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INFLUENCE OF PROCESSING PARAMETERS ON THE LASER MICROMACHINING AND TEXTURING OF ZIRCONIA (ZIRCONIUM DIOXIDE) 3Y-TZP COMPACTS DOPED WITH GRAPHITE

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Abstract. Ceramic materials are interesting for certain applications due to its properties, such as: low thermal conductivity, excellent wear resistance and chemical stability. Zirconia (ZrO₂) (3Y-TZP), for instance, stands as an example of high strength ceramic that has been extensively used as a material in dental and hip implants due to its outstanding mechanical properties, biocompatibility and aesthetics. However, mechanical and physical properties like thermal resistance, high hardness and brittleness, make ceramics, such as 3Y-TZP, difficult to shape and to work using conventional cutting tools, especially on its sintered form. In this sense, non-conventional machining processes, such as laser beam machining (LBM), are being tested on these materials. The aim of this work, was to optimize the process of laser micro-machining on green zirconia (3Y-TZP) by adding graphite powders on different contents (0.5%, 1% and 5% wt%) aiming at improving optical and thermal properties (higher light absorption and thermal conductivity). Different laser parameters were tested (laser speed, power, pulse duration) in order to study how the laser-matter interactions affect the characteristics of the laser machined/textured surfaces. Specimens were analyzed using Scanning Electron Microscopy (SEM) and White Light Interferometry (WLI).

Keywords: Zirconia, Graphite, Laser Machining, Green Ceramics

1.INTRODUCTION

Ceramic materials are interesting for certain applications due to its properties, such as: low thermal conductivity, excellent wear resistance and chemical stability. Zirconia (3Y-TZP), for instance, stands as an example of high strength ceramic that has been extensively used as a material in dental and hip implants due to its outstanding mechanical properties, biocompatibility and aesthetics (SOON et al., 2016).

Removal material techniques are important in mechanical engineering process; besides, the creation of textured surfaces has shown to be an useful process for implants. (MOURA, et al., 2020). However, for ceramic material the majority of traditional processes are ineffective due to material properties, mostly its hardness and mechanical resistance,

leading to higher processing time and costs. Therefore, studies involving laser micromachining and texturing on green zirconia are being developed (LIU et al., 2017). Mostly because of its contactless nature, automation capability and application effectiveness (SAMANTRA; DAHOTRE, 2008).

The laser equipment available for this work was mostly used in metallic materials, and it was not compatible with the low thermal conductivity and hard ceramics. For that reason, the zirconia (3Y-TZP) compacts were doped with graphite, aiming at studying its influence on thermal conductivity and laser absorption. Besides the material, the laser parameters strongly influence the final result, diversifying on laser speed, power and pulse duration (LIU et al., 2017). Adding higher amount of graphite can result on porosity on the final sintered material.

The graphite and zirconia (3Y-TZP) powder can be mixed with different techniques, such as mechanical and ultrasonic mixing. For this study the two techniques were consider, both with its vantages and disadvantages: the ultrasonic mix usually results in more homogenic compacts, however the particles properties of the zirconia (3Y-TZP) are damaged on the process, affecting rheological and compaction properties. The mechanical technique maintains these characteristics.

The aim of this work, was to optimize the process of laser micro-machining on green zirconia (3Y-TZP) by adding graphite powders on different contents aiming at improving optical and thermal properties (higher light absorption and thermal conductivity), and testing different lasers parameters, in order to choose the most effective parameters.

2. MATERIALS AND METHODS

2.1 GRAPHITE AND ZIRCONIA MIX AND COMPACTS COLD PRESS

This work used zircornia (3Y-TZP) – (99% - Tosoh) with atomized particles with average diameter of 60 μm and Graphite (Nacional de Grafite Ltda. – Micrograf 9905UJ) with average diameter of 5.5 μm .

