Two component tungsten powder injection molding – An effective mass production process ☆

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A B S T R A C T

Tungsten and tungsten alloys are presently considered to be the most promising materials for plasma facing components for future fusion power plants. The Karlsruhe Institute of Technology (KIT) divertor design concept for the future DEMO power plant is based on modular He cooled finger units and the development of suitable mass production methods for such parts was needed.

A time and cost effective near net shape forming process with the advantage of shape complexity, material utilization and high final density is Powder Injection Molding (PIM). This process allows also the joining of two different materials e.g. tungsten with a doped tungsten alloy, without brazing.

The complete technological process of 2 Component powder injection molding for tungsten materials and its application on producing real DEMO divertor parts, characterization results of the finished parts e.g. microstructure, hardness, density and joining zone quality are discussed in this contribution.

1. Introduction

Powder Injection Molding (PIM) is a fabrication process in powder metallurgy for shaping metals and ceramics near net shapes with reasonably tight tolerance and good surface finishes [1 3]. This process enables the mass production of low cost, high performance and complex geometries. Materials with high melting points such as tungsten or tungsten alloys could be effectively fabricated with this process.

The PIM process for tungsten, developed at Karlsruhe Institute of Technology (KIT), comprises four stages: kneading or extrusion of suitable feedstocks (combination of powder and binder), injection molding of green parts, debinding and the heat treatment process. The properties of the successfully manufactured divertor part tile, consisting only of pure tungsten, were a high density >98% T.D., a hardness of 457 HV0.1 and a microstructure without cracks or porosity [4]. Additionally, the further development of this process allows the joining of different materials via 2 Component PIM. Through the high strength and thermal conductivity, the low thermal expansion, low tritium inventory and low erosion rate, tungsten is an attractive material to be used in a wide range of applications in blanket first walls and divertor Plasma Facing Components (PFC) including plasma facing armor for future fusion power plants [5]. The development of divertor design concepts for the future DEMO power reactor at the KIT comprises the development of materials and fabrication technologies. One promising but very complex concept is based on modular He cooled finger units. More than 250,000 single parts are needed for this whole divertor system with aiming for a lifetime of 2 years [6]. The conventional fabrication of such parts by mechanical machining such as turning and milling is very difficult, time and cost intensive. However 2 Component PIM as a special process allows the suitable mass production of divertor parts and the joining of two different materials without brazing thus saving cost and time.

2. Experimental

2.1. Used powders

For the preparation of the binary tungsten powder particle systems two powders of pure tungsten, with an average grain size distribution in the range of 0.7 μm (W1) 1.7 μm (W2) Fisher Sub Sieve Size (FSSS) were used and mixtures of 50% W1 + 50% W2 compounded. For the doped tungsten alloys Lanthanum Oxide (La_2O_3) powder (FSSS < 2.50 μm) and Yttrium Oxide (Y_2O_3) powder (FSSS < 1.50 μm) are used.

2.2. Powder and feedstock preparation

For the powder preparation a planetary ball mill (Fritsch, Germany) was used and two different powder compositions were prepared. For the composition of W $2La_2O_3$, the binary tungsten powder particle system was doped with 2 wt.% La_2O_3 powder

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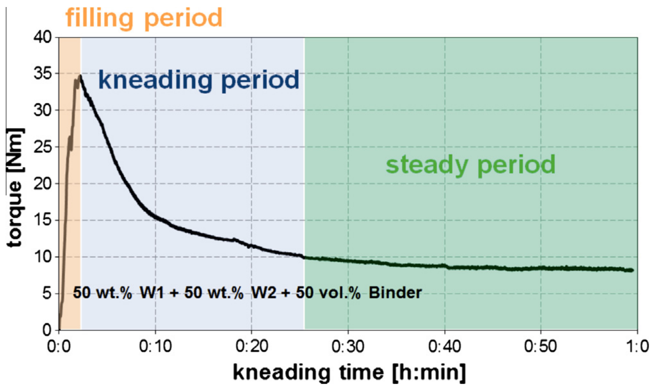

Fig. 1. The kneading curve of pure binary tungsten.

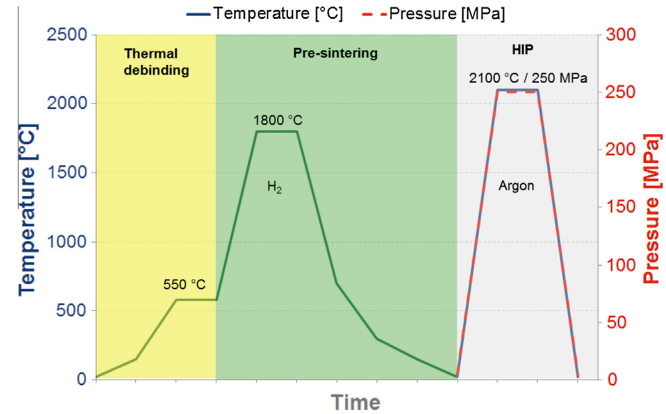

Fig. 3. The used heat-treatment process.

and for W 2Y_2O_3 with 2 wt.% Y_2O_3 powder. The details of the powder preparation are reported elsewhere [7].

