

PART QUALITY, SUSTAINABILITY, AND PERFORMANCE

A COMPARISON BETWEEN GRAPHITE AND METALLIC LINED MIM FURNACES

Timm OHNWEILER¹, Maximilian MUNGENAST¹, Fabian UHLEMANN-KÖLLY¹,
Tetsuro OHBA² and Katsuyoshi SHIMAMURA²

¹Carbolite Gero GmbH & Co KG, Neuhausen, Hesselbachstr. 15, Baden-Württemberg, 75242 Neuhausen, Germany

²Verder Scientific Co., Ltd., 10F Shimomoto Building, 46-3, Hatsudai 1-chome, Shibuya-ku, Tokyo 151-0061, Japan

LEADING HEAT TECHNOLOGY



ENABLING PROGRESS.

HEAT TREATMENT
ELEMENTAL ANALYSIS
MATERIALOGRAPHY & HARDNESS TESTING
MILLING & SIEVING
PARTICLE CHARACTERIZATION
PHARMACEUTICAL TESTING

Under the roof of VERDER SCIENTIFIC we support thousands of customers worldwide in realizing the ambition we share. As their technology partner behind the scenes, we deliver the solutions they need to make progress and to improve the everyday lives of countless people. Together, we make the world a healthier, safer and more sustainable place.

TABLE OF CONTENT

1	ABSTRACT.....	4
2	INTRODUCTION.....	4
3	EXPERIMENT.....	4
4	RESULTS.....	5
4.1	QUALITY AND RESOURCE CONSUMPTION.....	9
5	DISCUSSION AND CONCLUSION.....	10
6	REFERENCES.....	10

1 ABSTRACT

In Additive Manufacturing (AM) and Metal Injection Moulding (MIM) two different types of batch furnaces are in use: Either furnaces with a heating cassette made from graphite or from metallic materials such as tungsten or molybdenum. Metallic furnaces offer the best possible purity of the atmosphere, such as Argon, Nitrogen, Hydrogen or (high) vacuum. The running costs and the energy consumption are comparably high. Graphite furnaces result in a reduced energy consumption. However, those furnaces inevitably result in carbon particulates being present in the furnace atmosphere during heat treatment. This results in a coloration of the surface of metallic parts, but also in modified carbon content. This study reports on the parts quality and on the energy and resource consumption for both kinds of furnace for a typical batch type production process. The primary outcome achieved through the utilization of metallic lined Metal Injection Moulding (MIM) furnaces is that highest densities of 7.9 g/cm³ are achieved when using Hydrogen and a significant reduction in carbon content by 50 %. This advancement, however, is accompanied by a 30 % increase in the product's carbon footprint.

Keywords: Additive Manufacturing, Metal Injection Moulding, Quality, Sustainability, Furnaces

2 INTRODUCTION

316LG+[®] is a widely used feedstock for manufacturing parts by Metal Injection Moulding (MIM). This feedstock is used for the injection moulding machine to manufacture a total of 2880 jets (compare Fig. 2.), which is just enough to feed four times the debinding and the sintering furnace: In a typical industrial batch type MIM process, a total of 720 jets (compare Fig. 2.) can be processed during one cycle. After Injection Moulding, the parts are stacked on a typical sinter stack, which is then transferred to the catalytic debinding furnace. A typical processing time of 8 h at 120 °C in a mixture of evaporated nitric acid and Nitrogen gas is required to remove the main binder from the green parts. After this process, the so called brown parts are available. Brown parts still contain the rest binder, which must be removed thermally. The complete sinter stack is now transferred to the (rest-) debinding and sintering furnace with a typical processing time of 22 h. In graphite lined furnaces debinding and sintering can be performed only under Nitrogen or Argon, whereas a metallic lined furnace offers the possibility to use Hydrogen in partial pressure or atmospheric pressure over the full temperature range.

