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Analysis of Wire-EDM finishing cuts on large scale ZrO_2-TiN hybrid spark plasma sintered blanks

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Abstract

Technical ceramics have numerous application in the industrial and biomedical field because of material properties such as: high hardness, high Young’s modulus, chemical resistance and dimensional stability. These superior properties make it difficult to shape technical ceramics in a flexible and accurate way. By embedding an electrically conductive phase inside tough, hard and brittle non-conductive technical ceramics, e.g.: ZrO_2, Al_2O_3 & SiC, it is possible to create ceramic blanks which can be processed by electro-chemical processes, such as Wire Electrical Discharge Machining (W-EDM). Till today, research on EDM machining of ceramic parts is often limited to the machining of small sized samples, mainly due to the fact that larger blanks are difficult to produce or not commercially available.

Today, novel sintering techniques make it possible to create large fully dense ceramic composite blanks, allowing the production of bigger monolithic ceramic parts in a more cost effective manner. In the presented research, the effect of higher cutting dimensions on the Wire EDM performance is investigated. ZrO_2-TiN blanks (diameter: 250 mm, height: 16 mm) made by hybrid plasma spark sintering are processed by W-EDM. Different finishing steps are used to obtain R_a roughness values below 0.5 µm, and for each finishing step a detailed analysis is made of the surface characteristics and material removal mechanism. The surface quality and material removal mechanisms are compared to findings in literature for small scale blanks. Beside melting and evaporation, other phenomena such as chemical decomposition and recast layers can be observed. Furthermore the effect of the finishing cuts on the bending strength is tested using 4 point bending setup and correlated to the surface roughness. Analysis from this research show that large scale ZrO_2-TiN blanks are industry ready to be machined by W-EDM, hopefully bringing forward new applications.

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Keywords: Wire EDM; Ceramic composites; ZrO_2; Spark Plasma Sintering; Large scale ceramics

1. Introduction

For many high demanding applications in the industry and biomedical field technical ceramics (e.g.: Al_2O_3, SiC, B_4C,. Si_3N_4, etc.) provide solutions, because of their mechanical properties such as: high Young’s modulus, superior abrasion resistance, chemical resistance, high melting point and dimensional stability. In return these properties make it challenging for shaping ceramic materials in an accurate and cost effective manner. An alternative to conventional machining of ceramics, often involving diamond tools or complex machining operations, would be electro chemical processes, as for instance electrical discharge machining (EDM) which is well established in the industry. Although there are some setups which allow EDM processing of non-conductive materials [1], standard EDM processes do require the work piece to be electrically conductive [2].

Zirconium dioxide (ZrO_2), known to the general public for its use in the dental industry, is especially interesting because of its excellent fracture toughness. However this material cannot be machined using the conventional EDM setup, as it is one of the worst electro-conductive materials ($>10^{10}$ Ω.cm). This issue can be solved by embedding a conductive phase into the ZrO_2 material, creating a ceramic composite. Different conductive phases have already been embedded into different type of non-conductive ceramics. Early studies investigating

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EDM of ceramic composites have focused on the amount of conductive phase needed to perform EDM operations [3,4,5]. While later studies have made detailed analysis of the material removal mechanisms of ceramic composites [6] and the effect of EDM operations on mechanical performance [7].

Fig. 1. 250 mm Diameter ZrO_2-TiN blank shown next to smaller 150 mm and 40 mm blank.

In the present study ZrO_2 ceramic blanks with TiN as secondary conductive phase are processed by Wire EDM (W-EDM). Beside increasing the electro-conductivity the TiN content also increases the hardness and flexural strength [8]. In the past ZrO_2-TiN materials with similar compositions described in this work have been processed by W-EDM [6], within this study R_a values of 2.7 µm for roughing were reached and melting and evaporation was shown to be the principal material removal mechanism. However up to now most research is conducted mostly on small scale samples with a limited height. In this research a large scale blank with a diameter of 250 mm and a height of 16 mm is used as the rough work piece. To fully explore the use of large scale blanks, the ceramic blank is processed as it is at a height of 16 mm (rough thickness) and at a height of 70 mm (a rectangle cut from the rough blank and placed upright). Fig. 1 shows the large scale blank next to a smaller 150 mm and 40 mm in diameter blank.

