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MICROWAVE ASSISTED SELECTIVE LASER MELTING OF TECHNICAL CERAMICS

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Abstract

Direct processing of near fully dense technical ceramics is not possible with conventional additive manufacturing (AM) processes due to the very high temperatures that are required. Therefore, indirect AM approaches are often used. These indirect processes show great potential but require extensive post processing (e.g. debinding and sintering) leading to shrinkage, limited geometrical accuracy and eventually limiting overall part quality. To overcome these limitations, this paper presents a novel Microwave Assisted Selective Laser Melting process that enables direct processing of technical ceramics.

Introduction

Selective Laser Melting (SLM) is a widely used Additive Manufacturing technique for metals. Three-dimensional parts are built up in an additive way, in which a 3D CAD model is sliced into two-dimensional layers and subsequently built up until the entire 3D workpiece is completed. Every individual layer is formed by melting a thin (≈60µm) powder layer with a focused high power laser beam. By dividing the entire part into a sequence of two-dimensional layers, very complex geometries (e.g. internal cavities, lightweight structures) can be realized which would not be producible by conventional subtractive processes.

While Additive Manufacturing is more and more straightforward for polymers and metals, it remains a challenge to produce complex technical ceramic parts. Ceramics are more sensitive to process-induced defects (e.g. thermal cracks due to abrupt temperature fluctuations) and require a high preheating temperature. [1] When evaluating possible AM techniques for ceramics, great potential can be found in the **indirect** AM approach since it shifts the process focus to the consolidation of the binder material [1]. Despite its potential, the indirect approaches require several cost inefficient and time consuming post-process steps (e.g. debinding, sintering) that lead to shrinkage, limited geometrical accuracy and eventually low overall part quality [2] [3].

Within this work, a novel **direct** AM approach is presented that, in contrast to **indirect** approaches, enables the manufacturing of nearly fully dense, high-performance structural ceramic parts and limits any post-processing steps. This novel approach combines the conventional SLM process with microwave energy as an additional feature for preheating, lowering extreme temperature gradients, and for assisting in melt pool formation based on the *thermal-runaway effect*.

While the *"Microwave Assisted Selective Laser Melting"*-process was first patented by Christian Gerk in 2001 [4], an adequate machine description patented by 'Rolls-Royce Corporation' was published only in 2015 [5]. The main principles behind this process i.e. *'microwave preheating'*, *'thermal-runaway effect'* and *'laser-microwave material interaction'* [6] [7] [8] [9] would enable **direct** processing of complex ceramic parts. This multi-physics process can be seen as a remarkable more challenging process than SLM, and hence no literature was found dealing with a practical implementation or with process results. This is partly due to the fact that conventional SLM machines are not compatible with microwave power and often cannot cope with high preheating temperatures (≈1100°C).

Foremost among the interesting phenomena associated with microwave heating of ceramics is the *'thermal runaway effect'*. This phenomenon is illustrated in Figure 1, where a gentle temperature increase in the initial heating stage is followed by an exponential temperature boost at longer heating times. This sudden rise of temperature is referred to as thermal runaway.

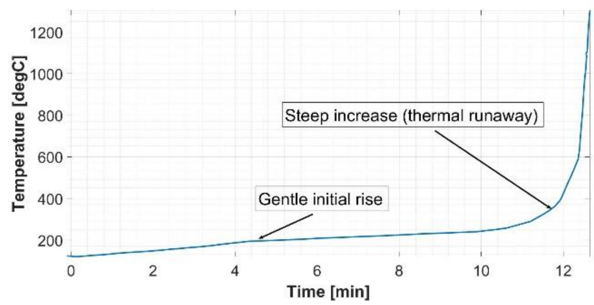


Figure 1:The thermal runaway effect under microwave heating showing the gentle initial rise of temperature followed by a steep increase during the microwave heating of a technical ceramic (CrO) at 500W [10]

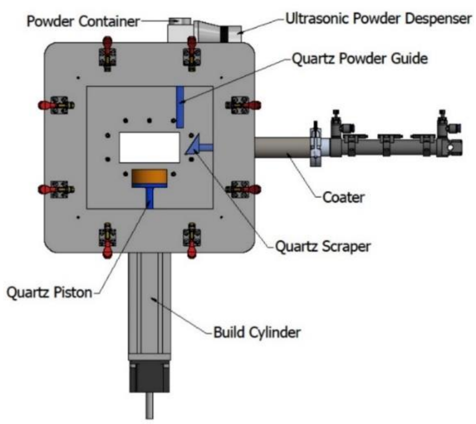
The understanding of microwave heating is still empirical and speculative due to its highly non-linear character. One aspect of this non-linear character is due to the nonlinear material parameters (e.g. magnetic permeability (μ) and electric permittivity (ε)) that change as a function of temperature, phase and frequency. Typical, a material will become more susceptive to microwave energy (magnetic- or electric-field) at higher temperatures, which triggers the *'thermal runaway effect'* since it is a recurring/avalanche effect.

