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Processing of complex near-net-shaped tungsten parts by PIM

Steffen Antusch[a,*], Jan Hoffmann[a], Alexander Klein[a], James P. Gunn[b], Michael Rieth[a], Tobias Weingaertner[a]

[a] Karlsruhe Institute of Technology (KIT), Institute for Applied Materials, P.O. Box 3640, Karlsruhe 76021, Germany
[b] CEA, IRFM, F-13108 Saint-Paul-Lez-Durance, France

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ABSTRACT

Powder Injection molding (PIM) as near-net-shape technology allows the effective mass fabrication of complex tungsten parts. Depending on the size and shape of the parts, for simple geometries only 20 s needed to produce a part. This method offers particularly the advantage of cost-saving. Isotropic materials, equiaxed grain orientation, good thermal shock resistance, shape complexity and high final density are several typical properties of powder injection molded tungsten. Therefore, this technique is an ideal tool for scientific investigations and for developing industrial products. This work describes the characterization and analyses of prototype materials produced via PIM. The investigation of pure tungsten and oxide or carbide doped tungsten materials comprises the mechanical testing, element allocation, and texture analyses. In addition, fabricated near-net-shape Langmuir probes for diagnostics for the French tokamak WEST were presented. Pure tungsten showed a ductile behavior at 200 °C. By addition of carbide and oxide particles the yield strength increases significantly compared to pure tungsten. This means, by varying the amount and type of the dopant (oxide or carbide powder) the material properties can be tailored within a broad range to meet different customer requirements and demonstrating its high potential for fusion applications.

1. Introduction

In the framework of the European material development programme for fusion power plants beyond the international thermonuclear experimental reactor (ITER), tungsten and tungsten-based materials are considered as potential plasma-facing materials. This is a result of their favorable properties such as the high melting point of 3422 °C (3695 K), high thermal conductivity (173 Wm-1K-1 at room temperature), low tritium retention, and high temperature strength. The main drawbacks are its brittleness and the time and cost aspect for commercial tungsten manufacturing. As the production of tungsten parts with conventional technologies is time and cost intensive, a promising fabrication method in view of large-scale production is powder injection moulding (PIM). With its high near-net-shape precision it offers the advantage of reduced costs compared to conventional machining.

In this work, the focus is set on the development of particle reinforced W-PIM materials. Therefore, powder mixtures of commercially available powders with the alloying elements Y_2O_3 and TiC are used. The characterization of the materials was performed by 4-point bending tests, Auger electron spectroscopy (AES), and texture analyses via electron backscatter diffraction (EBSD). The addition of oxide or carbide particles into the tungsten matrix modifies its mechanical properties, such as ductility and strength, in comparison to pure tungsten.

In the past, the feasibility of plasma confinement and a controlled fusion reaction have been successfully demonstrated in many tokamaks worldwide. The next steps on the road to commercial fusion power plants will be first the experimental reactor ITER, followed by demonstration power plants (DEMO). The materials used for such tokamaks (ITER and DEMO) have to withstand high particle fluxes, comprising electrons, ions, neutrons, atoms, and have to sustain high heat fluxes, mainly at the first wall and especially at the divertor. Plasma measurement and control requires data acquisition at many points near the divertor surface where temperatures well above 1000 °C may occur. This can be done by so-called Langmuir probes (named after the American chemist and physicist Irving Langmuir), which allow the determination of the temperature, density and electric potential of the fusion plasma. In principle, such probes are specially shaped massive tungsten plates with holes for fixing, isolating and connecting electrical wires. The French tokamak WEST (W Environment in Steady-state Tokamak), which is currently in the phase of commissioning, is intended to become one of EUROfusion's test benches for tungsten components under ITER-like conditions. Langmuir probes are foreseen in

* Corresponding author.
E-mail address: steffen.antusch@kit.edu (S. Antusch).

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the ITER divertor, but they will suffer even harsher conditions than the divertor targets themselves because they cannot be directly cooled. In addition, the available space available for their installation is limited. The WEST platform gave an opportunity to use PIM for the (mass-) fabrication of Langmuir probes [1] in order to address some of the engineering and physics issues of concern to ITER.

2. Experimental

2.1. Materials

PIM is a manufacturing process in powder metallurgy for shaping metals and ceramics to near-net-shapes with a reasonably tight tolerance and good surface finish. The mixture of an inorganic metal or ceramic powder with a small quantity of a polymer (binder) results in a so-called feedstock, which can be moulded.