The large scale blank has been produced by hybrid spark plasma sintering (SPS). Conventional SPS produces blanks through Joule heating by sending a pulsed DC current through a cylindrical pressing die (consisting of high impact graphite) and through the compacted powder material [10,11]. Hybrid SPS uses both Joule- and inductive heating at different stages of compression to reinsure even heat distribution from the center to the edge of the pressed blank [12], permitting the production of larger and more homogeneous blanks. Finally customized monolithic parts can be cut cheaper and faster from large blanks via W-EDM.

2. Experimental setup

2.1. Material

The large scale blank is obtained from 59.3 vol.% ZrO_2 and 39.6 vol.% TiN powder content, using 1.1 vol.% Al_2O_3 and 0.1 vol.% Y_2O_3 as sintering aids and stabilizers. The powder mixture was ball milled by a turbula mixer in an ethanol medium. Multiple batch sizes of mixing were used to prepare the powder going into the large scale blank. Each batch consisted of 500 grams, with 2.5 kg ZrO_2 balls having a diameter of 3 mm. After mixing the powder mixture is dried and sieved using a 400 µm mesh, before being placed into the SPS oven. The sintered material has a Vickers hardness of 1360 kg/mm^2 and flexural strength of the material is tested by a 4 point bending setup in which grinded samples of 3 x 4 x 50 mm were tested, resulting in an average flexural strength of 1104 MPa. The Weibull plot of the 4 point bending strength of the grinded material is included in Fig. 7 in section 3.3. and will be used as reference for determining the influence of the W-EDM process on the flexural strength.

2.2. Apparatus & Process settings

Two types of samples (height 16 and 70 mm) were examined. Samples of the height of 70 were prepared from a 70 x 50 mm^2 block cut from the cylindrical blank. At this height 7 finishing steps were examined. All samples are manufactured on an AgieCharmilles 300mS W-EDM machine using demineralized water as dielectric (11 µS/cm). A 0.25 mm diameter AC Brass wire with a tensile strength of 900 N/mm^2 is utilized as cutting electrode.

Table 1. Generator settings for the “isopulse” controlled cuts.

Cut	Height (*mm*)	Disch. time (*µs*)	Pulse off time (*µs*)	Curr. (*A*)	Open gap volt. (*V*)	Ref. ser. volt. (*V*)
1	16	0.56	7.4	5	80	62.0
	70	0.40	7.0	5	80	52.0
2	16	0.05	7.0	5	120	87.4
	70	0.05	6.6	5	120	64.0
3	16	0.1	14.0	3	100	60.4
	70	0.05	25.0	2	60	23.0

EDM settings are based on commercial hard metal settings, for the initial cut discharge time is decreased and pulse off time is increased to reduce the risk of wire breakage. In hard metals the machine manufacturer foresees an R_a roughness of 0.2 and 0.15 µm for the 6th and 7th finishing cut. The most important generator parameters to perform the first 3 cuts are displayed in Table 1. For these cuts the generator is set in “isopulse” mode, consequently discharge time and pulse off time are fixed, and the wire travel servo is controlled by the voltage during erosion (indicated by ‘Ref. ser. volt.’ in Table 1).

Table 2. Generator settings for “isofrequency” controlled cuts.