In the concept of *'Microwave Assisted Selective Laser Melting',* the applied laser power will induce a local/selective temperature rise on the powder bed. This causes a change in the material parameters (i.e. μ, ε) making the material more susceptive for the applied microwave power which will eventually trigger the *'thermal runaway effect'*. The laser source is used to initiate a temperature rise to start the *'thermal runaway effect'* and, in combination with the applied microwave power, form a melt pool that is 'dragged' along by means of the laser scan pattern. This approach results in a significantly lower required laser power in comparison with the conventional SLM process.

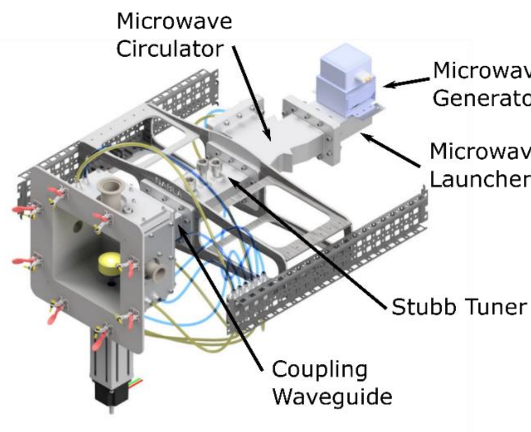
The development of equipment that enables the *'Microwave Assisted Selective Laser Melting'*-process is further discussed in the 'Experimental Setup' section, whereas the processing of single-line tracks and multi-layer parts are presented in the 'Results and Discussion' section.

Experimental details

To support the *Microwave Assisted Selective Laser Melting (MSLM)* process, an in-house experimental setup is developed by re-engineering typical Selective Laser Melting (SLM) machine modules (e.g. layer deposition, process chamber), specifically to enable the integration of a microwave source within a typical Selective Laser Melting setup. Figure 2 provides an overview of the physical layout of the in-house developed prototype with relevant components annotated.



(a)



(b)

Figure 2: (a) General overview of the process chamber and (b) total setup with relevant microwave components.

The following list provides an overview of the adaptations that were made to typical SLM modules to enable the development of the *Microwave Assisted Selective Laser Melting* setup.

1. *Layer Deposition Module*

   Since all components within the process chamber will be subjected to elevated temperature (≈ 1100°C) and high microwave power (2000W), they are manufactured of microwave transparent quartz to exclude any interaction with the E- or H-fields. They are specifically designed to limit temperature fluxes which could cause severe damage to clamping connections or secondary components. An ultrasonic powder dispensing system, directly connected on top of the process chamber, applies the powder material which is deposited on the baseplate by means of the coater. This solution limits the amount of components within the process chamber that might be subjected to elevated temperatures or interact with the electromagnetic field.

2. *Process Chamber/Cavity*

   The presented work focusses on direct SLM of technical ceramics (e.g. Al_2O_3)[1]. Since most pure oxides do not interact well with 2.45GHz microwave energy at room temperature [6], a custom developed SiC susceptor, which highly interacts with the H-field, is used to initiate the self-heating effect by hybrid heating [8] of the Al_2O_3 baseplate. While the E- and H-field distribution in the process chamber are dictated by the dimensions of the process chamber, location/orientation of the inlet waveguide and the applied microwave frequency, a specific process chamber/cavity is designed that allows the heating of the susceptor in its center when subjected to 2.45GHz microwave energy.

   Figure 3 shows the E- and H-field distribution within the designed process chamber/cavity. Figure 3(b) top provides the H-field distribution as viewed from the top in the process cavity. The red regions dictate where high energy H-fields can be found and thus the preferred X/Y location of the developed susceptor. Since the H-field is constant along the Z direction, the susceptor is free to move up and down without any noticeable coupling/temperature change allowing the build cylinder to lower the susceptor and baseplate during successive layer deposition.