After heating at 80 °C for removing moisture the powders are mixed to the so called feedstock with a 50 vol.% wax/thermoplastic binder system in a kneader (Brabender, Germany) at 120 °C. A typical kneading curve for pure binary tungsten is shown in Fig. 1. During the filling period, the torque increases through the addition of powder and binder. After the kneading period, follows the steady period with a homogenous torque the feedstock is ready for further processing. The finished granulated feedstock must be homogenous and free from agglomeration.

2.3. Producing of 2 Component PIM divertor mockups

To replicate the divertor mockup a new fully automatic PIM tool was developed (see Fig. 2). This tool allows the fabrication of the tungsten tile and the tungsten alloy thimble in one step, without additional brazing. The injection molding was carried out on an FERROMATIK MILACRON K50 injection molding machine (FERROMATIK MILACRON, Germany) at a feedstock temperature of 160 °C and a mold temperature of 50 60 °C. After injection molding the so called green parts were debinded: at first, solvent debinding in n Hexane is performed for 48 h at 50 °C followed by a thermal debinding step for 1/2 h at 550 °C in dry H_2 atmosphere. During the debinding step not only the binder and the impurities (mainly O and C) are removed, but also the high residual stresses generated during injection molding are released. The subsequent heat treatment process (see Fig. 3) consists of pre sintering in a sinter furnace (MUT, Germany) at 1800 °C in dry H_2 for 2 h to reach closed porosity (which is necessary for the HIP cycle) and compaction is achieved by a suitable HIP cycle (Dieffenbacher, Germany) up to 2100 °C under 250 MPa and argon atmosphere for 3 h. Fig. 4 shows the difference of the microstructure (fracture surface) of a pure binary tungsten part after pre sintering and as finished part (after pre sintering and HIP). The theoretical density after pre sintering is >95% T.D. and for the finished part >98% T.D. The HIP cycle generates parts with high density and low porosity. The finished 2 Component PIM divertor mockups after heat treatment are shown in Fig. 5. The shrinkage of the finished parts after the heat treatment process is nearly 20%.

2.4. Characterization methods

The metallographic analyses of the surfaces and the quality of the joining zone were examined with a scanning electron microscope (SEM, Zeiss SUPRA™55). 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. The density was measured with a He pyknometric

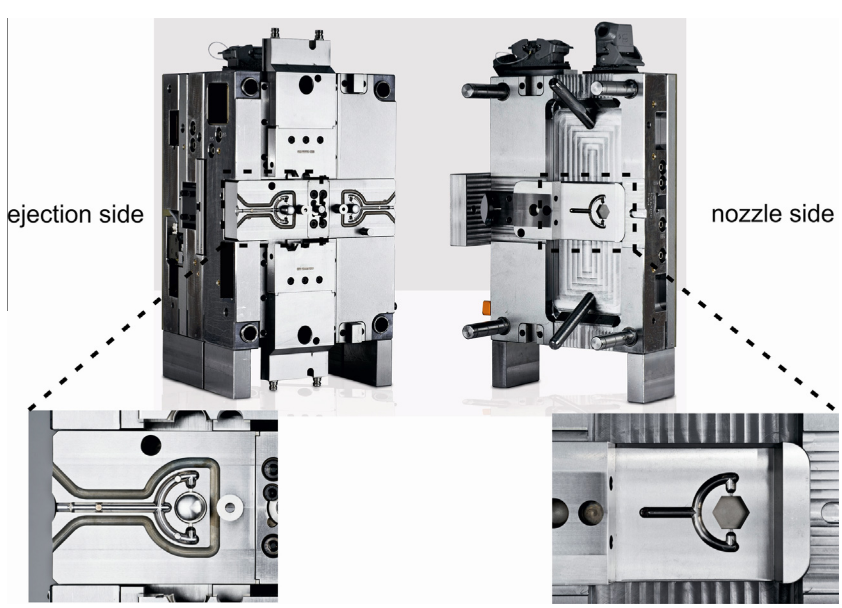

Fig. 2. The new fully automatic 2-Component PIM tool to replicate the divertor mockup.