The samples were prepared from the mechanical mixture of zirconia powder and Graphite. First, the mixtures were weighted and distributed in four proportions, 0%, 0.5%, 1% and 5% (% wt) of graphite. The mixtures were placed in plastic jars and taken to the roller mill, where they remained for 12 hours. Using a hydraulic press, 2.82g discs were dry pressed, with a load of 1.5 ton for 30s, in a 20mm diameter steel matrix. In this process 16 discs were manufactured: 4 discs for texturing and 12 more for the density test (following the proportions of 1 disc per Graphite content).

2.2 THERMAL CONDUCTIVITY TEST

To verify the effectiveness of the Graphite and zirconia mixture in the thermal conductivity measurements, two samples were prepared, one with 5% graphite in weight and the other of pure zirconia. 4g of powder were dry pressed into 30mm diameter matrix using a hydraulic press of 1.5ton load for 60s. The thermal conductivity test was performed on both sides of the samples using thermal conductivity analyzer (TCi C-Therm).

2.3 LASER TEXTURING AND MICROMACHINING

The green zirconia compacts were subjected to surface texturing, using an Ytterbium Pulsed Fiber Laser model YLPN-1-1x120-50-M (LMP-UFSC) (Figure 1). In all processes a pulse repetition rate (PRR) of 50kHz was used, which is the lowest rate that the equipment works and a wavelength of 1064nm.



Figure 1. YLPN-1-1x120-50-M (Ytterbium Pulsed Fiber Laser)

To test different parameters on the samples and for better use of the area space, matrices (5x5) of small squares with dimensions of 500 μm x 500 μm were drawn with different laser parameters. For each sample, with different graphite concentrations, two matrices were made with different pulse durations (8ns, 30ns) and inside the matrix the parameters change vertically, bottom up, in speed (200 mm/s – 1000 mm/s) and horizontally, left to right, in power (10 W – 50 W), as shown in the table below (Table 1)

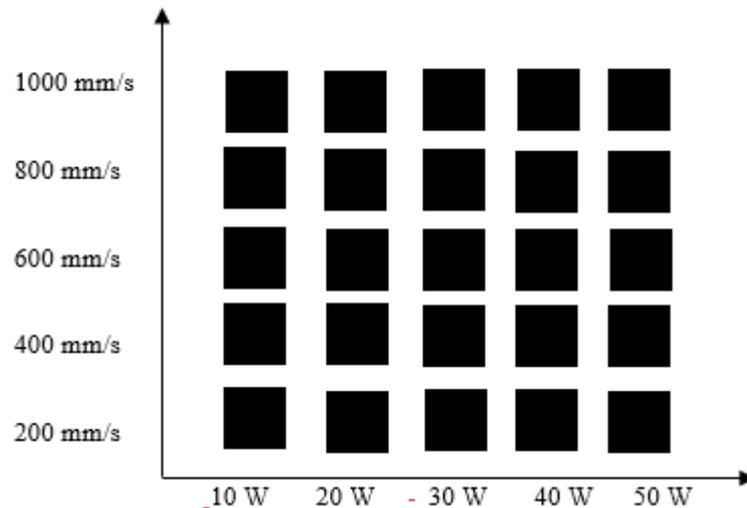


Table 1. Example texturing matrix made to evaluate the effect of different laser parameters, where each square has a different speed and power combination.

Samples that were not textured went through the sintering process. These samples were sintered in a high temperature furnace (EDG – Labconf – UFSC) at a sintering temperature of 1500°C, with a heating rate of 5°C/min, maintaining the maximum temperature for 2 hours. In the sintering process, with the high temperature and an atmosphere with air, graphite is reduced by oxygen in the air and totally removed from the samples, resulting in pure zirconia samples.

2.5 DENSITY BY ARCHIMEDES METHOD

The sintered samples (12) went through the density measurement process by the method of Archimedes, to determine the possible formation of porosity in the samples. First the samples were dry weighed ($w1$), then they were placed in a warming Becker with boiling water for 30 minutes, then they were again weighed ($w2$), and finally they were weighed using an auxiliary device, with a basket immersed in water, inside a Becker on the analytical balance ($w3$). With this data it was possible to measure the density of the samples using the equation $(d*w1) / (w2- w3)$, where d is the liquid density.

