a suitable element for the divertor in terms of low neutron activation and reduced potential for plasma contamination. 250-µm diameter W wires were placed apart in loose graphite powder, and then compacted at 35 MPa at ambient temperature in the SPS system. The compact was heat treated at 1800°C for 1 minute with a heating ramp of 300°C/min under a minimum load of 9 MPa. The resulting WC layer on the wire was 12 µm thick. To consolidate a sample from which two test specimens containing one embedded wire could be prepared, two coated wires were then laid separately and parallel in W powder of particle size 4-6 µm. The wires and powder were sintered at 1900°C for 15 minutes at 50 MPa. Notched specimens of the sintered composite were tested in three-point bending with the goal of observing debonding behavior during crack propagation. Notched three-point bend test samples were EDM cut with a test span, width, thickness, and notch depth of 10 mm, 3.6 mm, 3.6 mm, and 0.5 mm, respectively. The crack propagation direction was perpendicular to the wire direction. Bend testing was performed with a crosshead displacement rate of 0.05 mm/min at ambient temperature in air.

The W-Cu Hot-Pressed Composite System

To avoid the issue of Cu melting and wicking during the sintering of a W matrix, a hot-pressing fabrication route was developed for W wires sandwiched between W and Cu foils, as illustrated in Figure 2a. The top and bottom layers were 127-µm W foils. The inner layers were the 127-µm Cu foils, while the middle layer contained 250-µm W wires. The laminate was 5cm x 5cm, with an average of 50 µm at the shortest distance between the parallel fibers. After hot pressing at 900°C at 38 MPa for 5 minutes, the W-wires were embedded in the Cu, forming a 3-layer sandwich. Tensile test samples of the reinforcement were EDM cut with a gauge length, width, and thickness of 6.4 mm, 2.45 mm, and 0.8 mm, respectively. The tensile direction was parallel to the wire direction. Tensile testing was performed with crosshead displacement rates of 0.1 and 0.1125 mm/min at ambient temperature in air.

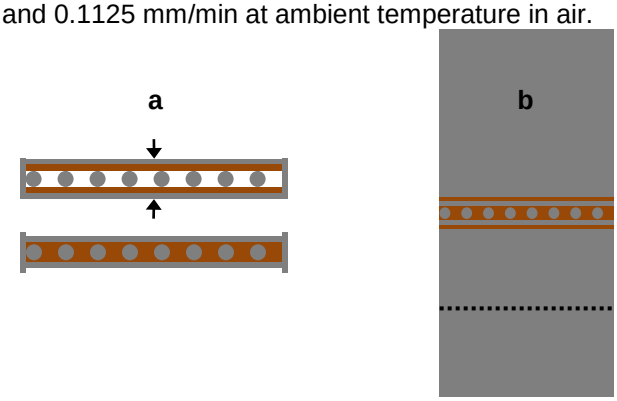


Figure 2. a) Schematic diagram of reinforcement cross-section before (top) and after (bottom) hot pressing.
b) Schematic diagram of laminate bend specimen cross-section. Dashed line indicates notch depth.

The W-foil//Cu//W-wire sandwich reinforcement was then hot pressed between 4-mm thick W plates, as illustrated in Figure 2b. Since the surface roughness of the reinforcement was ≈ 100 µm, reflecting the underlying topology of the stronger W-wires, a Cu bond layer of 127-µm foil was used between the W-plates and sandwich. However, in spite of this extra layer the volume/atom fraction of Cu is much lower at ≈ 10% in the laminate, compared to ≈ 50% in the heavy metal W-Cu composites studied previously. Notched three-point bend test samples were EDM cut with a test span, width, thickness, and notch depth of 27.6 mm, 8.9 mm, 3.1 mm, and 2 mm, respectively. The crack propagation direction was perpendicular to the wire direction. Bend testing was performed with a crosshead displacement rate of 0.02 mm/min at ambient temperature in air.

RESULTS

W-WC Sintered Composite

No bridging was observed in the W-WC sintered composite, since the cracks ran directly though the coated wire without any debonding. Despite the presence of some porosity at the WC-matrix interface, this may be due to the fact that the WC layer was strongly bonded to both the W matrix and wire. A representative load-displacement curve shown in Figure 3 illustrates the brittle response of the material.