3 EXPERIMENT

Rest debinding and sintering is performed multiple times to achieve samples which are treated under a total of four different conditions: For the first set of samples, 720 jets are heat treated in a graphite lined MIM furnace. Debinding is performed under fine vacuum conditions and sintering is performed under 500 mbar of Argon partial pressure with a sintering temperature of 1380 °C. For the second, third and fourth set of samples, further heat treatments are performed in a metallic lined MIM furnace of similar capacity: A process which is totally identical to the heat treatment in the graphite lined furnace, i.e. fine vacuum debinding and sintering under 500 mbar Argon partial pressure with a sintering temperature of 1380 °C. As a metallic lined furnace offers the possibility of full Hydrogen operation additional treatments are performed: For the third set of samples, debinding is performed under 500 mbar partial pressure with a mixture of Hydrogen and Nitrogen and sintering is performed under atmospheric Hydrogen partial pressure up to 1380 °C. For the fourth set of samples, debinding is performed under 950 mbar partial pressure with a mixture of Hydrogen and Nitrogen and sintering is performed under atmospheric Hydrogen partial pressure up to 1380 °C.

All the machines, injection moulding machine, catalytic debinding- and the sintering furnace enable full data logging of all the resources and energy consumptions. The data files can be collected for later evaluation. This data is used to calculate the Product Carbon Footprint (PCF) utilizing the software Umberto LCA+ which offers the possibility of a life cycle assessment according to ISO 14040 and 14044. It accesses the Ecoinvent 3.9.1 database which provides the values of the Global Warming Potential (GWP). However all the inputs and outputs must be defined precisely.

Some selected parts undergo a quality control procedure: For elemental characterization optical emission spectroscopy (OES) is applied. To determine the density a gas pycnometer is used. Additionally one jet from the graphite lined furnace and one part out of the metallic lined furnace, which is debinded under 500 mbar with a mixture of Hydrogen and Nitrogen and sintered in atmospheric pressure under pure Hydrogen, is selected. Both parts are studied with a Scanning Electron Microscope (SEM) for surface analysis and using Energy Dispersive X-ray (EDX) analysis to determine the elemental composition locally. The same microscope is equipped with a Focussed (Gallium-) Ion Beam (FIB), enabling to prepare a cross section of both parts for further SEM/EDX analysis of the cross section.

4 RESULTS

The density in the graphite lined furnace results in the lowest density of all sintered parts. The metallic furnace results in increased densities. Vacuum debinding and Argon partial pressure sintering results already in a slightly increased density. Further improvements of the density is achieved by using Hydrogen during both, debinding and sintering up to almost the full density of 7.9 g/cm³ (compare table 1 and Fig. 1). The Carbon content in the graphite lined furnace results in the highest value of all runs. Although the Carbon content is still within the specifications of the data sheet of 316LG⁺, optimum values must be clearly below 0.03 %. When using the metallic lined furnace, the Carbon content reduces by 50 %, which further results in improved corrosion resistance of those parts. The corrosion of the parts can be seen visually.

For the FIB/SEM analysis only the parts treated in the graphite lined furnace under the conditions of the run "V4" and the parts treated in the metallic lined furnace under the conditions of the run "V1" are used. Please compare Fig. 1. This selection is made, since "V1" is a typical adjustment in the industry when performing rest debinding and sintering of MIM parts. However, further improvements are possible by a variation of the individual parameters (compare Fig. 1). A visual inspection of the samples already shows a significant brighter color of all parts from the metallic furnace (compare Fig. 2). This becomes further evident, when the surface is inspected by the SEM. The surface of the parts sintered in the graphite lined furnace is somehow covered by an unregular structure (compare Fig. 3). Fig. 5., Fig. 6., Fig. 7. and Fig. 8. show a cross section of both parts. Cutting the cross section is achieved by tilting the sample and cutting the samples with the FIB. Using a Backscattered Electron (BsE) detector for the investigation of the cross section clearly shows, that a graphite lined furnace results in a dark layer on the surface, see Fig. 5. This layer has a maximum thickness of 160 nm. As it occurs dark, it must consist of light weight elements. The surface of the part treated in the metallic lined furnace shows a smooth surface without any coating by other materials (compare Fig. 4.). For elemental analysis of the surface EDX analysis was performed. A comparison between the surface of the parts from the graphite lined furnace with the parts from the metallic lined furnace shows an increased Carbon and Oxygen content as well as Aluminum contaminations on the surface of the parts out of the graphite lined furnace. Further, Aluminum contaminations can only be detected on the surface of the parts out of the graphite lined furnace. The metallic furnace does not show such kinds of contaminations and results in a further improved part quality (compare Fig. 9.).

