TWO-COMPONENT TUNGSTEN POWDER INJECTION MOLDING FOR MASS PRODUCTION OF He-COOLED DEMO DIVERTOR PARTS

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Fusion technology as a possible and promising alternative energy source for the future is intensively investigated at Karlsruhe Institute of Technology (KIT). The KIT divertor design for the future DEMO fusion power plant is based on a modular concept of He-cooling finger units. More than 250,000 single parts are needed for the whole divertor system, where the most promising divertor material, tungsten, must withstand steady-state heat loads of up to 10 MW/m^2.

Powder injection molding (PIM) as a mass-oriented manufacturing method of parts with high near-net-shape precision has been adapted and developed at KIT for producing tungsten parts, which provides a cost-saving alternative compared to conventional machining. While manufactured tungsten parts are normally composed of only one material, two-component PIM applied in this work allows the joining of two different materials, e.g., tungsten with a tungsten alloy, without brazing.

The complete technological process of two-component tungsten PIM of samples, including the subsequent heat-treatment process, is outlined. Characterization results of the finished samples, e.g., microstructure, hardness, density, and joining zone quality, are discussed.

KEYWORDS: *powder injection molding, tungsten, divertor components*

I. INTRODUCTION

The physical properties of tungsten (W) such as the high melting point at 3420°C, the high strength and thermal conductivity, the low thermal expansion, low activation and low erosion rate make this material attractive to be used for Plasma Facing Components (PFC) for future fusion power plants.[1,2]

The Karlsruhe Institute of Technology (KIT) divertor design concept of modular He-cooled finger units[3] needs for the future DEMO power plant more than 250,000 single parts for the whole divertor system aiming for a lifetime of 2 years. For the divertor parts tile and thimble tungsten and tungsten alloys are currently among by the most promising materials to withstand the high surface heat loads (up to 10 MW/m^2). But the brittleness and hardness of these materials make the fabrication by mechanical machining such as turning and milling very difficult, time and cost intensive. The development of suitable mass production methods for divertor parts was needed. A time and cost effective near-net-shape forming pro-cess

with the advantage of shape complexity, material utilization and high final density is Powder Injection Molding (PIM). This process was adapted and developed at KIT for tungsten and promising results have already been achieved.[4,5] The key steps in powder injection molding are kneading or extrusion of a suitable feedstock (combination of powder and binder), injection molding of the

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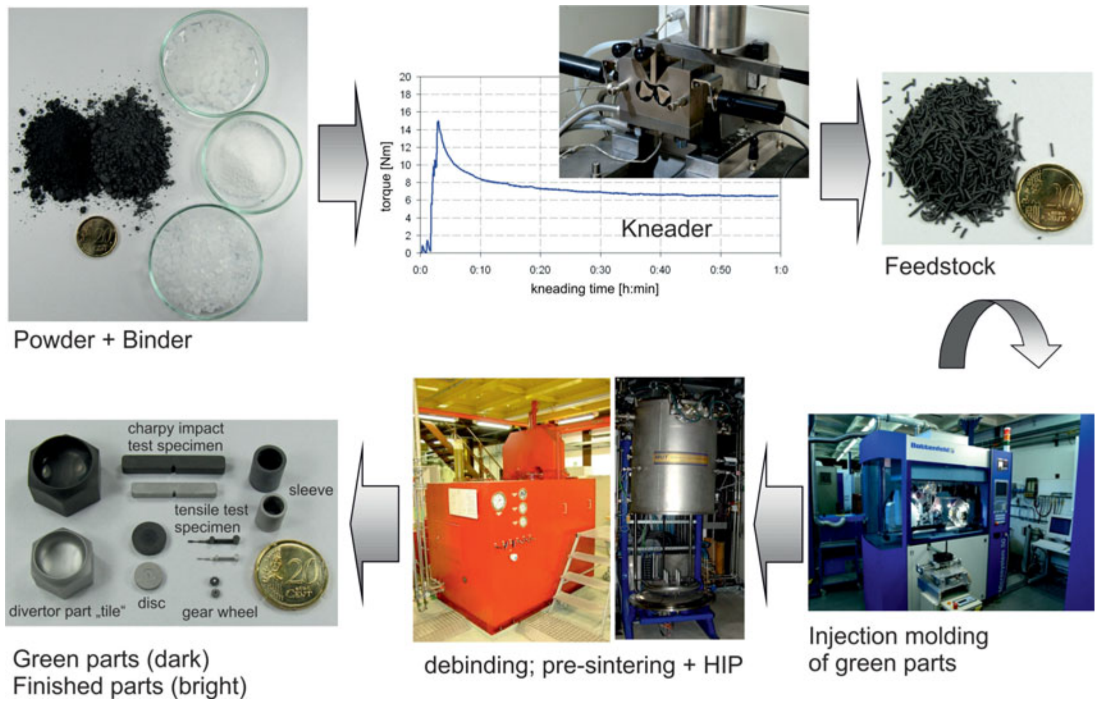