Depending on the size and shape of the parts, for simple geometries only 20 s are needed to produce a green part [2]. After shaping the green part (consisting of powder and binder), the polymeric binder must be extracted and the final sintering >2000°C leads to a density near to the theoretical density. In this way, the manufactured pure tungsten parts reach a density higher than 98% of the theoretical one and the parts are free of cracks [3]. This process is very effective. The easy up- and down-scaling in size and shape variation of the parts is shown in Fig. 1 [4].

Three W grade were developed: pure W (>99.97 wt.% W), W-1TiC (99 wt.% W + 1 wt.% TiC), and W-2Y_2O_3 (98 wt.% W + 2 wt.% Y_2O_3).

Rolled tungsten (>99.97 wt.% W) produced by PLANSEE was used as reference.

2.2. Material testing and characterization

To analyse the mechanical properties, in particular the brittle-to-ductile transition temperature (BDTT) and yield stress as a function of temperature, small samples of 12 mm × 1 mm × 1 mm (tolerance ± 0.01) were produced by electrical discharge machining (EDM). After polishing of all four sides of each sample, the samples were notched by EDM. The 4-point bending tests were carried out on an INSTRON 3366 testing machine with a constant strain rate of 3.3E-05, with the cross-head velocity adjusted in each test to account for the different dimensions of the beams, at temperatures from 20 °C to 400 °C [5–7]. The raw load displacement data were then converted into stress-strain data.

AES with an Auger Nanoprobe PHI 680 (Physical Electronics, USA) was used to characterize the element allocation of tungsten and the oxide or carbide particles. Each sample was broken inside the ultra high vacuum chamber (at 4*10^−7 Pa) of the device to produce clean fresh surfaces for the examination. Acceleration voltage of the exciting electron beam was fixed at 10 keV, and the sample current was aligned to 20nA. The used pixel raster for the detection of the elemental maps, was 512 × 512 pixels over the complete field of view.

The EBSD analysis was performed using a Zeiss Auriga CrossBeam electron microscope (FIB/SEM) equipped with an EDAX Hikari camera. The experiments were done using an acceleration voltage of 20 kV with a 120 µm aperture in high current mode. This produces a beam current of approximately 10 nA. For the mappings, a step size of 40 nm (W-TiC), 80 nm (W-Y_2O_3) and 900 nm (pure W) was chosen. Points with a confidence index (CI) lower than 0.1 were removed during post-processing. After a grain CI standardization, a grain dilation algorithm with a tolerance angle of 5° was used to clean the datasets. Misorientations exceeding 5° were considered grain boundaries and are highlighted in black in the inverse pole figure (IPF) maps. The IPF maps are displayed with respect to the parallel sample directions.

3. Results

3.1. 4-point bending tests

The results are shown in Fig. 2.

Pure tungsten shows ductility starting at 200 °C and W-1TiC at 300 °C. W-2Y_2O_3 shows brittle failure up to 300 °C, while above 400 °C no fracture was observed. By addition of carbide and oxide particles the yield strength increases significantly compared to pure tungsten. This means, by varying the amount and type of the dopant (oxide or carbide powder) the material properties can be tailored within a broad range to meet different customer requirements. Rolled tungsten from PLANSEE is used as a reference material (see Fig. 2(b)). This material shows a very high stress level, but in comparison to pure W-PIM (Fig. 2(a)) less strain to failure indicating limited ductility in this material.

3.2. Element allocation

Fig. 3 shows the results for the oxide and carbide doped materials. It can be clearly seen that the added elements (red spots) are embedded mainly at the tungsten grain boundaries.

That is, the tungsten grains are surrounded by tiny particles, which provides a stabilisation of the tungsten microstructure. In other words, the carbide or oxide particles act as grain growth inhibitors. The authors estimate that the achieved grain size of the particle stabilized materials varies between 4 and 8 µm only. Compared to pure W with typical grain sizes of 50–100 µm this is a major improvement.

3.3. Crystallographic texture

Another benefit of the PIM materials is their isotropic microstructure (equiaxed grain orientation). This has been already been demonstrated by Antusch et al. [8]. Fig. 4 shows the results of EBSD

Fig. 1. Up- and down scaling in size dimensions and shape complexity of finished W-PIM parts. The range of dimension of the produced parts are from a micro gearwheel 3 mm in diameter and a weight of 0.050 g, up to a 1.4 kilo plate with the dimensions 60 mm × 60 mm × 20 mm.

