One- and Two-Component Tungsten Powder Injection Molding for Manufacturing Fusion Reactor Devices

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Abstract

Future fusion power plants will require a large number of complex shaped tungsten components. Due to its high economic efficiency powder injection molding (WPIM) represents a promising option for this purpose. At KIT a special process sequence based on compaction by both pre-sintering and hot isostatic pressing has been developed. Examples for reactor parts already produced as well as metallographic analysis concerning grain growth and density will be presented.

Beside pure tungsten, a wide range of tungsten alloys are of special interest for fusion applications, therefore, feedstocks containing W-La_2O_3 or W-Y_2O_3, respectively, have been tested.

As further improvement two-component W-PIM offers the possibility to manufacture integrated parts without additional mounting steps. Combinations of pure tungsten plus tungsten alloy or of different tungsten alloys have been realized showing nearly defect-free interfaces even after sintering. Examples for such Divertor pieces consisting of pure tungsten and tungsten alloys are the so-called “tile” and “thimble” components.

Keywords: Tungsten powder injection molding, two-component injection molding, tungsten alloys, fusion technology

1. Introduction

Powder Injection Molding (PIM) represents a fabrication process for near-net manufacturing of metal and ceramic devices with reasonable tight tolerances and good surface qualities [1, 2, 3, 4]. In case of mass production it enables low cost, high performance products of complex geometries. Additionally, the further development of this manufacturing technology allows for the joining of different materials via 2-Component PIM (2C-WPIM) [5, 6]. Concerning applicable materials PIM reveals the decisive advantage of being suitable for processing of materials with high melting points such as tungsten or tungsten alloys quite effectively [7].

The PIM procedure for tungsten (WPIM) as developed at Karlsruhe Institute of Technology (KIT) comprises five main stages: kneading or extrusion of suitable feedstocks (= mixture of powder and binder), injection molding of green parts, debinding and the heat-treatment process consisting of a pre-sintering and a HIP step, see Fig. 1.

Applying this procedure Divertor components consisting of pure tungsten only - the so-called “tiles” - could be manufactured successfully with a high density of >98% T.D., a hardness of 457 HV0.1 and a microstructure with nearly no cracks or porosity [8].

Due to 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). The latter includes plasma facing armor for future fusion power plants [9]. Therefore, development of Divertor concepts for the future DEMO power reactor comprises the development of materials and related fabrication technologies at KIT in parallel. One promising but quite complex Divertor concept is based on modular He-cooled finger units.

More than 250,000 of such single parts are needed to equip one entire Divertor system aiming for a lifetime of 2 years in minimum. The conventional fabrication of such parts by mechanical machining such as turning and milling would be very difficult, time and cost intensive. In contrast, 2-Component PIM (2C-WPIM) as a special variant allows the effective mass production of Divertor parts and the joining of two different materials without additional joining steps thus considerably saving of costs and time.