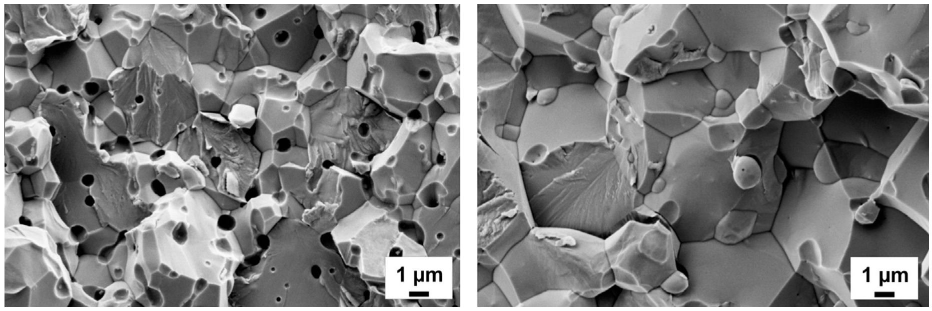

Fig. 4. SEM images of the fracture surface: only pre-sintering pure binary W (left) and pre-sintering + HIP pure binary W (right).

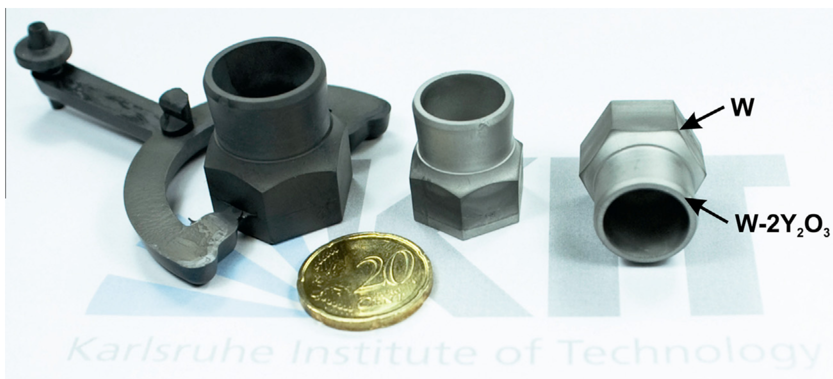

Fig. 5. Green part with gating system (left) and finished parts after heat-treatment (middle + right).

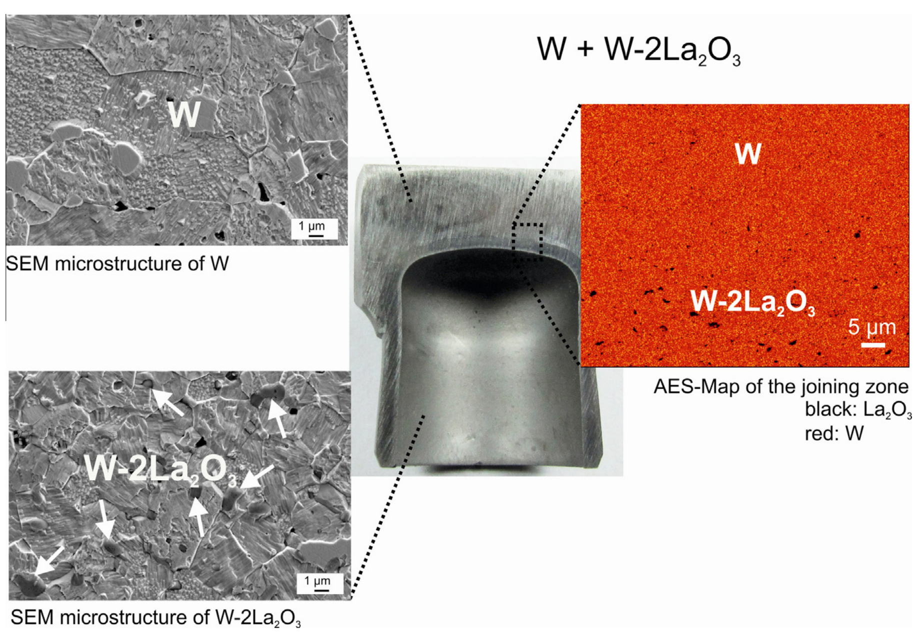

Fig. 6. Analyses of the joining zone quality via SEM and AES for the mockup combination W + W-2La_2O_3.

analyzer and for the Vickers hardness on the polished surface of the samples a Shimadzu HMV 2000 hardness tester are used.