2.6 MICROSTRUCTURE AND ROUGHNESS CHARACTERIZATION

The surfaces of the samples were analyzed by a Scanning Electron Microscopy – SEM (TM3030 – Hitachi), with tungsten filament. The samples were coated with a micrometric layer of Carbon to allow electrical conduction. Preliminary roughness measurements were also conducted on some samples using a white light interferometer (Zygo NewView 7300 – Labmat UFSC).

3. RESULTS AND DISCUSSION:

3.1 THERMAL CONDUCTIVITY ANALYSES

The thermal conductivity test of green samples showed an increase of this property for compacts with a graphite content of 5% compared to pure zirconia. Using the average of the k factor (W/mK) for both graphite grades, it was possible to observe an average value of 0.398 W/mK for the sample with 5% graphite and an average value of 0.132 W/mK for pure zirconia. Therefore, zirconia/graphite samples presented a 3-fold k value of that of pure zirconia samples (Table 2).

Sample	Effusivity (Ws ^{1/2} /m ² K)	Thermal conductivity k (W/mK)	R ²	Ambient T°C
ZrO ₂ +Graphite	ε=956,3	k=0,301		
Sample	ε=954,8	k=0,298	0,9996	22,8
(Side 1)	ε=955,9	k=0,300		
ZrO ₂ +Graphite	ε=1088,8	k=0,511		
Sample	ε=1079,3	k=0,496	0,9995	22,9
(Side 2)	ε=1071,1	k=0,483		
ZrO ₂ Sample	ε=879,3	k=0,180		
(Side 1)	ε=888,1	k=0,194	0,9995	23,1
	ε=886,7	k=0,192		
ZrO ₂ Sample	ε=807,7	k=0,070		
(Side 2)	ε=813,5	k=0,079	0,9995	23,2
	ε=813,5	k=0,078		

Table 2. Thermal conductivity measurement on no sintered samples.

3.2 DENSITY TEST BY ARCHIMEDES METHOD

The density test using the Archimedes method on sintered samples presented a considerable decrease in density in samples with content of graphite above 1% (Figure 2). Samples with graphite content of 5% reached an average density of 5.47 g/cm³, which represents 90.48% of the pure zirconia disc density (6.05 g/cm³), can impair the mechanical properties of the final product. That has probably occurred due to the formation of porosities resulting from the graphite removal from the discs during the sintering process.