Fig. 1. The PIM process for tungsten—developed at KIT.

TABLE I

Particle Size Distribution and BET Surfaces of the Used Powders

Powder	Particle Size (μm FSSS)	D10 (μm)	D50 (μm)	D90 (μm)	BET Specific Surface (m^2/g)
Tungsten (W1)	0.70	0.14	0.47	1.25	1.27
Tungsten (W2)	1.70	0.55	1.80	4.91	0.43
Lanthanum oxide (La_2O_3)	<1.50	0.41	1.17	4.32	11.10
Yttrium oxide (Y_2O_3)	<2.50	0.35	1.58	3.67	13.91

so-called "green parts," debinding (solvent and thermal) and the heat-treatment process. The PIM process for tungsten—developed at KIT—is shown in Fig. 1.

In the course of this work two new tungsten alloy feedstocks were developed and parts were injection molded via insert PIM. After the heat-treatment process the quality of the joining zone was investigated and characterized. This special process allows the joining of two different materials without brazing thus saving cost and time.

II. EXPERIMENTAL

II.A. Materials

Two powders of pure tungsten, with an average grain size distribution in the range of 0.7 μm (W1) to 1.7 μm (W2) Fisher Sub-Sieve Size (FSSS) were used for preparation of binary tungsten powder particle systems. For pure tungsten parts mixtures of 50% W1 + 50% W2 are used while the doped tungsten alloys consist of 90% W1 + 10% W2.

The particle size of the powders for the preparation of the doped tungsten alloys was for the Lanthanum Oxide (La_2O_3) powder < 2.50 μm and for the Yttrium Oxide (Y_2O_3) powder < 1.50 μm. The characteristical data of the powders are summarized in Table I.

II.B. Powder Preparation

Two different tungsten compositions were produced by mechanical alloying each at 160 rpm for 2 hours in a planetary ball mill (Fritsch, Germany) using ZrO_2 balls and cans. For the first composition (W-2La_2O_3) the binary tungsten powder particle system (90% W1 + 10% W2) was doped with 2 wt.% La_2O_3 powder and for the second (W-2Y_2O_3) with 2 wt.% Y_2O_3.

II.C. Feedstock Preparation

The most critical point of the feedstock preparation is a homogeneous distribution of agglomerate free powder in the binder matrix. After heating the powders at 80°C for removing moisture a portion was mixed with a 50 vol.% wax/thermoplastic binder system in a kneader (Brabender, Germany) at 120°C. After that, the feedstocks were then compounded and granulated. The feedstock compositions used for injection molding were (1) pure binary W (50% W1 + 50% W2), (2) W-2La_2O_3, and (3) W-2Y_2O_3.

II.D. Producing of Parts

Injection molding was carried out on a Battenfeld Microsystem 50 injection molding machine (Battenfeld, Austria) at a feedstock temperature of 160°C and a mold temperature of 50–60°C. The first step is the injection molding of parts made of pure binary W. These are small discs with a thickness of 1 mm and a diameter of 11 mm. After that, the small tungsten disc is inserted in the cavity of the machine tool, heated up to a temperature of 80–90°C and the material composition (W-2La_2O_3 or W-2Y_2O_3) molded on it. The finished two component disc is 2 mm thick.

After injection molding and before heat-treatment the green parts have undergone solvent debinding. At first solvent debinding in n-Hexane for 48 hours at 50°C followed by thermal debinding for $\frac{1}{2}$ hour at 550°C in dry H_2 atmosphere.