[1] While the focus of this work lies on direct processing of technical ceramics, the presented *Microwave Assisted Selective Laser Melting (MASLM)* is not limited to ceramics.

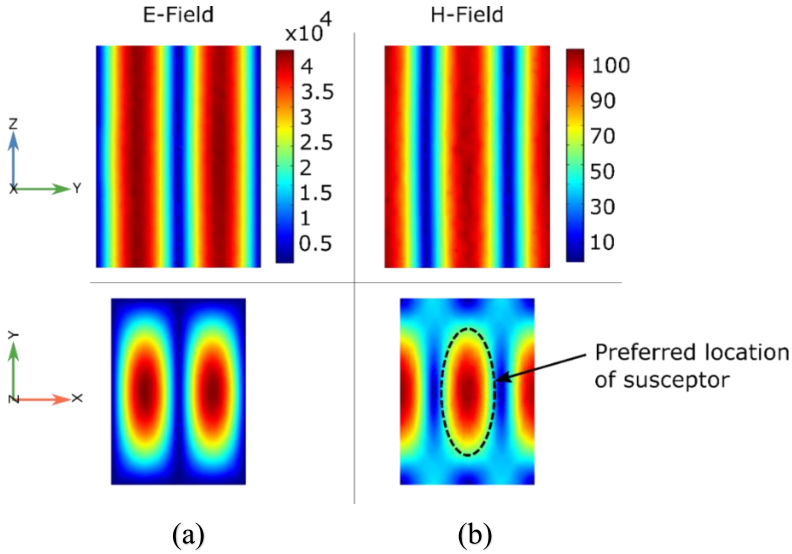


Figure 3: Side and top view of the (a) E-field and (b) H-field distribution within the process chamber.

3. <u>*Laser aperture*</u>

The selective laser melting process requires the ability to selective illuminate the powder bed by means of laser light in order to selectively melt the exposed powder material. This requires the development of an aperture in which the laser light is able to enter the process chamber/cavity[2] and selectively illuminate the powder material (Al_2O_3). Creating apertures within a microwave contained cavity requires special safety attention. In this specific case, the aperture is designed to allow the laser light to enter the cavity and to prohibit any microwave leakages that cause severe harmful radiation.

Figure 4 plots the electric (E), magnetic (H) and power (P) field within the designed laser aperture. Here, the red zones indicate high intensity levels whereas the blue zones are low intensity levels. The intensity levels for all fields are scaled according to the maximum allowed radiofrequency exposure guidelines denoted by Canada's Health Safety Code 6 (2015) in controlled spaces[3] [11]. These plots clearly show that the attenuation of all microwave fields are well within the safety requirements.

In the presented experimental setup, the laser aperture is closed at the top by a WG41012 - Ø2" UVFS broadband precision window provided by *Thorlabs*. This protective window prohibits the propagation of convection heat towards temperature sensitive optical components.

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[2] Multiple process chamber/cavity apertures can be designed for other purposes (e.g. build cylinder actuator, coater actuator, visual view port, thermocouple/IR temperature measurements, etc.)

[3] Based on the region where this prototype is used, different exposure guidelines might be applicable.

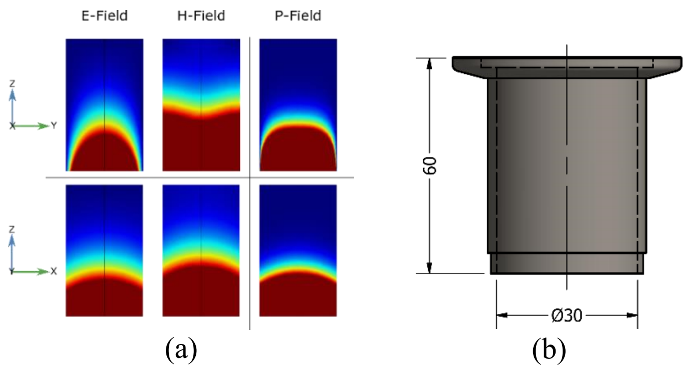


Figure 4: (a) Electric field, magnetic field and power distribution within the laser cavity aperture. The applied color map is scaled according to the recommended occupational exposure limits in controlled spaces.(b) Detail of the laser cavity aperture with annotated dimensions.