Cut	Height (*mm*)	Pulse on time (*µs*)	Pulse off time (*µs*)	Curr. (*A*)	Open gap volt. (*V*)	Speed. (*mm/s*)
4	16	0.4	3.0	5	120	8.93
	70	0.4	3.0	5	120	4.42
5	16	0.5	1.0	2.5	120	9.37
	70	0.5	1.0	2.5	120	4.80
6	16	0.5	0.6	3	120	5.06
	70	0.5	0.6	3	120	2.50
7	16	n.a.	n.a.	n.a.	n.a.	n.a.
	70	0.4	0.6	3	60	2.50

Table 2 shows the most important generator settings for the final finishing cuts (cut 4 to 7). To perform these cuts the generator is set in “isofrequency” mode. The speed of travel of the wire is kept constant (indicated by ‘Speed’ in Table 2.). The generator is put on for a period indicated by ‘Pulse on time’ in Table 2. The real discharge time during these cuts is unknown.

Table 3. Machining path offset values for the different cuts.

Sample height (mm)	Cut (μm)						
	1	2	3	4	5	6	7
16	171	141	134	132	131	131	n.a.
70	220	163	144	135	133	133.5	132.5

Machining path offset values for the different cuts are shown in Table 3. Concerning flushing the first cut flushing is set at 17 l/min at the height of 16 mm and 21 l/min at a height of 70 mm. For the remaining cuts flushing is set at 2 and 4 l/min respectively, for the 16 and 70 mm samples. The higher flow is needed for higher parts since initial tests showed pitting marks, due to trapped debris in the sparking gap when using a 2 l/min.

3. Experimental results

3.1. Material Removal Mechanism (MRM)

SEM images of the W-EDM processed surfaces (Fig. 2. & Fig. 3.) show the surface structure getting more refined with a higher number of finishing cuts. There are similarities for the samples with a height of 16 and 70 mm. Recast layers can be observed on the cross sectional SEM images (Fig. 4.) suggesting melting and re-solidification. Cracks are shown on the surface and cross sectional images. The crack depth is limited to the thickness of the recast layer, which suggests that cracks are caused by shrinkage during re-solidification.

$$2TiN + 4H_2O \rightarrow 2TiO_2 + 4H_2 + N_2 \quad (1)$$

Pores can also be observed most likely due to escape of nitrogen gas (N_2) from the surface. Since water is used as a dielectric it is plausible that the TiN oxidizes to TiO_2 producing hydrogen and nitrogen gas, as illustrated by equation (1). This hypothesis is further backed up by Energy Dispersive Analysis of X-rays (EDAX). In Table 4. the count for most prominent elements in the polished versus an EDM finished surface are presented. The table shows a reduction of nitrogen and an increase of oxygen for an EDM surface.

Table 4. EDAX of polished material vs. EDM surface material.

Element	Polished surface			EDM surface		
	Wgt.%	Atm.%	Err. %	Wgt.%	Atm.%	Err. %
Zr	30.56	9.40	1.73	30.63	8.49	1.83
O	19.08	33.46	10.52	17.20	27.18	10.33
Ti	27.37	16.03	2.45	17.75	9.37	2.65
N	20.06	40.18	9.58	2.95	5.33	17.66
Y	2.93	0.93	2.90	3.13	0.89	3.01
Al	n.a.	n.a.	n.a.	0.28	0.26	9.53
C	n.a.	n.a.	n.a.	22.17	46.68	11.41
Pd	n.a.	n.a.	n.a.	3.34	0.79	4.02
Cu	n.a.	n.a.	n.a.	2.56	1.02	10.30

Oxidation of TiN is a process which typically occurs above 500 °C. TiO_2 has a melting point of 1750 °C and boiling point above 3000 °C. Although melting and evaporation is the main MRM for both 16 and 70 mm samples, there seems to be a difference concerning decomposition. The 70 mm samples partially show foamy structures also suggesting decomposition. This is probably due to the energy flux, which is a function of the pulse energy over the cutting height and cutting speed. For the 16 mm samples the energy flux is about 2 times higher compared to the 70 mm samples, allowing melting and evaporation. Chemical decomposition occurs earlier, hence the 70 mm cuts decompose and partly melt and evaporate.