The developed heat-treatment process is a two-step procedure, first pre-sintering in a MUT sinter furnace (MUT, Germany) at 1800°C in dry H_2 for 2 hours in order to reach a state where the material contains only closed porosity which is necessary for the HIP treatment. After that, the samples were compacted by use of a suitable HIP-cycle, performed by a HIP 3000 (Dieffenbacher, Germany). The samples were heated up to 2100°C under 250 MPa and argon atmosphere for 3 hours. Figure 2 shows the finished parts after heat-treatment.

II.E. Characterization Methods

Density measurements used a He-pyknometric analyzer and the Vickers-hardness was measured with a Shimadzu HMV-2000 hardness tester on the polished surface of the samples.

The metallographic analyses of the surfaces were examined with a scanning electron microscope (SEM, Zeiss SUPRA^{TM}55).

In order to interpret the quality of the joining zone of the finished parts, Auger electron spectroscopy (AES) with an Auger Nanoprobe PHI 680 (Physical Electronics, USA) was used to characterize the interface between tungsten and the tungsten alloys. Before analysis, the surface has been etched with argon ions to remove usual contaminations caused by sample preparation and handling in air.

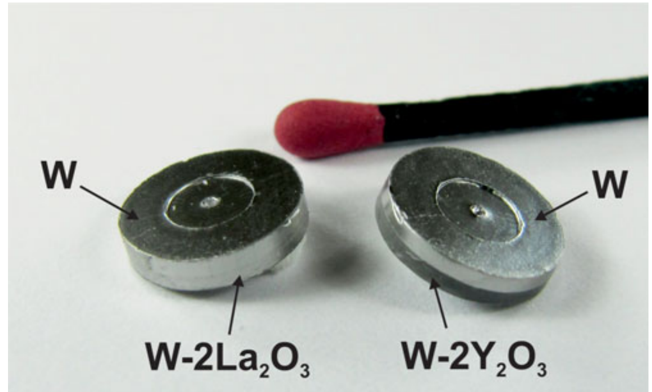

Fig. 2. 2-Component insert W-PIM: finished parts after heat-treatment.

III. PROPERTIES OF THE FINISHED PARTS

III.A. Microstructure, Density, and Hardness

Figures 3–5 show the resulting microstructure (fracture surface and metallographic section) of the finished parts after the heat-treatment process (pre-sintering and HIP).

A combination of trans- and intergranular fractures typical for tungsten was observed at the SEM images of the fracture surface for pure binary (50% W1 + 50% W2) tungsten (transgranular fracture marked by arrows in Fig. 3, left). The grain size of this material is approximately between 3 and 7 μm, the theoretical density 98.6–99% TD and the Vickers-hardness 457 HV0.1.

Figure 4 shows the result for the material composition W-2La_2O_3. The embedded spherical La_2O_3 particles in the tungsten matrix are marked by arrows. The theoretical density of this material composition is 96.5–97.2% TD and the Vickers-Hardness 586 HV0.1. Limited by the smaller initial grain size of the used binary tungsten powder (90% W1 + 10% W2), the grain size for this composition is also smaller, with an average value of 3 μm.

The grain size of the material composition W-2Y_2O_3 is also in a range of 3 μm. Figure 5 shows the embedded Y_2O_3 particles (see the marking) at the tungsten grain boundaries. The samples achieve a density of 96.3–97.1% TD and a Vickers-hardness of 617 HV0.1.

The linear shrinkage of all three materials is nearly 20% and density and hardness results for the finished parts after heat-treatment are summarized in Table II.