To calculate the energy and resource consumption, the following steps are undertaken: As the raw material is 316LG⁺ the data sheet is available. 720 parts, equal 35 kg of feedstock, which equals 354 kg of CO₂ equivalent which goes in the injection moulding machine. For the injection moulding step the inputs are the raw material, the transport of the raw material to the injection moulding machine, which equals a distance of 154 km in this case. Using the injection moulding machine requires hydraulic oil. As this is replaced only every five years, the CO₂ equivalent is negligible. The data logging of the injection moulding machine allows to calculate the total energy consumption to mould all 720 parts, which equals 47 kWh. As output the green parts are available, ready for catalytic debinding and sintering. Both furnaces offer the possibility of full data logging, hence resource and energy consumptions are available. Catalytic debinding is an 8 h process which consumes, 30 kg Nitrogen, 2 kg nitric acid, 4 kg Propane gas for the combustions of the byproducts, 41 m³ of compressed air and a total of 274 kWh current consumption. The outputs are 4 kg CO₂, 2 kg of water and 9 kg of Formaldehyde which is then combusted. The brown parts are now transferred to the metallic sintering furnace, which requires 42 kg of Nitrogen, 3 kg of Hydrogen, 89 m³ of compressed air, 36 kg of Propane gas, 3 kg of vacuum pump oil and a total current consumption of 1770 kWh. The outputs consist of 10 kg CO₂ and 4 kg of water. Brown parts are also transferred to the graphite lined sintering furnace, which requires 42 kg of Nitrogen, no Hydrogen, since it cannot be used in a graphite lined furnace due to reactions at elevated temperatures, 89 m³ of compressed air, 3 kg of vacuum pump oil and a total current consumption of 962 kWh. The outputs consist of 4 kg water. Both sintering furnaces require a total of 22 h for the full process. All the individual inputs and outputs are translated in the CO₂ equivalent. It results in the figures given in table 1.

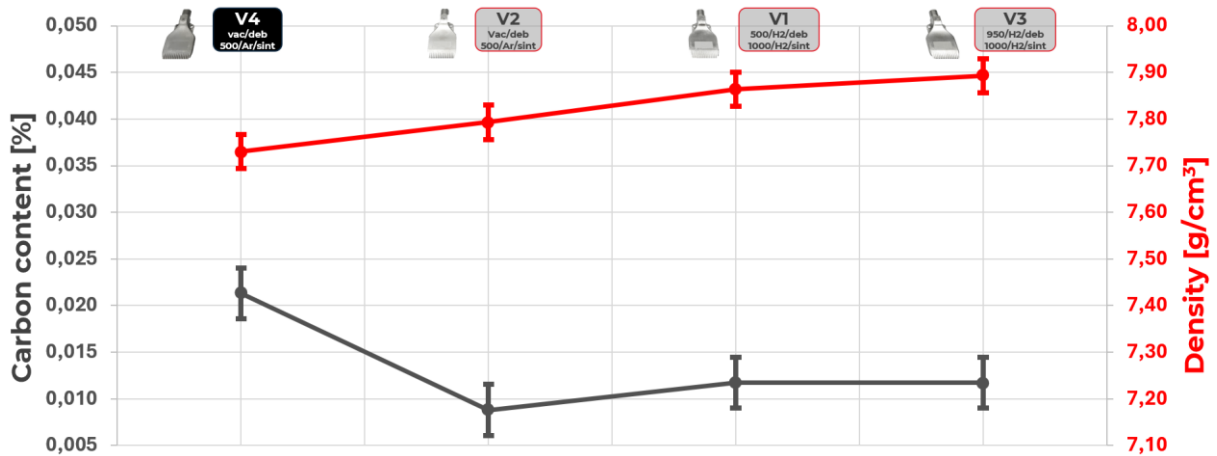


Fig. 1. Carbon content and final density of parts sintered in a graphite- and metallic lined furnace under various conditions:

First set of samples (V4): 720 parts treated in the graphite lined furnace. Debinding is performed in full vacuum, sintering is performed under 500 mbar Argon partial pressure up to 1380°C.