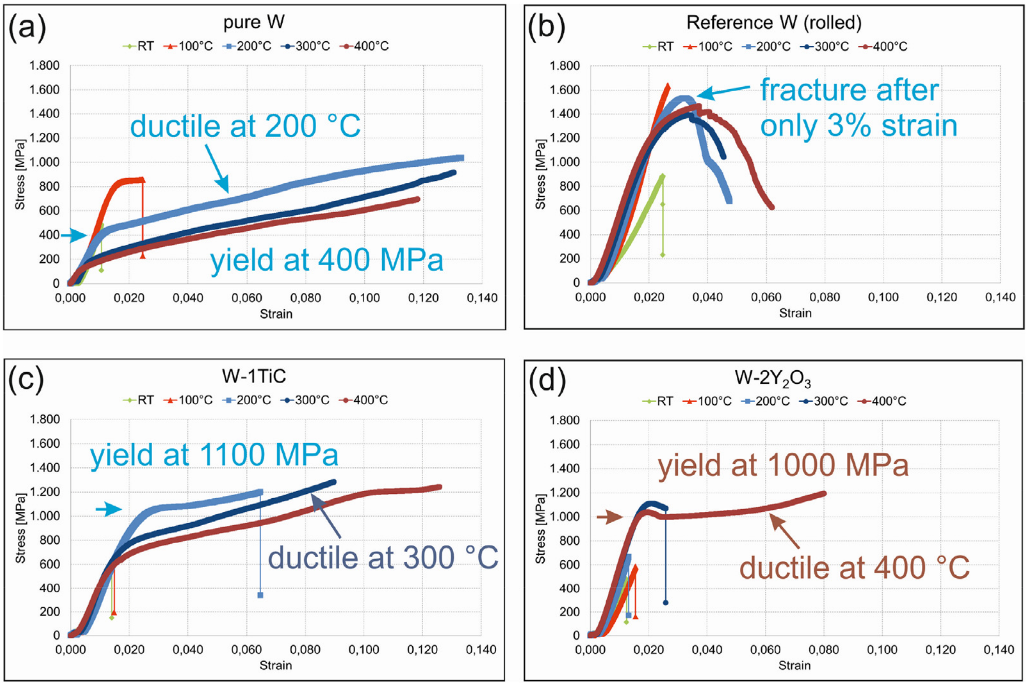

Fig. 2. Complete stress-strain curves of 4-point bending tests carried out from 20 °C to 400 °C (a) pure W, (b) reference W (rolled), (c) W-1TiC, and (d) W-$2Y_2O_3$.

measurements.

The maps show the isotropic grain structure for the powder injection moulded materials, which is independent of the added particle type. Unindexed black spots in the maps are in the same size range as the particles measured by AES (see Fig. 3). Since the oxide particles produce significantly weaker EBSD patterns than the surrounding W-matrix, they could not be indexed at the same time with the bulk material. The authors come to the conclusion that the unindexed parts belong to the respective strengthening particles.

The smaller grain size of the particle added materials demonstrates that TiC and Y_2O_3 are rather effective in pinning (stabilising) the grains during the sintering process. Given the very high sintering temperature >2000 °C, both doped tungsten alloys can certainly be considered as fully recrystallized and grain growth resistant during the consolidation. This may be an indicator for raised application temperatures of up to 1800 °C or even higher. Ageing studies which could confirm this behavior are planned and under investigation. In this context and with respect to the observed grain size, TiC-additions seem to be a bit more effective compared to Y_2O_3. A comparison to commercially available tungsten material is shown in Fig. 4(d). This material is not in a recrystallized state and shows grains with a prominent sub-grain-structure inside. This can be seen by the large color gradients inside the grains as opposed to the solid-colored grains to the PIM materials. The color in Fig. 4(c) proved to be artifacts of the sample preparation are not a real property of the material. In terms the overall microstructure, the commercial material has significantly smaller grain size.

3.4. W-PIM samples for WEST

Fig. 5 shows these Langmuir probes, which will be used for the WEST diagnostics.

Each of the delivered 70 probes is 25 mm long, 17 mm high and only 2 mm thick, and has to withstand the harsh conditions inside tokamaks. This is the first demonstration of the successful use of W-PIM as a production route for functional parts within a fusion plasma environment.