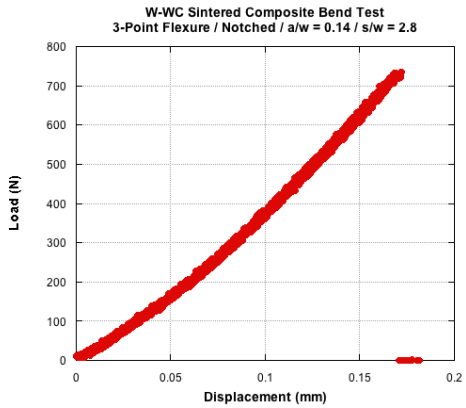
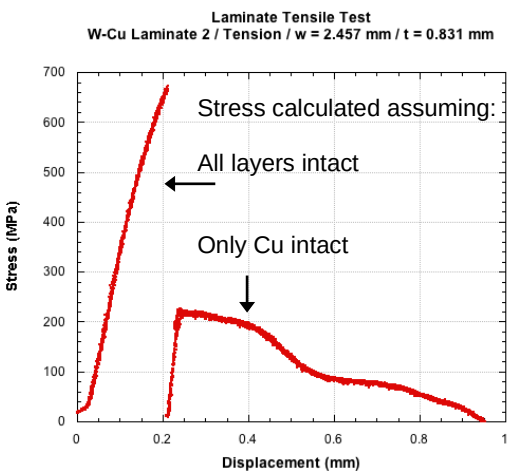


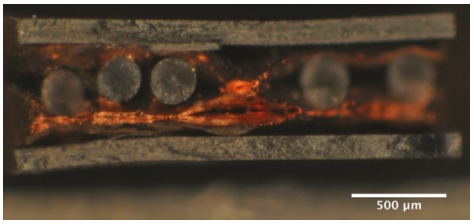
Figure 3. Representative load-crosshead displacement curve for notched 3-point bend tests on W-WC sintered composite, showing brittle failure.

W-Cu Hot-Pressed Composite

Reinforcement Tensile Testing

A representative stress-crosshead displacement curve and images of two different broken test specimens are shown in Figure 4. Note that the plot shows one curve with a discontinuity, as the test was paused and resumed after the W foils cracked. The reinforcement showed some evidence of fiber pullout but no indication of plasticity in the W-wires. The outer W-foils cracked at the peak stress, followed by fracture of the W-wires. Deformation occurred in the Cu layer at a much lower stress until up to ductile failure. Determination of the stress contribution from wire pullout is in progress.





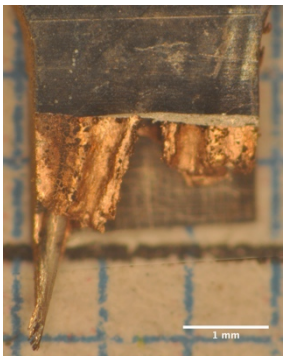


Figure 4. Left: stress-displacement curve for laminate tensile test 2. Top right: fracture surface of specimen 1. Bottom right: protruding W wire from specimen 2 suggests energy dissipation by pullout.

Laminate Notched Three-Point Bend Testing

The results are summarized in Figure 5. The stress was calculated using the applied load and initial geometry of the test specimen. In one test (specimen 1), the crack propagated through the W plate up to

the embedded laminate and re-nucleated on the other side, leaving the reinforcement sandwich intact. At this point the specimen could only sustain a low load, of ≈ 130 N. In a second test (specimen 2), cracks grew from the notch parallel to the sandwich, before branching with one crack deflecting 90° and propagating up to the sandwich at the first pop-in stress drop. This crack arrested and the stress increases again until the crack re-nucleated at the back of the sandwich marked by the second stress drop. However, the laminate composite was still able to sustain a significant load in this case, which actually then increased prior to a gradual drop-off associated with continued deformation of the sandwich layer. Removing the Cu using an acid bath showed that several of the W wires had fractured approximately 0.075 to 1.4 mm away from the bending point, indicating that fiber pullout could play a role in the mechanical response of the composite. Peak-load stress intensity factor values ranged from 20 to 34 MPa√m. These results are very encouraging and suggest that sandwich reinforcements could provide a route to developing tough W composites.