4. *H-field geometry based SiC susceptor*

Many pure oxides (e.g. Al_2O_3 and ZrO_2) with predominant ionic bonding do not couple well with 2.45 GHz microwave energy at room temperature [6]. SiC or graphite as a susceptor material allows the microwave energy to first couple strongly with the susceptor, which rapidly heat and in turn heat the poor microwave-absorbing Al_2O_3 (baseplate and powder material) by conduction heating, up to a critical temperature where microwave absorption is sufficient to allow self-heating [8].

Loading the process cavity with high microwave-absorbing materials (SiC susceptor) might influence/distort the field distributions. Resulting from these field distortions, unwanted hotspots and a strong inhomogeneous temperature distribution might occur within the susceptor. Since most technical ceramics do not cope with these high temperature differences, the Al_2O_3 baseplate and/or the part that is being manufactured, will crack, causing the job to drastically fail.

To limit the field distortion, the preferred area of the susceptor is matched to the expected H-field distribution [12]. To illustrate this, Figure 5(a) shows the expected H-field top view contours, inside the process cavity. The red contours represent high H-field intensities, whereas the green contours represent low H-field intensities. Matched on these contours, Figure 5(b) illustrates a simplified susceptor geometry with an Al_2O_3 baseplate.

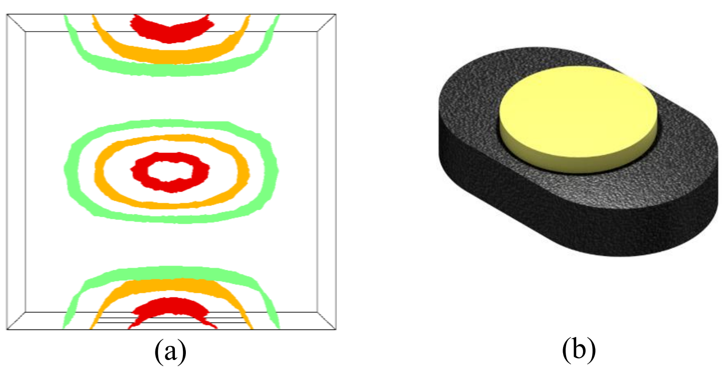
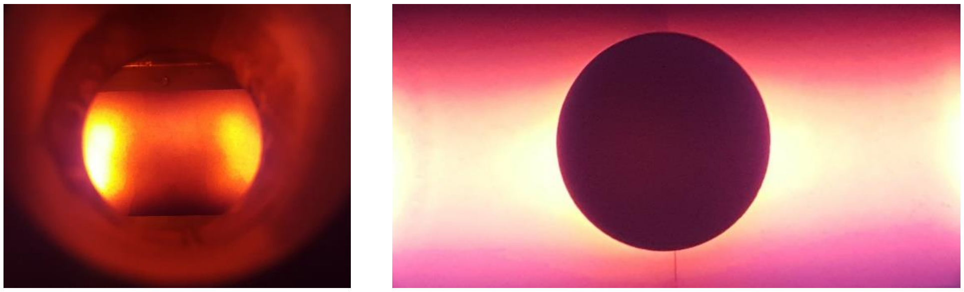


Figure 5: (a) Top view of the magnetic field contours inside the process cavity. Red contours represents high intensities whereas green contours represents lower intensities. (b) Simplified susceptor geometry based on magnetic contours with Al_2O_3 baseplate on top.

As mentioned earlier, the formation of hotspots can lead to severe inhomogeneous temperatures throughout the susceptor. To illustrate this, pictures were taken through the laser aperture of a microwave heated non-matched H-field susceptor (Figure 6(a)) and a matched H-field susceptor (Figure 6(b)). Figure 6(a) clearly indicates hotspots, whereas Figure 6(b) shows a more homogeneous temperature distribution throughout the susceptor with an Al_2O_3 baseplate on top.[4]

[4] Placing the Al_2O_3 baseplate on a susceptor that suffers from hotspots will cause the baseplate to crack due to thermal stress.



(a) (b)

Figure 6: (a) Top view of a heated non-optimal geometry susceptor with visible hotspots on the left and right. (b) Top view of the heated susceptor with H-field distribution based geometry in combination with an Al_2O_3 baseplate. Notice that due to the magnetic field distribution design (b), a more homogeneous temperature distribution within the susceptor is achieved and no hotspots are visible.

5. <u>*Optical Setup*</u>

The optical setup for the prototype is based on a conventional SLM optical setup enhanced with melt pool monitoring [13]. Figure 7 provides an overview of the optical setup with all relevant optical components annotated. In addition to the main purpose of a melt pool monitoring, i.e. capturing the process light emitted by the melt pool, the melt pool photodiode serves also as a baseplate temperature measurement device that is further discussed in item 6.