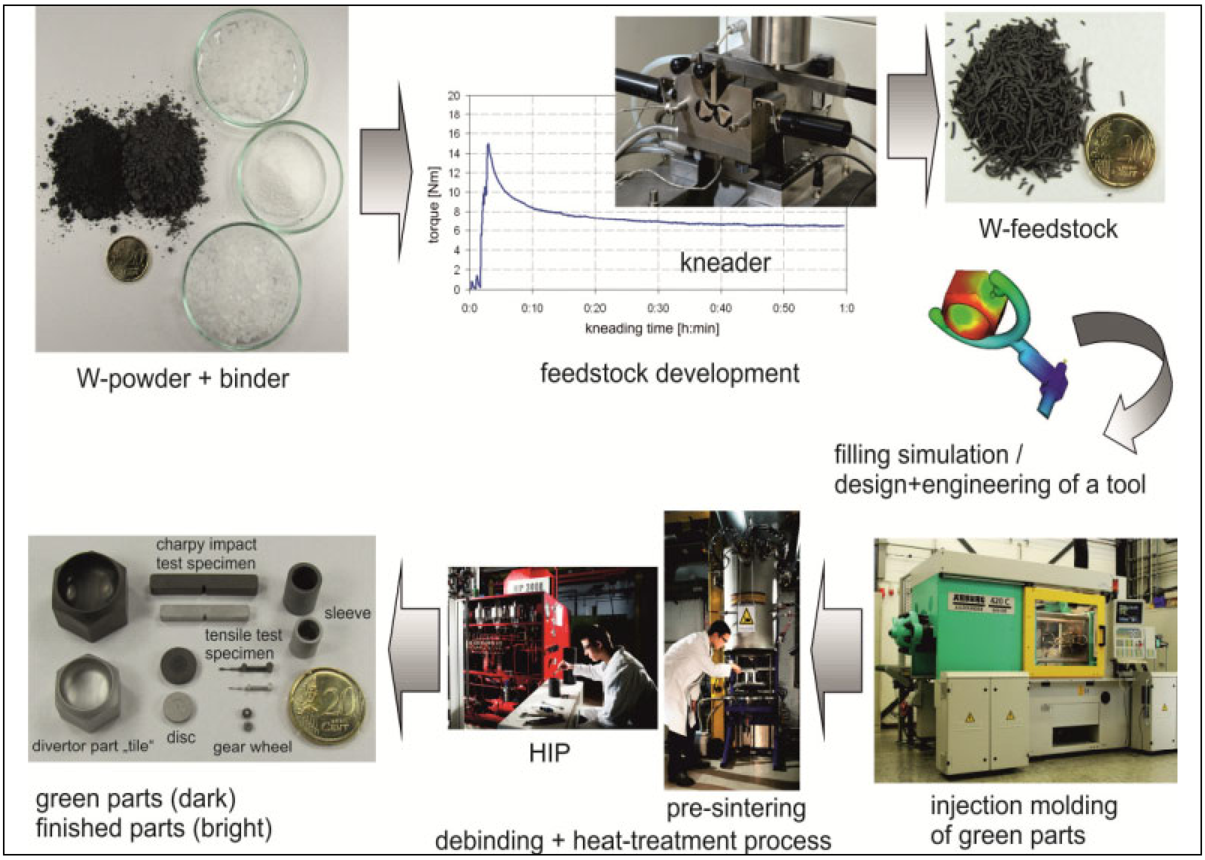

Fig. 1: Scheme of the W-PIM process as developed at KIT

2. Experimental

2.1. Powders used

Former R+D activities showed that a binary tungsten powder particle system favors sintering activity and microstructure density. To prepare such binary systems 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 and mixtures of 50% W1 + 50% W2 were 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) were used.

2.2. Powder and feedstock preparation

Two different powder compositions were prepared applying a planetary ball mill (Fritsch, Germany). The binary tungsten powder particle system was doped with 2wt.-% La_2O_3 (in the following called W-2La_2O_3) or with 2wt.-% Y_2O_3 powder (for W-2Y_2O_3), respectively. More details concerning the powder preparation and composition are reported, for example, in [10].

After heating at 80℃ to remove moisture the powders were mixed to the feedstock containing a 50 vol.-% wax/thermoplastic binder system in a kneader (Brabender, Germany) at 120℃. The kneading curve showed - after the torque increased during the filling period - a steady period with a nearly constant torque. This allows the conclusion that the mixture was mostly homogenous and free from agglomeration, i.e. the feedstock was ready for further processing.

2.3. Producing of 2C-PIM Divertor components

For successful replication of the Divertor components a new fully automatically running PIM tool had been developed (Fig. 2). The probably most remarkable feature of the new tool is that it allows for the fabrication of the tungsten tile and the tungsten alloy thimble in one cycle, without additional brazing steps. The injection molding trials were performed on a two-component K50 S2F 50t injection molding machine (Ferromatik Milacron, Germany) utilizing a feedstock temperature of 160℃ and a mold temperature of 50-60℃.