Second set of samples (V2): 720 parts treated in the metallic lined furnace. Debinding is performed in full vacuum, sintering is performed under 500 mbar Argon partial pressure up to 1380 °C.

Third set of samples (V1): 720 parts treated in the metallic lined furnace. Debinding is performed under 500 mbar partial pressure with a mixture of Hydrogen and Nitrogen, sintering is performed under atmospheric Hydrogen partial pressure up to 1380°C.

Fourth set of samples (V3): 720 parts treated in the metallic lined furnace. Debinding is performed under 950 mbar partial pressure with a mixture of Hydrogen and Nitrogen, sintering is performed under atmospheric Hydrogen partial pressure up to 1380 °C.

Due to the data sheet 316LG+® must result in a Carbon content below 0.03 % and in a density of ideally 7.9 g/cm³. The results achieved in the metallic lined MIM furnace are clearly superior compared to parts treated in a graphite lined furnace. The possibility to select Hydrogen further improves the results.



Fig. 2. The left part is heat treated in the graphite lined furnace. The other parts are treated in the metallic lined furnace.

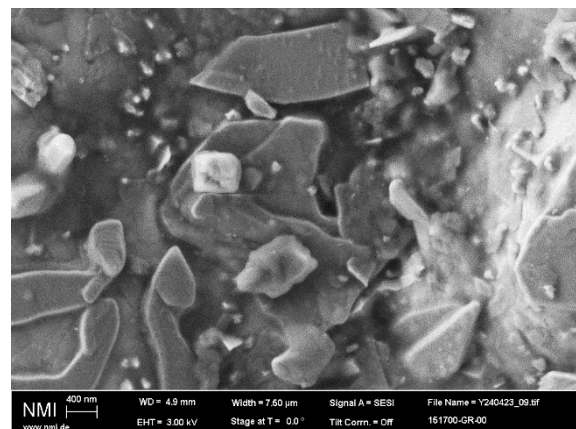


Fig. 3. SEM picture of the part from the graphite lined furnace.

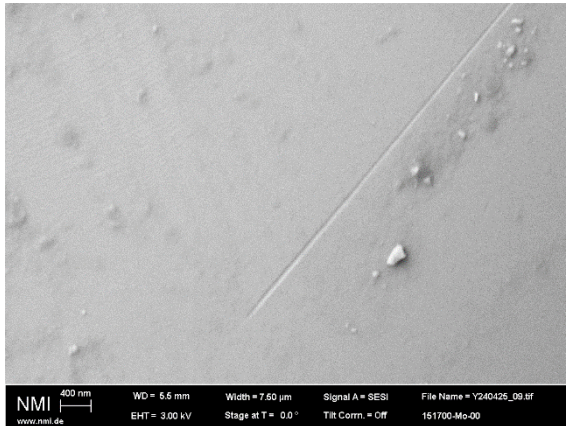


Fig. 4. SEM picture of the part from the metallic lined furnace.

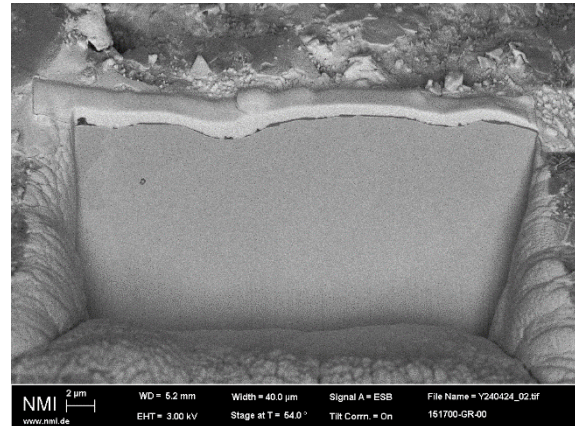


Fig. 5. SEM picture of the cross section from parts treated in the graphite lined furnace. As the backscattered electrons are detected, light-weight elements occur dark.