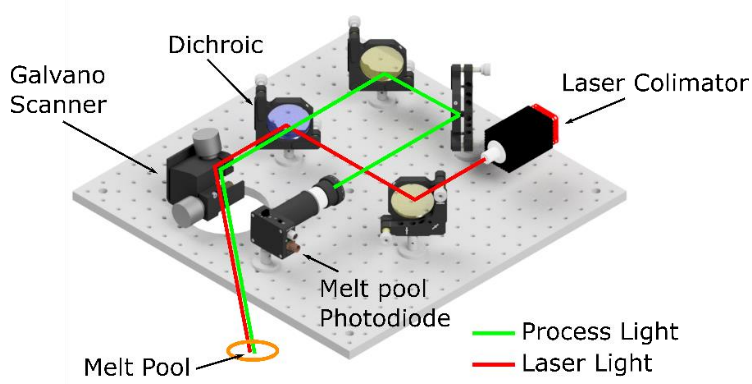


Figure 7: Optical Setup with melt pool monitoring

The prototype is constructed to work in two different operation modes during scanning, i.e. *'Scanning with microwave source off'* and *'Scanning with microwave source on'*. The latter mode will initiate the 'Thermal Runaway' effect' [7] [4]. If not controlled, the thermal runaway will cause a drastic local temperature rise that will lead to cracking, arching, and eventually job failure [9]. While an uncontrolled thermal runaway is undesired, the resulting local temperature rise is beneficial to the process of melting the Al_2O_3 powder material. To overcome an uncontrolled thermal runaway, the melt pool photodiode actively controls the laser- and microwave-power in such way that the melt pool intensity remains constant while scanning [14].

The thermal-runaway effect does not initiate instantaneous when scanning starts. Depending on the pre-heating temperature, microwave and laser power, it can take several seconds to observe any noticeable melt pool intensity peaks. To ensure proper starting conditions, the scanning commences only when the melt pool photodiode detects the thermal-runaway. After the scanning initiation, the melt pool photodiode is switched to - the previous mentioned - laser and microwave power control.

6. <u>*Temperature calibration*</u>

During the process, the Al_2O_3 baseplate and powder material are preheated up to ≈1100°C. This requires a temperature measurement that can actively control the microwave power. While a typical thermocouple could serve this purpose[5], the melt pool photodiode is used to measure the top temperature of the baseplate. Since the photodiode integrates all wavelengths within the process light, within the field of view and in function of its sensitivity to different wavelengths, it does not provide a direct temperature readout for a specific area.

To calibrate the photodiode signal to a baseplate temperature, the scanning system operates in two different modes, i.e. *'Layer scanning'* and *'Temperature Scanning'*. While the former does not deviate from typical SLM scanning techniques, the latter scans a 40x40mm square, without laser power, constantly during the preheating phase and intermediate during the scanning of layers. During *'Temperature Scanning'*, the photodiode signal is mapped to the X/Y coordinates of the scanner. From this map, the area of the baseplate is extracted and calibrated to the real temperature of the Al_2O_3 baseplate. The calibration value is obtained by a, one-time, thermocouple measurement within the area of the baseplate.

To illustrate this, Figure 8 plots different views of the mapped photodiode signal. While the temperature of the SiC susceptor and the Al_2O_3 baseplate have more or less the same temperature, two noticeable intensity peaks are observed. These peaks are related to the difference in emissivity of the two materials where the susceptor emits more light at higher frequencies in contrast to the baseplate at the same temperature. Figure 8(a) and (b) plot the addition of the calibrated baseplate temperature. To exclude any interference of the susceptor on the temperature readout, the areas, other than the baseplate area, were discarded during temperature measurement.