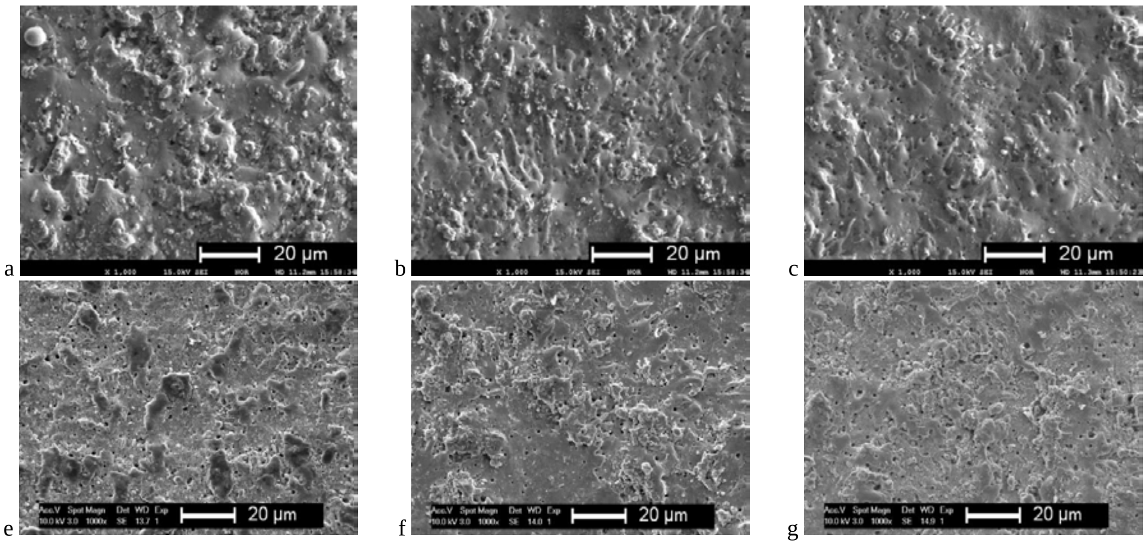

Fig. 2. SEM images of the EDM ZrO_2-TiN surfaces “isopulse” controlled cuts, height 16 mm cut 1 (a), 2 (b) & 3 (c), and height 70 mm cut 1 (e), 2 (f) & 3 (g).