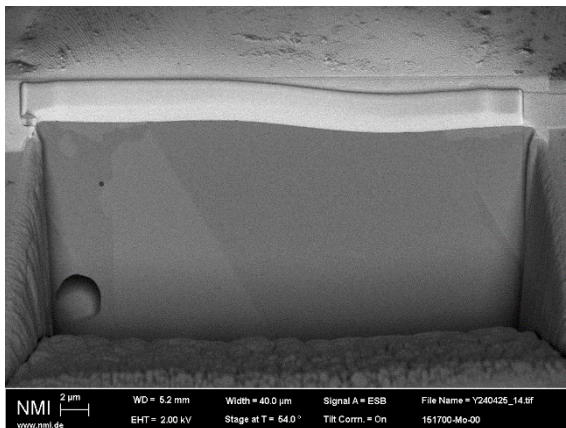


Fig. 6. SEM picture of the cross section from parts treated in the metallic lined furnace. The backscattered electrons are detected. No intermediate layer occurs for all parts from the metallic lined furnace.

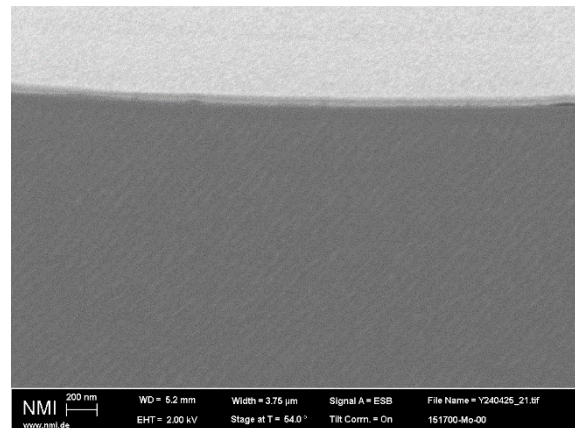


Fig. 7. SEM picture of the cross section from parts treated in the metallic lined furnace.

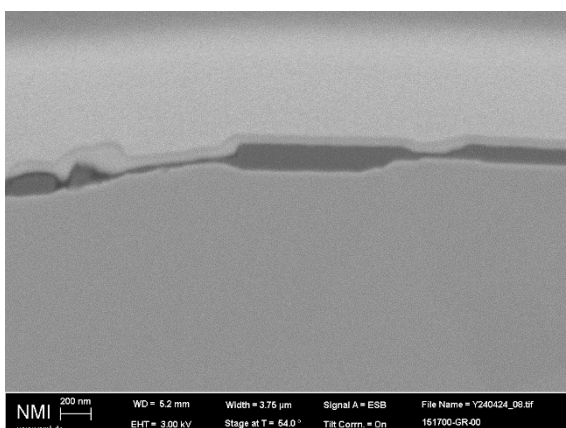


Fig. 8. SEM picture of the cross section from parts treated in the graphite lined furnace in a higher resolution. The dark intermediate layer has a maximum thickness of 160 nm.