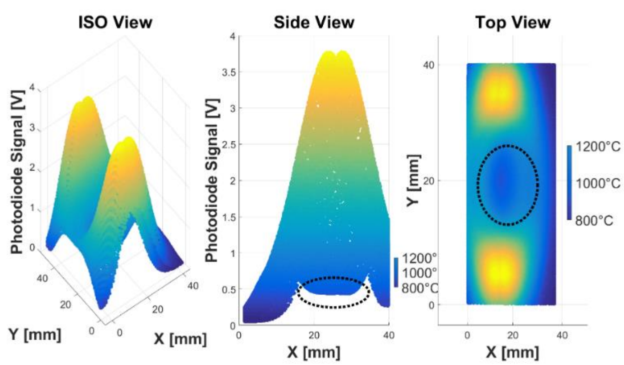


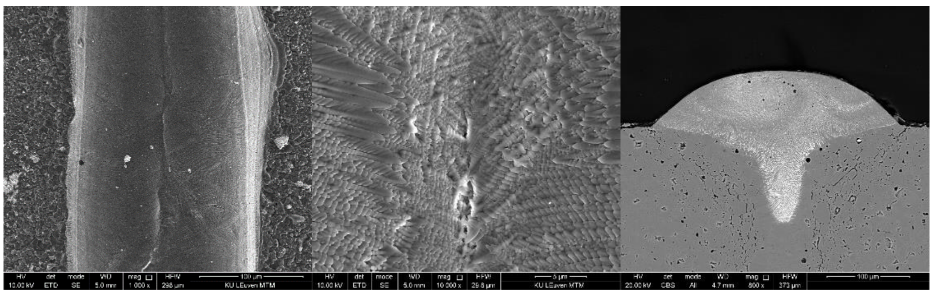
Figure 8: X/Y mapped photodiode signal while scanning over the susceptor with an Al_2O_3 baseplate at 1000°C. (a) ISO View of X/Y mapped photodiode signal, (b) Side View of X/Y mapped photodiode signal with calibrated temperature area at the Al_2O_3 baseplate. (c) Top view of X/Y mapped photodiode signal with calibrated temperature area at the Al_2O_3 baseplate.

[5] A thermocouple housed in a thin metallic tube can be used inside a microwave heated cavity. It causes the least interference if it is placed perpendicular to the electric field. If the thermocouple is placed parallel to these fields, sparking could occur. It is of great importance that the metallic sealed thermocouple is galvanically connected to the cavity wall at the point of insertion. If this is not the case, strong common mode current will be able to flow along the shielding and out of the cavity where harmful radiation levels will occur [15].

Results and discussions

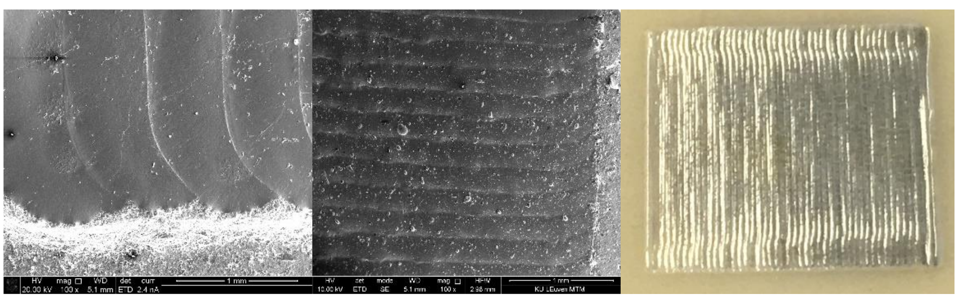
To validate the in-house developed SLM machine that supports the *'Microwave assisted Selective Laser Melting'*-process for technical ceramics, single line scan-tracks and square-single layers were processed. A small amount of pure ZrO_2 was added to the Al_2O_3 powder to ensure good flowability at the elevated preheating temperature of 1100°C. While the addition of ZrO_2 results in a deviation of the foreseen processing of pure Al_2O_3, this powder mixture is still considered as a relevant technical ceramic and suits as a validation powder.

Layers with a thickness of 50µm were deposited on a pure Al_2O_3 baseplate. A preheating temperature of 1100°C and *'scanning with microwave source on'* was selected to initiate the controlled *'thermal runaway'*-effect. After processing, the part was cooled down to room temperature, in a controlled way, to limit thermal cracks. As a result, Figure 9 provides the SEM analyses of a successful single scan track, whereas Figure 10 provides the SEM analyses of a successful square single layer.



(a) (b) (c)

Figure 9: SEM analyses of (a) single line track, (b) dendritic microstructure of the single line track and (c) cross-section of single line track.



(a) (b) (c)

Figure 10: (a) (b) Scanning electron micrographs and (c) optical microscopy top view photo of square single layer

During validation, it was noticed that by selecting different process parameters and control strategies, different densities and translucencies could be achieved. Here, Figure 10 (c) clearly shows a high transparent layer, on top of an Al_2O_3 base pate, indicating a high density.