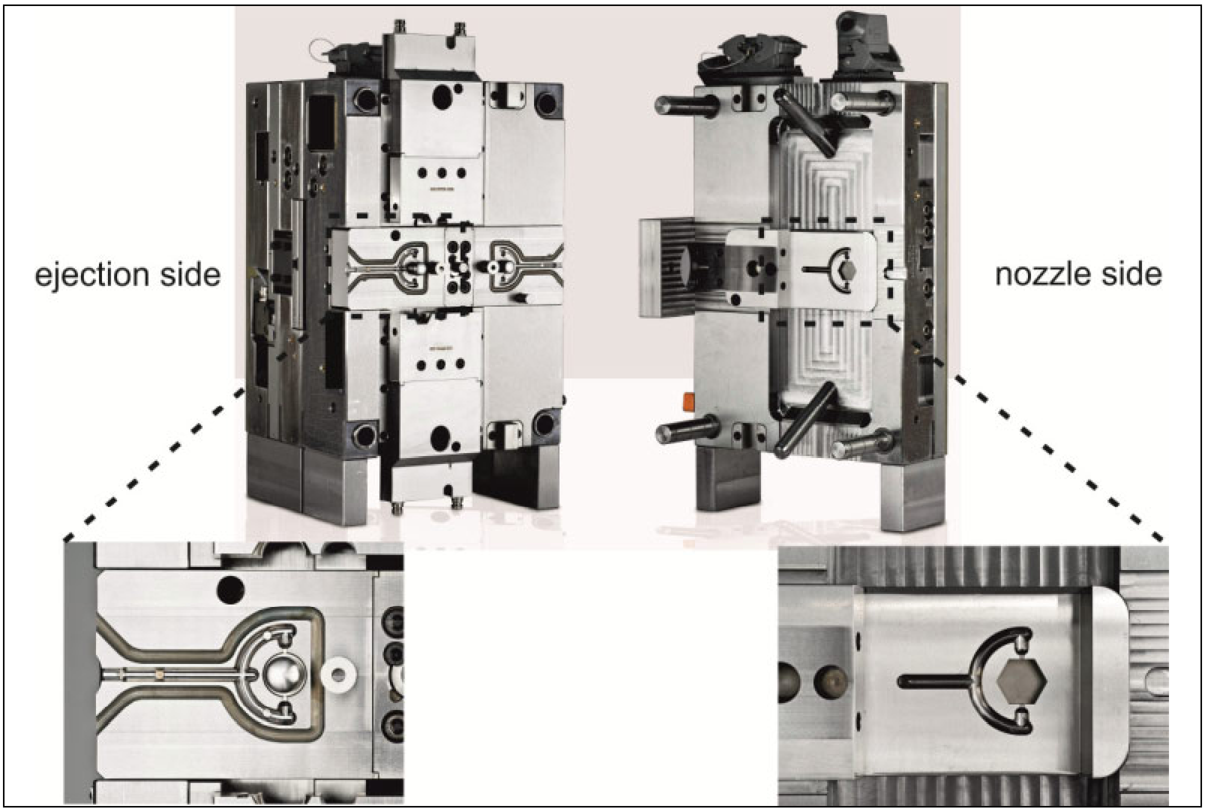

Fig. 2: The new fully automatic 2-Component PIM tool for injection molding of the Divertor components. It is equipped with two cavities, one for the “thimble” (bottom left) and one for the “tile” (bottom right).

Subsequently green parts were debinded: at first, solvent debinding was carried out for 48 hours at 50°C in n-Hexane followed by a thermal debinding step lasting 0.5 hours at 550°C in dry hydrogen atmosphere. Especially during the last debinding step not only the binder and the impurities (mainly oxygen and carbon) were extracted, furthermore also the high residual stresses generated during injection molding could be released up to a certain extent (Fig. 3, left). The following heat-treatment process (Fig. 1) consisted of a pre-sintering step in a sinter furnace (MUT, Germany) for 2 hours at 1800°C in dry hydrogen to reach a closed porosity (which is essential for the HIP-cycle) whereas final compaction was accomplished by a suitable HIP-cycle lasting for 3 hours up to 2100°C under a maximum pressure of 250MPa in argon atmosphere. The theoretical densities were >95% T.D. after pre-sintering and >98% T.D. for the final part. The HIP procedure led to parts with high density and low porosity. The finished 2-Component PIM Divertor components after heat-treatment are shown in Fig. 3. The shrinkage of the finished parts after the heat-treatment processes is nearly 20%.