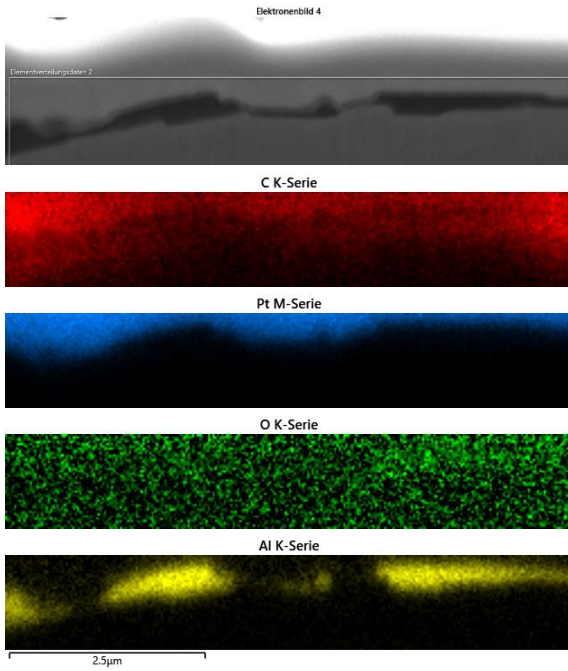


Fig. 10. SEM picture of the cross section from parts treated in the metallic lined furnace followed by the EDX spectra of this intermediate layer. The Carbon content is reduced and is only detected in the Platinum protection layer which is due to the precursor, which contains Carbon as well. The Platinum content is due to the protection layer, which is needed to protect the sample during the FIB cutting process. The Oxygen content is reduced. No contaminations occur at the samples from the metallic lined furnace.

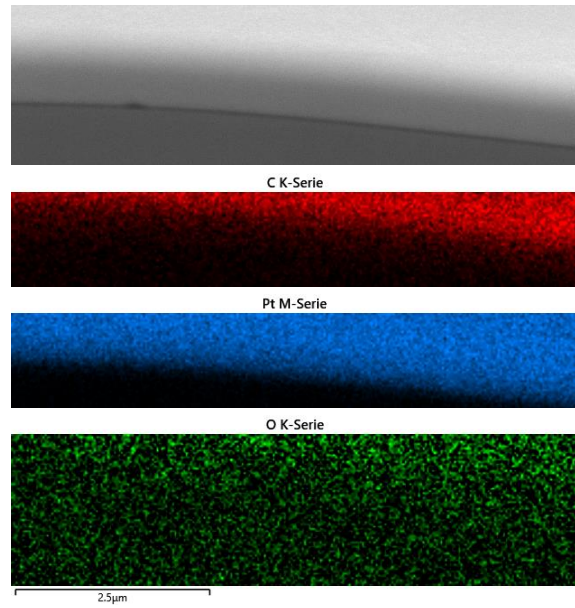


Fig. 9. SEM picture of the cross section from parts treated in the graphite lined furnace followed by the EDX spectra of this intermediate layer. The Carbon content is increased. The Platinum content is due to the protection layer, which is needed to protect the sample during the FIB cutting process. The Oxygen content is increased. The Aluminum peak is somehow unexpected and can only be explained by contaminations in the furnace.

4.1 QUALITY AND RESOURCE CONSUMPTION

Item	Graphite lined furnace (V4)	Metallic lined furnace (V1)	Data sheet
Debinding atmosphere	Fine vacuum	500 mbar (H ₂)	-
Sintering atmosphere	500 mbar (H ₂)	1000 mbar (H ₂)	-
Sintering temperature	1380 °C	1380 °C	1380 °C
Density	7.73 ± 0.17 g/cm ³	7.86 ± 0.11 g/cm ³	7.9 g/cm ³
Carbon content OES	0.02 %	0.01 %	0
Surface	rough	Smooth	-
Carbon EDX peak	2.5 eV	2.0 eV	0
Contaminations	Al	None	-
Corrosion	Yes	No	-
Power consumption	1280 kWh	2090 kWh	-
Product carbon footprint (PCF) or CO₂ equivalent per part grey current	1.2 kg	1.6 kg	-
PCF contribution of feedstock	56 %	29 %	
Of Injection Moulding	3 %	3 %	
Of catalytic debinding	18 %	10 %	
Of sintering	24 %	58 %	

Table 1. Quality and resource consumption: The different adjustments in the graphite- and metallic lined furnace result in different densities, Carbon contents and surface quality. Additionally the graphite furnace results in Aluminum contaminations at the surface of the parts. Both runs take a total of 22 hours. Please note, that the PCF only contains the following: the PCF of the feedstock, the Injection Moulding step, the catalytic debinding step and the final rest debinding and sintering.