4. Conclusions

The obvious drawback of pure PIM-W is its coarse-grained microstructure. Nevertheless, for functional applications where the mechanical properties play a minor role, PIM is a cost-efficient and, therefore,

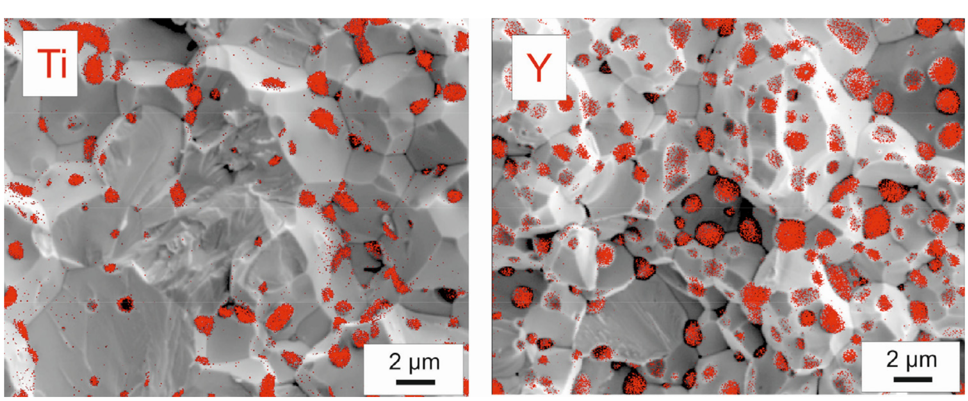

Fig. 3. (left) W-1TiC: Ti containing particles (red spots) around the W grains (right) W-$2Y_2O_3$: Y containing particles (red spots) around the W grains. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