Fig. 3: Divertor components produced by WPIM, here “thimble” and “tile” as one two-component part. Green bodies (left) and after heat treatment (right). The shrinkage during sintering is clearly visible.

2.4. Characterization methods

The metallographic analyses of the surfaces and the characterization of the junction area were mainly carried out using a scanning electron microscope (SEM, Zeiss SUPRATM55). The densities were measured with a He-pycnometric analyzer and for the Vickers-hardness on the polished surface of the samples a Shimadzu HMV-2000 hardness tester was applied.

3. Microstructure and interface characteristics of the final 2C-PIM Divertor components

Metallographic examination revealed that the material connection of the two-component powder injection molding combinations (W + W-2La_2O_3 and W + W-2Y_2O_3) were performed successfully. No cracks or cavities could be detected in the seam of the joining zone between the W tile and the W-alloy thimble, i.e. for both material combinations a solid bond of the material interface was achieved (Fig. 4).

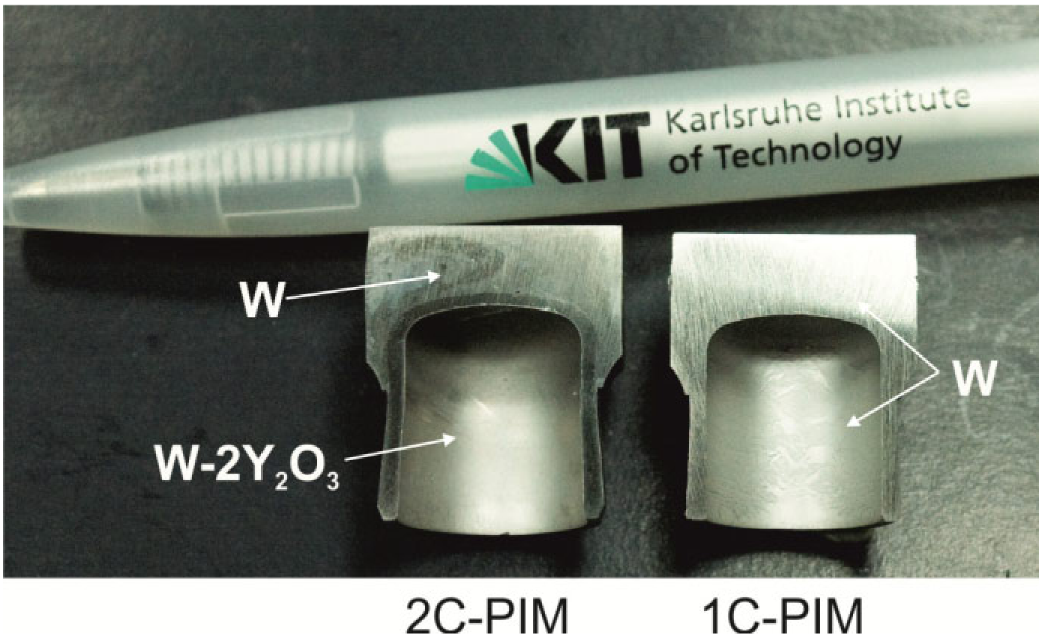

Fig. 4: Cut views and comparison of “thimble+tile” combinations, one made by two-component PIM (left) and the other made by one-component PIM (right). Besides the fact that the 2C-sample consists of two different materials it has to be mentioned that the boundary line is still visible, however, no cracks or pores can be detected.

The resulting microstructure (metallographic section) of the finished samples showed homogenously embedded spherical particles (La_2O_3 or Y_2O_3, respectively) in the tungsten matrix. The pure PIM tungsten parts achieved densities of 98.6 - 99% T.D., a Vickers-hardness of 457 HV0.1 and a grain size between 5 and 7µm. The grain size for the material composition W-2La_2O_3, however, was smaller, with an average value of 3µm and the relative density was higher than 97% T.D. In contrast the grain size for W-2Y_2O_3 was smaller reaching values below 3 µm. These composition achieved also a density of more than 97% T.D. but the Vickers-hardness of 617 HV0.1 is significantly higher than for W-2La_2O_3.