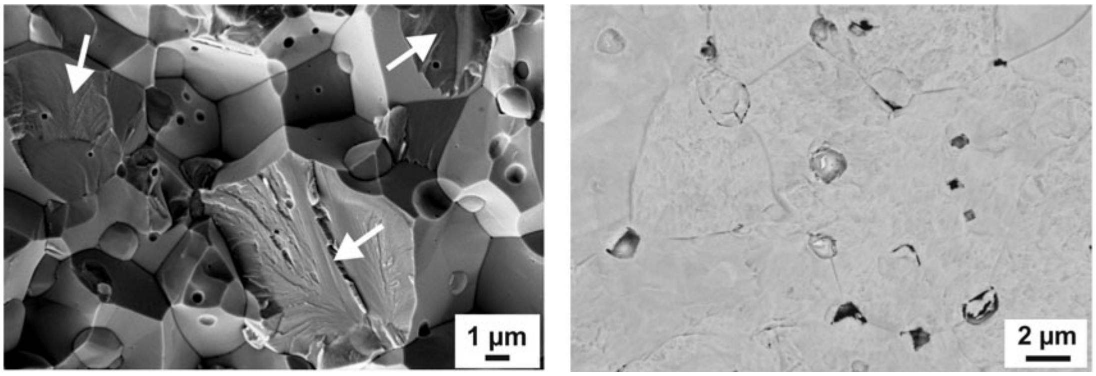

Fig. 3. SEM images of pure binary tungsten (50% W1 + 50% W2): fracture surface (left) and metallographic section (right). The areas of transgranular fracture are marked by arrows.

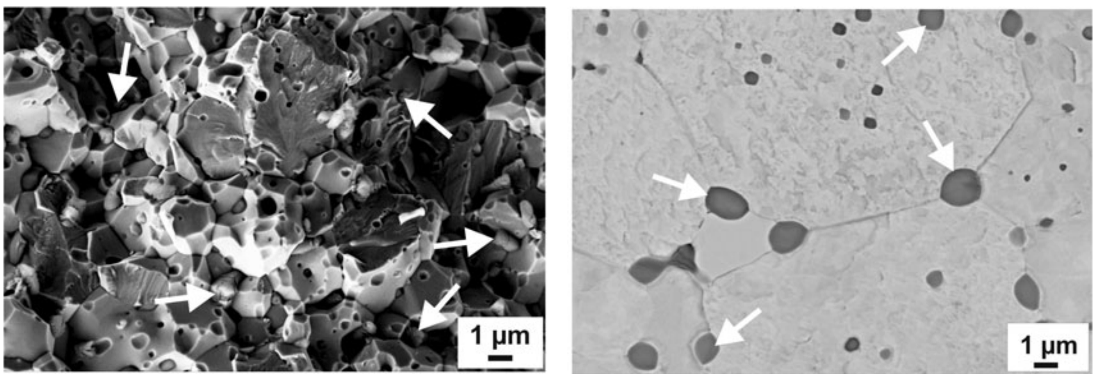

Fig. 4. SEM images of W-2La_2O_3: fracture surface (left) and metallographic section (right). A selection of the La_2O_3-particles is marked by arrows.

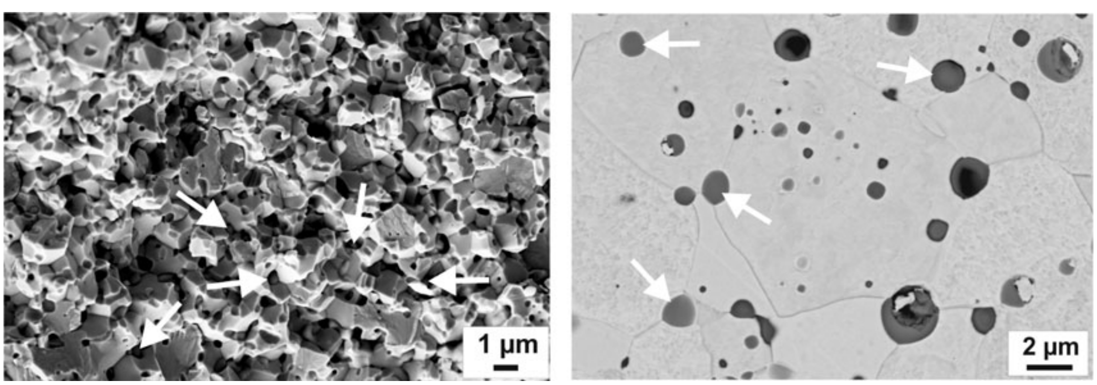

Fig. 5. SEM images of W-2Y_2O_3: fracture surface (left) and metallographic section (right). A selection of the Y_2O_3-particles is marked by arrows.