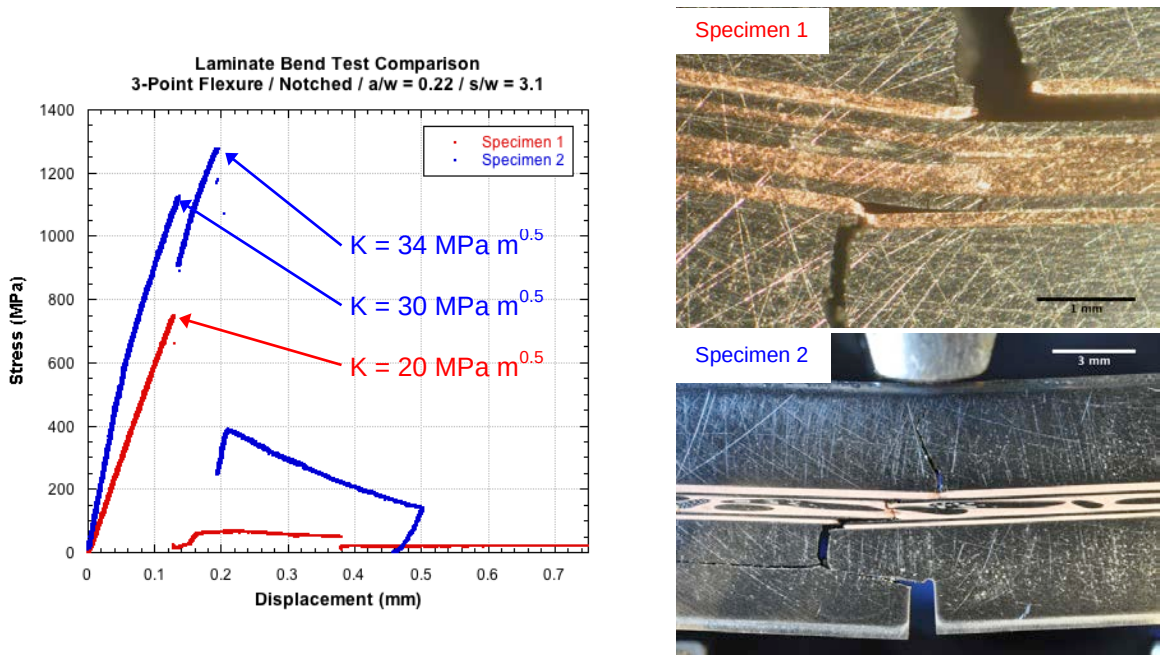


Figure 5. Stress-displacement curves for two bend specimens. Right top: specimen 1 after testing Right bottom: specimen 2 after testing.

ONGOING AND FUTURE WORK

The next steps underway are to fabricate multilayer laminate composites by hot pressing 1-mm W plate with various metallic foils, including copper, iron, stainless steels, and vanadium. These composites will be notched, precracked, and tested in three-point bending to measure relative resistance to crack growth. These experiments will be complemented by bridging model calculations. The model, along with fracture data, the method and cost of synthesis, and microstructure examinations will be used to guide the future design of improved W-composites. We will link our composite development effort to thermal-mechanical simulations of W-based divertor components and will attempt to fabricate and evaluate some divertor-type components. Ultimately, a clear understanding of the materials science of W-composites will be developed with relation to: a) the choice and behavior of the ductile phase; b) the synthesis, processing, and fabrication of composites; and c) the composite's microstructure as it evolves during service.

REFERENCES

[1] K.H. Cunningham, K. Fields, D. Gragg, F.W. Zok, C.H. Henager, Jr, R.J. Kurtz, and T. Roosendaal, Recent Progress in the Development of Ductile-Phase Toughened Tungsten for Plasma-Facing Materials, in DOE/ER-0313/54 - Volume 54, Semiannual Progress Report, June 30, 2013. 2013, US DOE: ORNL, TN.

[2] C.H. Henager, Jr, R. J. Kurtz, T. J. Roosendaal, B. A. Borlaug, G. R. Odette, K. H. Cunningham, K. Fields, D. Gragg, and F. W. Zok, Recent Progress in the Development of Ductile-Phase Toughened Tungsten for Plasma-Facing Materials, in DOE/ER-0313/55 - Volume 55, Semiannual Progress Report, Dec. 31, 2013. 2013, US DOE: ORNL, TN.