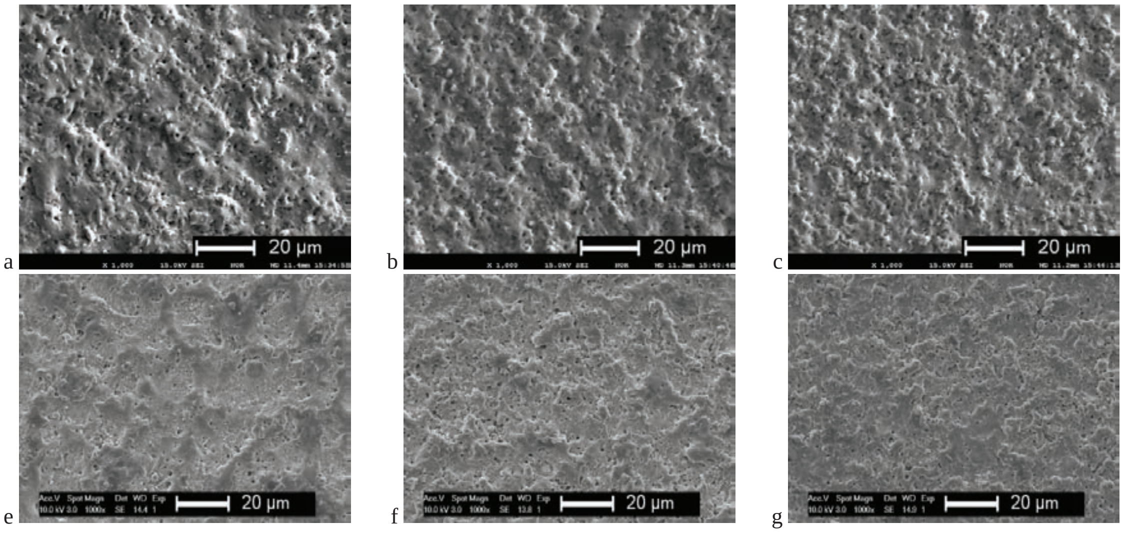

Fig. 3. SEM images of the EDM ZrO_2-TiN surfaces “isofrequency” controlled cuts, height 16 mm cut 4 (a), 5 (b) & 6 (c), and height 70 mm cut 4 (e), 5 (f) & 6 (g).

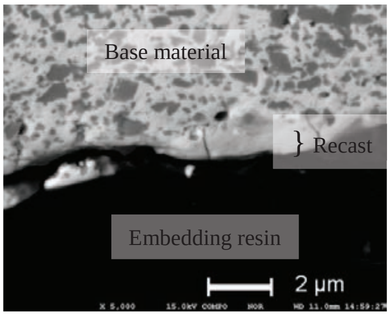

Fig. 4. Cross sectional images showing the recast layer and cracks, cut 4 from the 70 mm high sample.

3.2. Surface roughness

As a finer and finer structure is revealed for each finishing cut, surface roughness also decreases for most finishing steps. Fig. 5 shows the R_a and R_z roughness values at different cutting steps. All roughness values are performed using the ISO 4288 (DIN 4768) standard, with a 4 mm sampling length and 0.8 mm cut-off length.

The initial cut can only produce an R_a above 2 µm and 1.5 µm respectively, for the 16 and 70 mm samples. This cut is merely used to get the rough shape of the part, more importantly is to what extend the surface roughness can be reduced in the cuts following after. After 6 finishing steps the 16 mm samples have an average roughness of 0.51 µm R_a (and 3.46 µm R_z). The 70 mm samples can even reach an average R_a of 0.42 µm (and 2.64 µm R_z). The extra 7th finishing cut performed on the 70 mm samples can even lower the R_a to 0.30 µm (and 1.93 µm R_z).

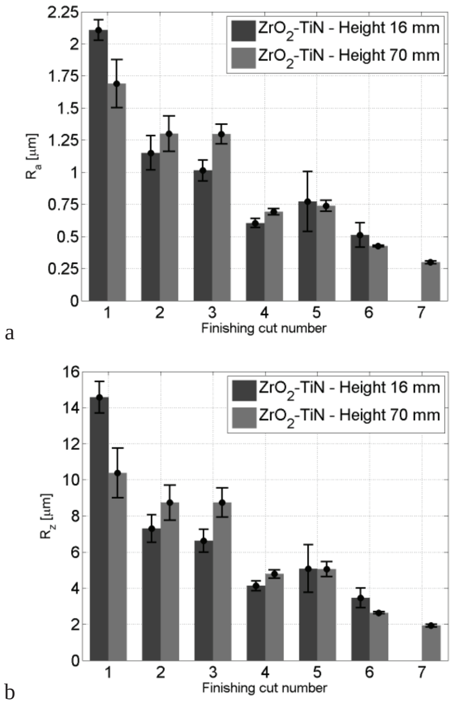

Fig. 5. (a) R_a and (b) R_z surface roughness of the different finishing cuts.

Furthermore variation on the roughness is smaller for cut 5 to 6 on the 70 mm high parts. An explanation for both the lower roughness and smaller variation are burn marks (displayed in Fig. 6) which were only present after the 5th and 6th cut on the 16 mm samples. The large burn marks after the 5th finishing cut explain the large variation in roughness at this cut. This cut can probably be omitted for both 16 and 70 mm high cuts, as it doesn't improve surface roughness over the previous cut.