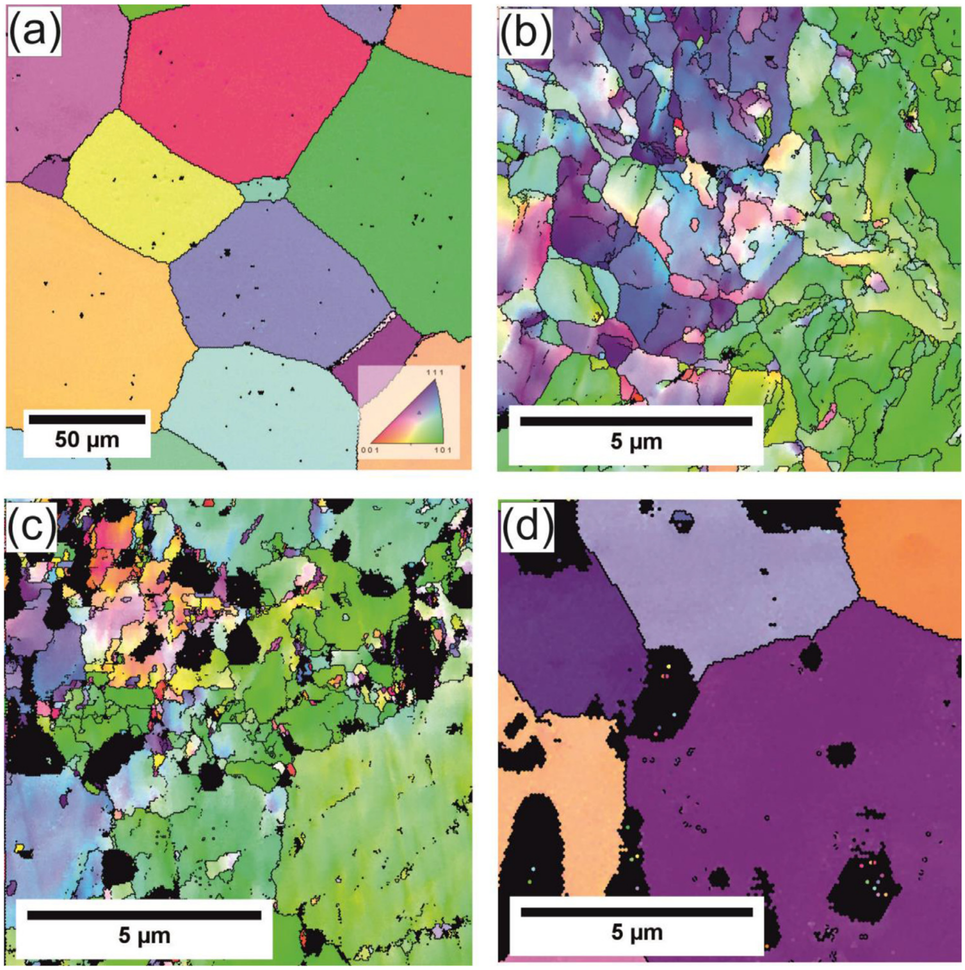

Fig. 4. EBSD maps: (a) pure W, (b) reference W (rolled, RD from top to bottom), (c) W-1TiC, and (d) W-2Y_2O_3. The black spots in (c) correspond to TiC and to Y_2O_3 particles in (d).

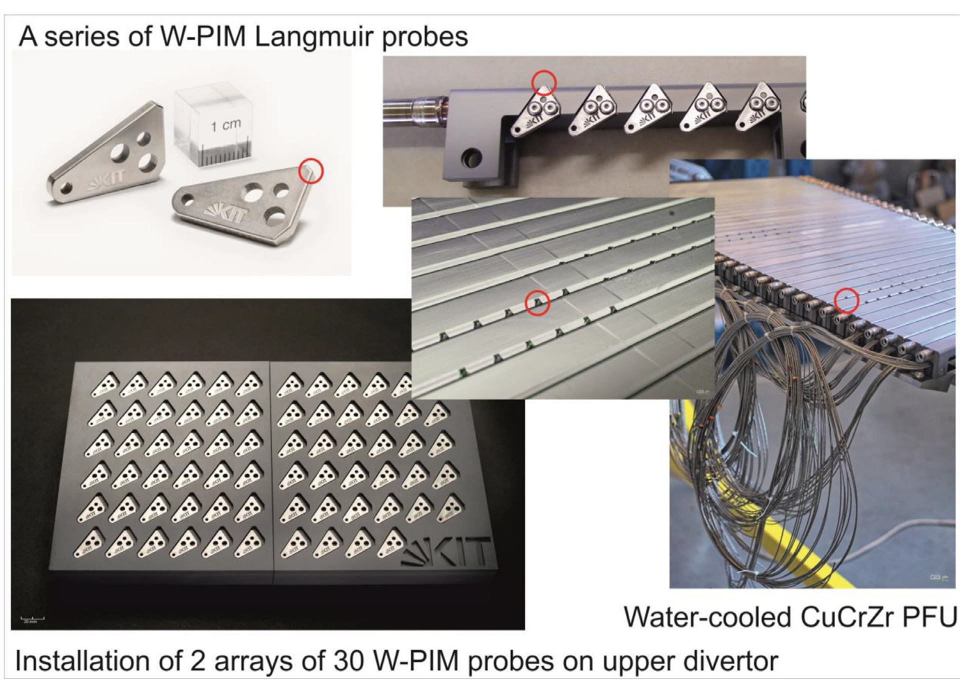

Fig. 5. W-PIM Langmuir probes for WEST.

competitive mass-fabrication route for functional, highly dense tungsten parts. This has been demonstrated by the successful production of 70 Langmuir probes for the WEST tokamak.

For structural and armour applications, the requirements of tungsten materials in terms of ductility, strength, recrystallization and grain growth resistance are decisive. In this context, PIM can be applied as a materials development tool for rapid tungsten alloys prototyping, which is the basis for fast-screening possible or promising compositions. This has been explored for W-Y_2O_3 and W-TiC with two main results:

(I) The mechanical properties, such as ductility and strength, are tunable in a wide range. This allows the rapid fabrication of tungsten materials with optimum properties for a given application.
(II) The added particles stabilise the tungsten grain boundaries. As a result, the produced tungsten alloys show an isotropic fine-grained microstructure with an extremely high recrystallization resistance.

Moreover, fracture mechanics and high heat flux tests indicate lower brittle-ductile transition temperatures and higher crack resistance compared to tungsten materials produced by common PM routes. Therefore, tungsten alloys produced by PIM might be a applicable in high-heat flux areas in nuclear fusion [9]. Based on these encouraging results, the PIM process will certainly be used for the development of new custom-made tungsten materials as well as for further scientific investigations on prototype materials. The aim is to broaden the range of applications in the general field of energy conversion and other areas typical for refractory alloys.

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