3. Microstructure and interface characteristics of the finished 2-Component PIM divertor mockups

Metallographic investigations reveal that the material connection of the two component powder injection molding combinations W + W 2La_2O_3 and W + W 2Y_2O_3 are successful (see Figs. 6 and 7). No cracks or gaps in the seam of the joining zone between the W tile and the W alloy thimble are visible. The Auger electron measurements (Figs. 6 and 7, right) show the elemental concentration of tungsten across the interface of the finished parts. In both figures, the color of the W matrix is shown in red and the color of the embedded particles (La_2O_3 respectively Y_2O_3) is black. For both material combinations a solid bond of the material interface was achieved.

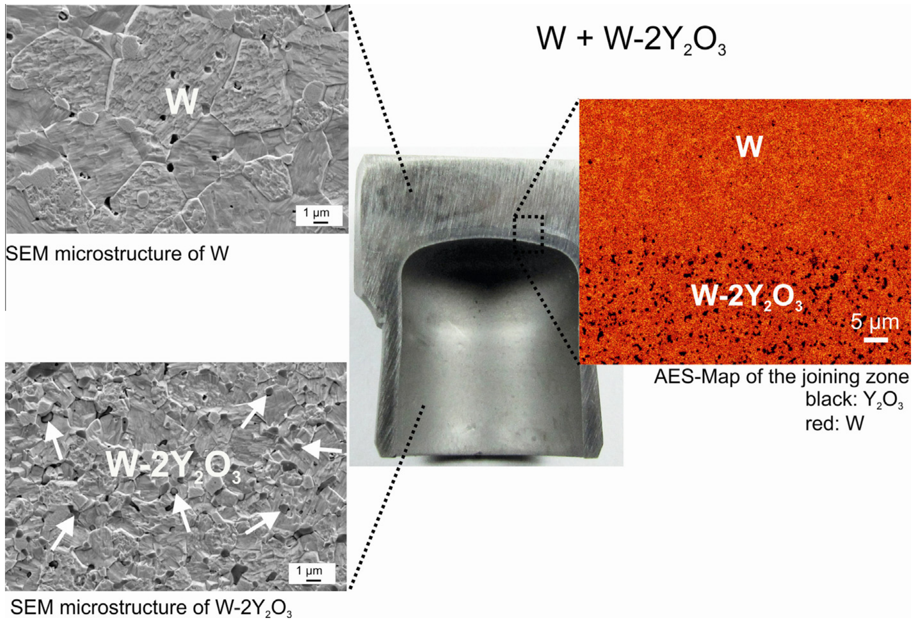

Fig. 7. Analyses of the joining zone quality via SEM and AES for the mockup combination W + $W2Y_2O_3$.

Table 1
Results of density, hardness and grain size of the finished parts after heat-treatment.

Material	Theoretical density (% TD)	Vickers-hardness (HV0.1)	Grain size (µm)
W	98.6–99.0	457	5–7
W-$2La_2O_3$	96.5–97.2	586	>3
W-$2Y_2O_3$	96.3–97.1	617	<3

The resulting microstructure (metallographic section) of the finished mockups is also shown. The embedded spherical particles (La_2O_3 respectively Y_2O_3) in the tungsten matrix are marked by white arrows. Pure PIM tungsten parts achieve a density of 98.6 99% T.D., a Vickers hardness of 457 HV0.1 and a grain size between 5 and 7 µm. The grain size for the material composition W $2La_2O_3$ is smaller, with an average value of 3 µm and the relative density is over 97% T.D. and the Vickers hardness 586 HV0.1. Once again is the grain size smaller for W $2Y_2O_3$ with values below 3 µm. These composition achieves also a density of over 97% T.D. but the Vickers hardness is 617 HV0.1 and still higher than for W $2La_2O_3$. Density, hardness and grain size results for the finished parts after heat treatment are summarized in Table 1. For both doped materials the embedded particles act as grain growth inhib itor and generate a small grain size in comparison to pure PIM tungsten. The effect of the grain boundary strengthening is also seen in the results of the Vickers hardness. These results are very significant for W $2Y_2O_3$.

4. Conclusions and outlook

The motivation for this work was the investigation of a suitable manufacturing method for divertor components consisting of dif ferent tungsten materials. Based on the previous results of the tungsten PIM divertor part tile, the experience and the knowledge of the material development and pretests of basic two component PIM parts was transferred to produce real divertor components via powder injection molding. A newly developed fully automatic two component powder injection molding tool allows the replication of fusion relevant components such as the tungsten tile and the tung sten alloy thimble in one step without additional brazing. The microstructure of the finished samples, and the quality of the joining zone, were characterized and found to be worthwhile for further investigations. This is a promising outcome to further en hance the use of mass production PIM parts for the DEMO fusion reactor.

Further steps will be the investigation of material properties by mechanical characterization and via high heat flux tests.

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