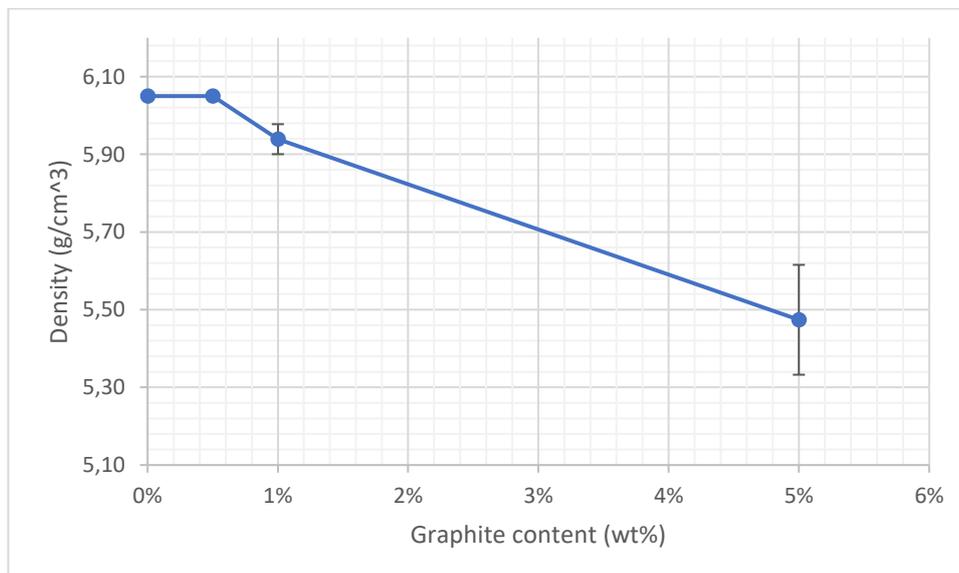


Figure 2. Density measurement of the sintered samples.

3.3 INFLUENCE OF LASER PARAMETERS ON THE QUALITY OF TEXTURED AND MICROMACHINED ZIRCONIA SURFACE

3.3.1 UNWANTED PHENOMENA IN THE LASER REACTION WITH THE MATERIAL

The heat generated by the different laser parameter showed different reactions in the material. Besides the desired material removal, some heat zones beyond the limits were created, that may generate cracks and damage the final product in its mechanical properties and aesthetic values (Figure 3a). It is also possible to notice that at speeds below 400mm/s other phenomena makes the process unfeasible in such parameters.

Using a closer approximation in a groove that has been processed using 400 mm/s and 40 W (Figure 3b), it can be noticed a different phenomenon that have occurred, such as loss of shape of the machined object, irregularities in the depth of the groove, and possibly formation of unwanted sintered particles.

The different laser reactions using different parameters in ceramic materials, such as fusion, sublimation, vaporization, dissociation, plasma formations, ablation and formation of unwanted sintered layers are widely discussed in (LIU, et al. 2017) and served as a reference for this excerpt of the work.

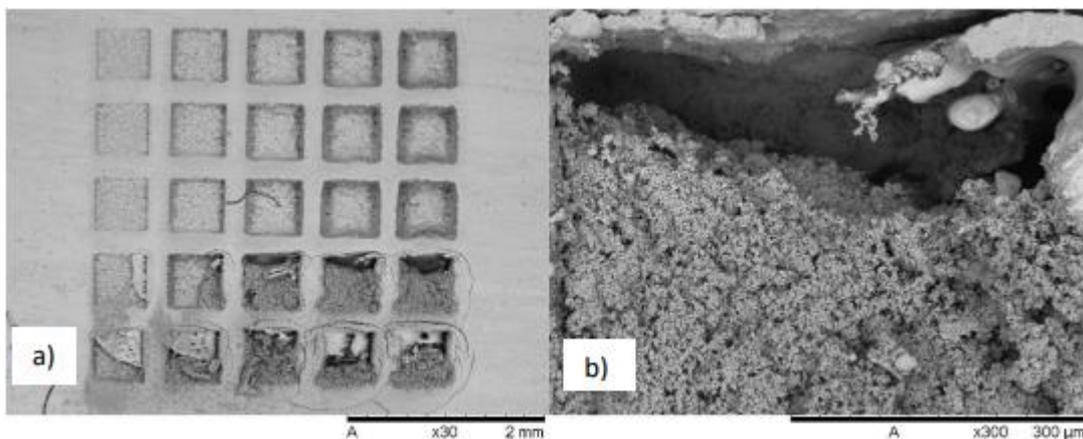


Figure 3. SEM images (a) 0.5% graphite sample, 30 ns pulse duration, speed between 1000 mm/s – 200 mm/s and power between 10 W – 50 W 30x magnification and (b) same sample, 300x magnification on the 400 mm/s e 40 W groove.

3.3.2 MORPHOLOGY OF THE TEXTURED/MACHINED SURFACE

At higher speeds, between 600 mm/s and 1000 mm/s, a better result was obtained in relation to the lower speeds, where removal of the material was achieved, as desired. It is possible to observe greater uniformity in the groove (Figure 4), however, the lateral edges showed a greater depth in relation to the remaining of the area (Figure 5). This occurred in virtually all parameters and it is probably justified by the laser trajectory which caused more damage to the extremities.