Table 1: Values for density, hardness and grain size of the final 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

It can be assumed that for both doped materials the embedded particles act as grain growth inhibitors and generate a smaller grain size compared to pure PIM tungsten. The effect of the grain boundary strengthening can also be regarded as the reason for the increased Vickers-hardness values. These interdependencies are mostly significant in case of W-2Y_2O_3.

4. Summary and Outlook

The main objective of this work was the investigation of an effective manufacturing process conduct for Divertor components consisting of different tungsten materials. The experience and the knowledge of the material development gained by pretests of basic two-component PIM parts had been transferred to produce real Divertor components via powder injection molding. A newly designed fully automatic two-component powder injection molding tool allowed for the replication of fusion relevant components such as the tungsten tile and the tungsten alloy thimble in one step without additional brazing. The microstructure of the finished samples and the quality of the joining areas were characterized and turned out to be encouraging for further R+D activities. This can be regarded as a promising basis to further enhance the use of mass production PIM parts for the DEMO fusion reactor.

The samples produced by 2C-WPIM will undergo high heat flux tests at the Efremov Institute, St. Petersburg, Russia, within the frame of the EFDA Divertor finger mock-up test program.

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References

[1] R.M. German: Metal Injection Molding; Metal Powder Industries Fed., Princeton N.J., ISBN 978-0-9819496-6-6, 2011.

[2] V. Piotter, “A review of the current status of MicroPIM”,
Part 1: Materials, processing, microspecific considerations and applications, Powder Injection Molding, Vol. 5, No. 3, Inovar Communications Ltd. (2011), pp. 27-36.
Part 2: Screw injection units, simulation and process variants, Powder Injection Molding, Vol. 5, No. 4, Inovar Communications Ltd. (2011), pp. 25-30.

[3] K. Nishiyabu, I. Andrews, S. Tanaka, “Accuracy evaluation of ultra-compact gears manufactured by the microMIM process”, Powder Injection Molding International, Vol. 2 No. 4; Inovar Communications Ltd., (2008), pp. 60-63.

[4] F. Petzoldt, “Micro powder injection Molding – challenges and opportunities”, Powder Injection Molding International, Vol. 2, No. 1, Inovar Communications Ltd., (2008), pp. 37-42.

[5] V. Piotter, J. Prokop, H.-J. Ritzhaupt-Kleissl, A. Ruh, J. Haußelt, “Multi-component micro injection molding - trends and developments”, International Journal of Advanced Manufacturing Technology, 47, (2010), pp. 63-71.

[6] F. Petzoldt, “Multifunctional parts by two-component Powder Injection Molding (2C-PIM)”, Powder Injection Molding International, Vol. 4, No. 1, (2010), pp. 21-27.

[7] V. Piotter, B. Zeep, P. Norajitra, R. Ruprecht, A. von der Weth, J. Hausselt, “Development of a powder metallurgy process for tungsten components”, Journal of Fusion Engineering and Design 83, (2008), pp. 1517-1520.

[8] S. Antusch, P. Norajitra, V. Piotter, H.-J. Ritzhaupt-Kleissl, L. Spatafora, “Powder Injection Molding – An innovative manufacturing method for He-cooled DEMO Divertor components”, Fusion Engineering and Design 86, (2011), pp. 1575-1578.

[9] M. Rieth, et al., “Review on the EFDA programme on tungsten materials technology and science”, Journal of Nuclear Materials 417 (2011), pp. 463-467.

[10] S. Antusch, M. Müller, P. Norajitra, G. Pintsuk, V. Piotter, H.-J. Ritzhaupt-Kleissl, T. Weingärtner, “Two component tungsten powder injection molding for mass production of He-cooled DEMO Divertor parts”, Fusion Science and Technology, (2012), submitted.