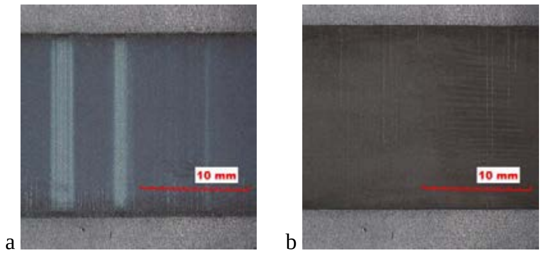

Fig. 6. Burn marks on the surface of the 5th (a) and 6th (b) cut of the 16mm high sample.

The burn marks are due to incorrect settings of machining path offset values in combination with the generator settings, since this latter controls the sparking gap. Fine tuning the offset value will require the least of effort since only one setting needs to be adjusted and will be investigated in future research. The reason the offset value is so critical for the last finishing cuts is because the process is no longer servo controlled ("isofrequency" mode). There is no feedback from the spark duration to the machining speed, and the wire is moving along the cutting surface at a fixed speed to ensure a consistent surface quality. If the distance between the wire and cutting surface is too large for the open gap voltage to bridge, no sparks will be generated and there is no machining of the surface. Contrariwise, if the wire is going to deep into the material and energy is insufficient to remove all the material. The wire will move over the surface in short circuit, not generating sparks. This is why non-servo controlled cutting is applied during the final finishing steps, it requires a certain level of finishing (flatness and roughness) but also reinsures a more consistent surface finish when applied correctly.

3.3. Flexural strength

To evaluate the effect of the EDM processing on the flexural strength of the ZrO_2-TiN material, 4 point bending tests were performed. Samples of 3 x 4 x 50 mm were produced by firstly cutting 3 x 50 mm rectangles by W-EDM from the blank using the 16 mm high settings. Next these shapes where cut into 3 x 4.5 mm beams by W-EDM. Finally the beams were than grinded down to 3 x 4 mm. In this manner the surfaces perpendicular to the compression load are EDM processed, while parallel surfaces are ground. Fig. 7 demonstrates the 4 point bending setup, the hatched surface in this figure represents the grinded face, all other faces are machined by EDM. In total 3 samples were produced per finishing cut (cut 1 to 6), resulting in a total of 18 samples.

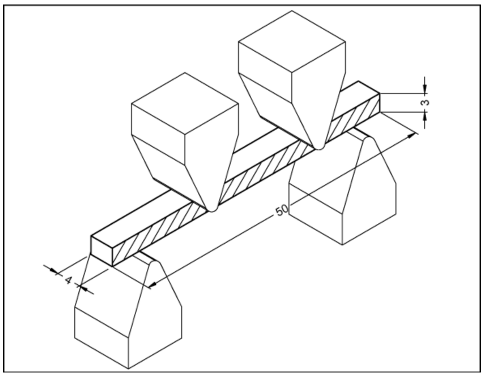

Fig. 7. Setup of the 4 point bending test.

SEM images of the EDM machined surface and cross section revealed cracks (Fig. 2., Fig. 3. & Fig. 4.), these cracks will have a significant influence on the ZrO_2-TiN behavior. Fig. 8 displays the results for the 4 point bending test on different finishing cuts. The bar plot on this figure shows the average for each finishing cut, the error bars indicate the maximum and minimum measured strength. The average and maximum strength doesn't show a clear trend. The minimum strength does however increase for each finishing cut. Since only 3 samples per finishing were produced a Weibull analysis for each cut cannot be composed. However, the increase in minimal strength does suggest that the chance of failure is smaller for a higher finishing cut.