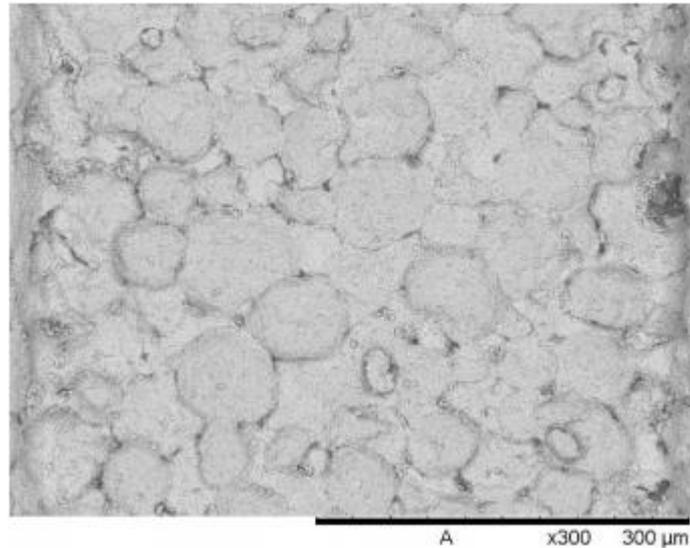


Figure 4. SEM: 1% graphite sample, 30 ns pulse duration, groove with 1000 mm/s and 10 W.

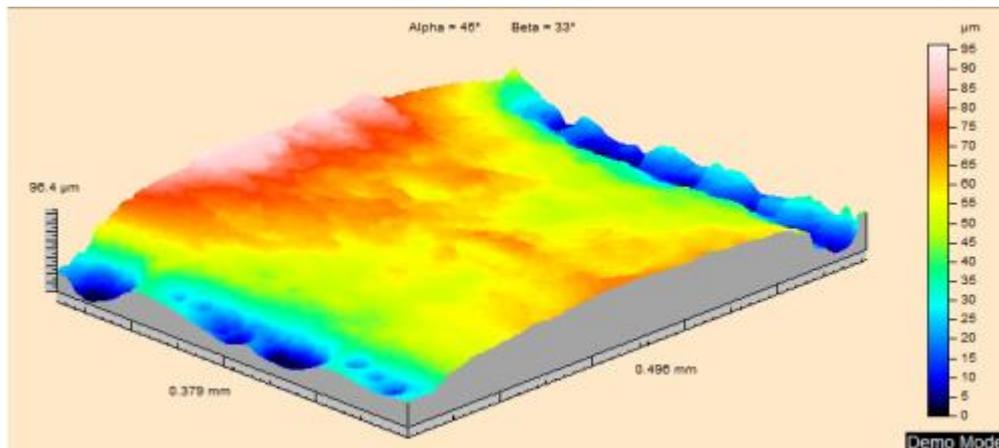


Figure 5. Interferometer image of one groove.

3.3.3 QUANTITATIVE ANALYSIS OF THE TOTAL AREA OF THE GROOVES

Looking at the effects at higher speeds, where the results were more satisfactory, it is notable the deformation of the area in some parameters, in general as the power increases, material is removed beyond what is desired at the edges. To quantify this increase of area, the total damaged area measurements were taken for each square that showed the desired phenomena in order to observe the influence of the parameters on the accuracy of the texturing on the material.

Using as reference the intention to create squares measuring 0.5 mm x 0.5 mm, the ideal total area would be 0.25 mm², this result was not obtained for any parameter. The closer to the ideal that were achieved ranged between 0.261 mm² and 0.266 mm², all of which were obtained for pure zirconia samples. For pulse durations of 8 ns (Figure 6), the best results were for parameters: 1000 mm/s and 20 W (0.264 mm²) and 30 W (0.264 mm²), 800 mm/s – 20 W (0.266 mm²) – 30 W (0.265 mm²), 600 mm/s – 20 W (0.265 mm²). For pulse durations of 30 ns (Figure 7), the best results were: 800 mm/s – 10W (0.261mm²), 600mm/s -10W (0.264mm²). These results shows that the graphite addition has low influence on the laser precision.