Conclusion

The work presented in this paper discusses a practical implementation of the *'Microwave Assisted Selective Laser Melting'*-process to enable a **direct** Additive Manufacturing technique for successfully processing ceramics directly from the melt. By the implementation of a microwave source and adequate equuipment and process control techniques, a preheating temperature of ≈1200°C is achievable with the aid of a custom engineered SiC susceptor. The resulting controlled '*thermal-runaway'*-effect assists in the formation of a melt pool and eventually the consolidation of the ceramic. First exploratory work indicates that single line tracks and single square layers could be produced with high densities and without any thermal cracks. In contrast to the existing indirect AM techniques, no cost inefficient nor time-consuming post-process steps (e.g. debinding, sintering) are required.

Acknowledgements

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References

[1] J. Deckers, J. Vleugels and J.-P. Kruth, "Additive Manufacturing of Ceramics: A Review," *Journal of Ceramics Science and Technology,* vol. 5, pp. 245-260, 2014.

[2] A. Zocca, P. Colombo, C. M. Gomes and J. Gunster, Additive Manufacturing of Ceramics: Issues, Potentialities and Opportunities, Soc.: J. Am. Ceram., 1983-2001.

[3] J. Wilkes, Y. Hagedorn, W. Meiners and K. Wissenbach, "Additive manufacturing of ZrO2-Al2O3 ceramic components by selective laser melting.," *Rapid Prototype J.,* Vols. 19(1):51-7, 2013.

[4] C. Gerk and M. W. Porada, "Verfahren zur Erwärmung von Materialien und Materialverbunden mittels Laser- und Mikrowellenenergie, Vorrichtung zur Durchführung des Verfahrens und nach dem Verfahren hergestelltes Bauteil". Germany Patent DE19951143 A1, 26 April 2001.

[5] Y. Q. Shuck and J. S. Bader, "Method and apparatus for forming three-dimensional articles". US Patent EP 2851180 A1 - US20150084240, 25 03 2015.

[6] Y. R. K. a. S. V. Bykov, "High-temperature microwave processing of materials," *J. Phys. D: Appl. Phys.,* no. R55-R75, p. 34, 2001.

[7] G. Kriegsmann, "Thermal runaway in microwave heated ceramics," *Applied Physics,* p. 71, 1992.

[8] W. W. L. E. Manoj Gupta, Microwave and Metals, Singapore: John Wiley & Suns(Asia) Pte Ltd, 2007.

[9] J. J. Thomas, X. Wu and W. A. Davis, "Control of thermal runaway in microwave resonant cavities," *Journal of Applied Physics,* vol. 92, no. 6, p. 3374, 2002.

[10] K. V.M., S. L. and W. M.W., "Theory of microwave Interactions in ceramic materials: the phenomenon of thermal runaway," *Journal of materials science,* vol. 26, pp. 2483-2489, 1991.

[11] Canada.ca, "Safety Code 6: Health Canada's Radiofrequency Exposure Guidelines," 2015. [Online]. Available: https://www.canada.ca/en/health-canada/services/environmental-workplace-health/reports-publications/radiation/safety-code-6-health-canada-radiofrequency-exposure-guidelines-environmental-workplace-health-health-canada.html.

[12] A. Toossi, M. Deneshmand and D. E. Sameoto, "Microwave Heating with susceptor". United States Patent US 2013/0334218 A1, 19 December 2013.

[13] S. Clijsters, T. Craeghs, S. Buls, K. Kempen and J.-P. Kruth, "In situ quality control of the selective laser melting process using a high-speed, real-time melt pool monitoring system.," 2014.

[14] S. Buls, "A Smart Machine for Selective Laser Melting - A Controlled and Synchronized System Approach," *PHD KULeuven,* 2018.

[15] H. C. R. TSE V. Chow Ting Chang, Understanding Microwave Heating Cavities, Norwood: Artech House, INC, 2000.

[16] G. o. Canada, "Safety Code 6: Health Canada's Radiofrequency Exposure Guidelines," 03 06 1015. [Online]. Available: https://www.canada.ca/en/health-canada/services/environmental-workplace-health/reports-publications/radiation/safety-code-6-health-canada-radiofrequency-exposure-guidelines-environmental-workplace-health-health-canada.html.

[17] J. P. Bently, Principle of Measurment Systems, vol. 4, Harlow: Pearson Education Limited, 2015.