5 DISCUSSION AND CONCLUSION

Metallic lined debinding and sintering furnaces result in superior parts quality. The ability to be able to use Hydrogen during the complete heat treatment cycle further increases the quality of the parts regarding, density, Carbon content and surface quality. The improved surface results in less corrosion and further makes the part better suited for coating post processing steps. The possibility to precisely adjust each individual segment of the heat treatment, regarding temperature, gas flow, pressure and gas mixture offers the possibility to further optimize the parts quality. Mainly the possibility to use Hydrogen over the full temperature range under partial

pressure down to 100 mbar and up to atmospheric pressure, results in highest flexibility to tailor each individual parameter of the part, such as Carbon content, density, etc. Studies on the optimum gas flow by CFD simulations are already available at Carbolite Gero. The improved parts quality comes along with an increased energy consumption of the metallic lined MIM furnace. Calculating the energy consumption and the carbon footprint of the complete production run, i.e. Injection Moulding, catalytic debinding, rest debinding and sintering results in a 30 % increased carbon footprint, when using the metallic lined furnace. Future studies need to take the full life cycle into consideration, as the full life cycle is not calculated here and the following contributions are neglected: employees CO₂ footprint by commuting etc., energy consumption of the workshop, CO₂ equivalent of the manufacturing process of the injection moulding machine and of the furnaces.

FIB/SEM and EDX analysis offers a deep insight into the effects from using two different sintering furnaces. Heat treatment in both furnaces yield parts that conform to the target data of 316LG[®]. However, parts from the metallic lined furnace are clearly superior compared to those treated in the graphite lined furnace.

Additionally the running costs are calculated (compare table1). It starts with the feedstock, the consumptions of the Injection Moulding process, of the catalytic debinding step and finally of the rest debinding and sintering heat treatment. Besides the feedstock, the resulting CO₂ equivalent is mainly driven by the current consumptions of the machines. A smaller fraction depends on the further resources, such as N₂, H₂, HNO₃, CH₂O, compressed air and vacuum pump oil. Please contact us if you are interested in the individual consumptions and their related CO₂ equivalents. For this study the CO₂ equivalent of the so called grey current, with a CO₂ equivalent of 35 kg for 100 kWh, was used. Grey electricity is a form of energy produced by mainly burning fossil fuels. It is the most common form of electricity generation and accounts for the majority of electricity consumed worldwide. Coal, natural gas and petroleum are all sources of grey electricity. This is why changing from grey current to green current would reduce the product carbon footprint by more than 80 %. Therefore, there are two important factors to reduce the PCF of the MIM process. First develop machines which are more energy efficient, i.e. by reducing the heat transfers convection, conduction and radiation, by reducing the resource consumption by a improved gas management, by an optimization of the sintering time and by an intelligent software with an improved energy management for the pumps, cooling water etc. These optimizations can be supported by CFD simulations. More studies are needed in this field to determine the possible reduction which is achievable here.

The utilization of renewable energy sources for production processes represents a critical and even more pivotal factor in reducing environmental impact. Numerous companies are already transitioning towards green energy solutions, such as solar power, hydroelectric power, geothermal energy, biomass and other sustainable alternatives, to power their operations. This strategic shift towards sustainability is imperative in the context of global efforts to combat climate change and reduce reliance on fossil fuels. Specifically within the Metal Injection Moulding (MIM) sector, the transition to green energy solutions represents both a challenge and an opportunity. The transition to renewable energy sources also presents economic benefits in the long term, including potential cost savings on energy expenditures and protection against volatile fossil fuel prices. Furthermore, leveraging green energy can enhance a company's brand image, appealing to a growing segment of consumers and stakeholders who prioritize sustainability.

6 REFERENCES

- 1) R.M. German, (2014). Sintering: From Empirical Observations to Scientific Principles. Elsevier.
- 2) B. Kieback, A. Neubrand, H. Riedel, (2011). Processing of Metal Powders. Springer.
- 3) M. Randall, (2006). Industrial Heating Equipment Association: Guide to the Design and Construction of Industrial Heating Equipment. Industrial Press.

SIGN UP FOR OUR NEWSLETTER!

You will occasionally receive **exclusive information** on seminars/webinars, applications and product news.

Subscribe now:



www.carbolite-gero.com/newsletter
(You may unsubscribe any time)