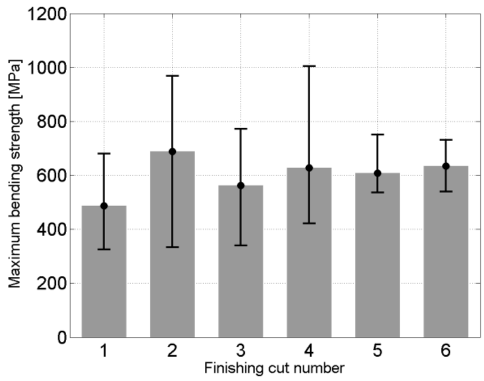

Fig. 8. Flexural strength at different EDM finishing cuts.

To position the flexural strength of EDM finished surfaces with respect to flexural strength of ground surfaces a Weibull plot of all EDM produced samples is created (Fig. 9), in this figure the probability of failure (from 0 to 100%) is plotted versus the flexural strength. Furthermore a linear first order function (line) is fitted through these points.

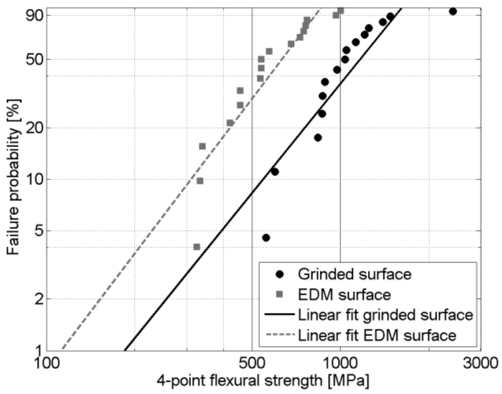

Fig. 9. Weibull entire EDM samples population.

The Weibull of the EDM samples shows that the angle of the linear fitted Weibull plot does not change in comparison to grinded samples. There is a translation of the linear Weibull regression of about 500 MPa. This points to the fact that the distribution of flexural strength does not change, but the distribution is biased. So an EDM surface has the same distribution of failure as a ground surface and will fail at a lower strains.

4. Conclusions

Large scale ceramic composite blanks open up new possibilities for creating large monolithic parts for applications in different fields, e.g.: the tooling industry, aero-space, bio-medical, etc.. An analysis has been made of the effect of the W-EDM process on ZrO_2-TiN material coming from a large scale hybrid SPS blank with a diameter of 250 mm and thickness of 16 mm. To examine this behavior different finishing cuts were performed on samples with a height of 16 and 70 mm. SEM images of the EDM processed surfaces and cross sections suggest melting and evaporation as main MRM. Additionally EDAX also suggests chemical decomposition for the 70 mm samples. This is most likely because more energy is put into the surfaces of the 16 mm high cuts.

Important factors for the implementation of this material in the industry are the surface quality and mechanical behavior. To examine the surface quality roughness measurements are performed. With the suggested W-EDM settings (generator, machining path offset and flushing) an R_a roughness of 0.51 µm and 0.30 µm can be obtained for respectively, the 16 and 70 mm high samples. These values look promising for the implementation of this material in the industry. Furthermore the variation on the roughness is lower for the 70 mm high samples on the final finishing cuts (cut 5 and 6). However burn marks are still present in some finishing regimes, future work will focus on optimizing settings reducing these marks and further improving surface quality. Moreover the 5^{th} cut can be omitted for both heights, since it increases the roughness in comparison to the previous cut.

Finally the flexural strength of samples produced with different finishing cuts is tested, by means of a 4 point bending test. As SEM images showed cracks on the EDM machined surfaces it is expected beforehand that strength will be lower compared to ground samples coming from the same blank material. 4 Point bending tests revealed that only the minimal strength seems to increase with a higher finishing cut, no clear trend is visible in the average and maximal flexural strength. Although these test results should be dealt with carefully seeing only 3 replica are produced per finishing cut. More replica will need to be produced in order to reach final conclusions on the strength per finishing. To conclude a Weibull plot of all EDM samples reveals a decrease of about 500 MPa, regardless of which EDM finishing is used. This value is rather disappointing, presumably because the first cuts are also included in the Weibull plot and these samples show larger cracks as compared to the high finished samples.

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