III.B. Interface Characteristics

Metallographic investigations reveal that the material connection of the insert two-component powder injection molding combinations W + W-2La_2O_3 and W + W-2Y_2O_3 are successful (see Figs. 6 and 7, left and middle). In the seam of the joining zone no cracks or gaps are visible. In order to obtain detailed information on the

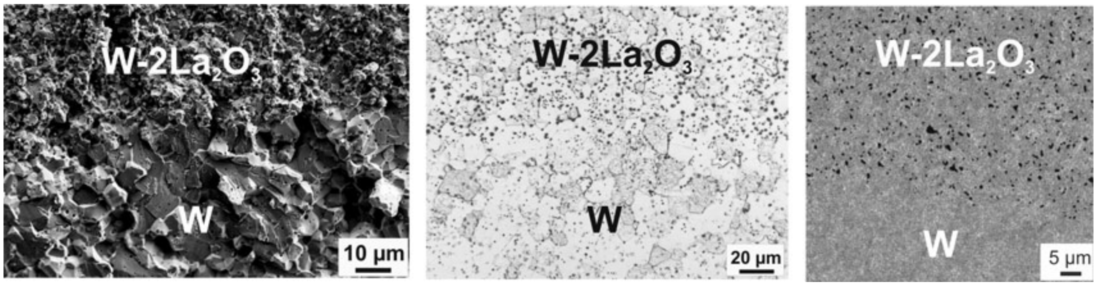

Fig. 6. Interface characteristics of W + W-2La_2O_3: SEM images of the fracture surface (left), metallographic section (middle), AES-Map (right). The W matrix is shown in gray and the color of the embedded La_2O_3 particles is black.

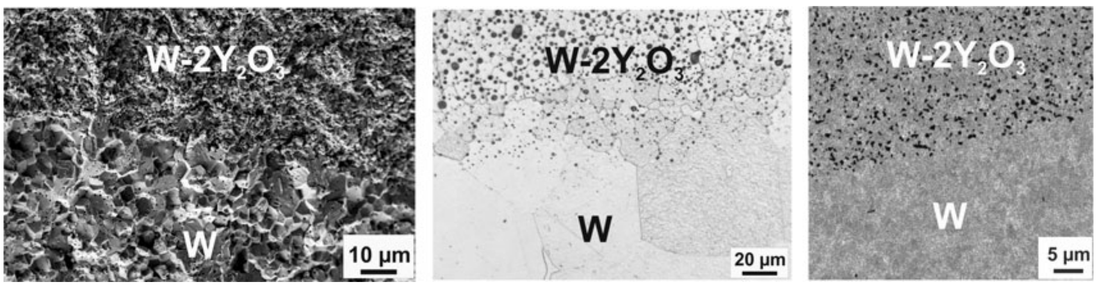

Fig. 7. Interface characteristics of W + W-2Y_2O_3: SEM images of the fracture surface (left), metallographic section (middle), AES-Map (right). The W matrix is shown in gray and the color of the embedded Y_2O_3 particles is black.

TABLE II

The Results of Density and Hardness of the Finished Parts After Heat-Treatment

Material	Theoretical Density (% TD)	Vickers Hardness (HV0.1)
Pure binary tungsten (50% W1 + 50% W2)	98.6–99.0	457
W-2La_2O_3	96.5–97.2	586
W-2Y_2O_3	96.3–97.1	617

transition from one material to the other at the interface, Auger electron spectra were recorded. Figure 6 and 7 (right) show the elemental concentration across the interface of the finished parts. The tungsten matrix is shown in gray and the color of the embedded particles (La_2O_3 respectively Y_2O_3) is black. It can be seen that a solid bond of the material interface was obtained.

IV. CONCLUSIONS AND OUTLOOK

The motivation for this work was the investigation of a joining method for two different materials by using insert two-component powder injection molding. The experience, the knowledge and the results of past pretests on conventional one component parts such as the tungsten tile was transferred to produce basic two-component parts via insert injection molding. An investigation of the joining zone was done by analyses using scanning electron microscopy (SEM) and Auger electron spectroscopy (AES). The results of the analyses, e.g. density, hardness, microstructure of the finished samples, and the quality of the joining zone, were characterized and found to be acceptable for further investigations. This is a promising outcome for manufacturing of tungsten and tungsten alloy divertor parts by the two-component powder injection molding process. The development of new material combinations processible with a new fully automatically two-component powder injection molding tool to replicate fusion relevant components such as the tungsten tile and the tungsten alloy thimble in one step without brazing will further enhance the use of mass production PIM parts for the DEMO fusion reactor. The so produced mockups will undergo high heat flux tests at the Efremov Institute, St. Petersburg, Russia, within the frame of divertor finger mock-up test program.

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