When analyzing the trends in area results, it is possible to observe, mainly for pulse durations of 30ns (Figure 7), the increase in the total area, i.e., deviations from the targeted, as the power increases. We can observe that areas change, for pure zirconia samples, with pulse durations of 30 ns and speed of 800 mm/s from 0,261 mm² (10 W) to 0.353 mm² (40 W) and for samples with 0.5% of graphite, with pulse duration of 30 ns and speed 800 mm/s the variation is from 0.282 mm² (10 W) to 0.348 mm² (40 W). It shows that the power has strong influence on the laser precision, mostly for higher pulse durations.

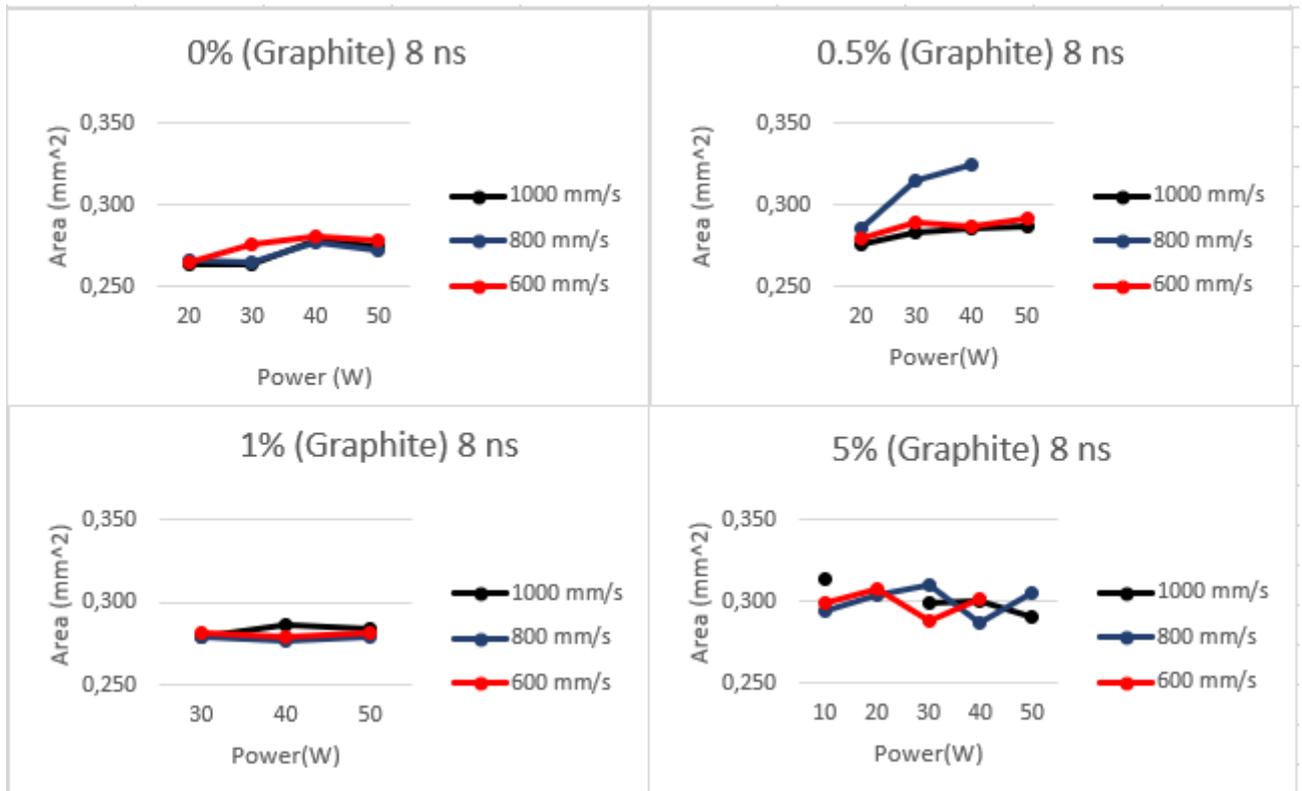


Figure 6. Area measurement for grooves using different laser parameters varying in speed and power with fixed pulse duration (8 ns), graphite content in weight (a) 0% (b) 0.5% (c) 1% (d) 5%

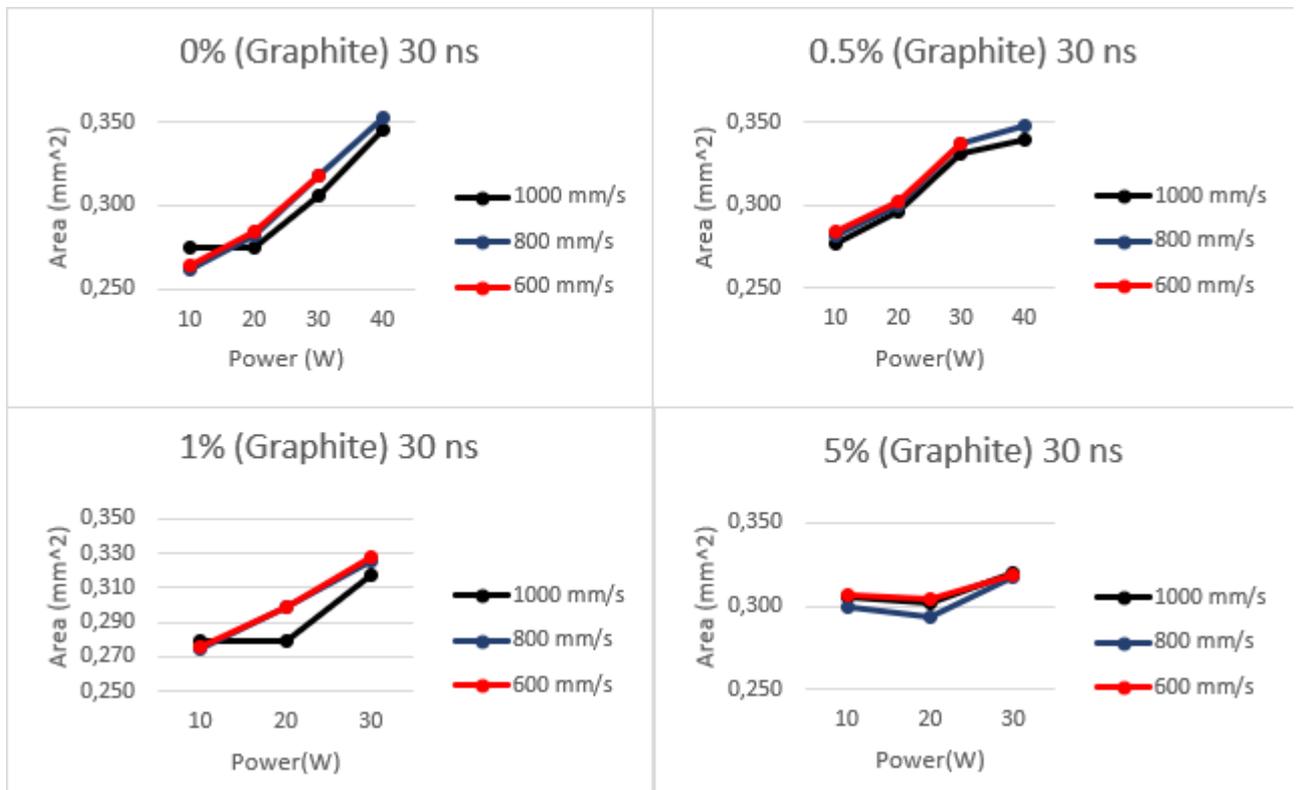


Figure 7. Area measurement for grooves using different laser parameters varying in speed and power with fixed pulse duration (30 ns), graphite content in weight (a) 0% (b) 0.5% (c) 1% (d) 5%

3.4 INFLUENCE OF GRAPHITE ADDITION ON OPTICAL PROPERTIES

Ceramics, such as zirconia, have a high reflectivity on their surface, this factor can also be harmful for laser absorption. Studies show that the addition of Graphite on the material can decrease its reflectivity and optimize the laser effect in the process (KUZIN, V. V.; GRIGOR'EV, S. N.; ERMOLIN, V. N. 2019). For the texturized samples on this process, as in the cited article, it was possible to observe the changing on the color of the compacts after the graphite addition, as showed on Figure 8.

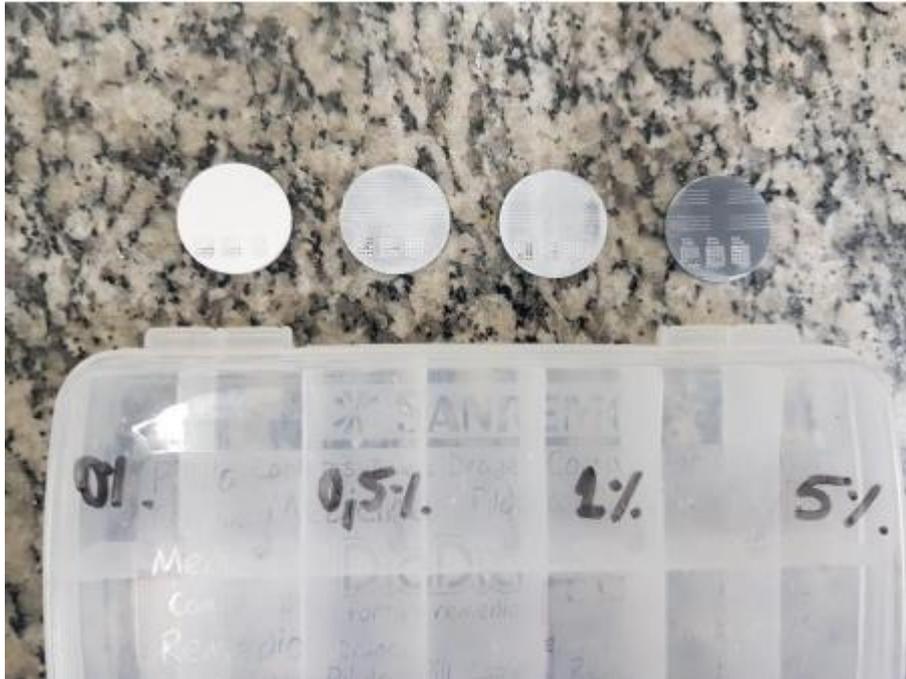


Figure 8. Photo of the textured, no sintered compacts with different graphite content.

We can observe that as the graphite content increases, the samples lose their whiteness and start getting a greyish coloration due to graphite. This factor can influence on the optical properties (reflectiveness), on the texturing process.

4. CONCLUSION

- Compacts with 5% graphite in weight showed higher thermal conductivity on their surface when compared with pure zirconia.
- Sintered compacts showed considerable density decrease for graphite contents of 1% and 5%.
- Textures with the parameters used in the present work showed unwanted phenomena for speeds below 400 mm/s.
- The increase of the power has the tendency to increase the unwanted removal of material, mostly for pulse durations lower than 30 ns.
- The addition of graphite to the compacts has shown no influence on the laser precision.

5. ACKNOWLEDGEMENTS

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7.RESPONSIBILITY NOTICE

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