27th ABCM International Congress of Mechanical Engineering
December 04-08, 2023, Florianópolis-SC, Brazil

COB-2023-2424
INFLUENCE OF PROCESSING PARAMETERS ON THE LASER MICROMACHINING AND TEXTURING OF3Y-TZP COMPACTS DOPED WITH GRAPHITE

Pedro Griebeler Oliveira
Douglas Fabris
Márcio Celso Fredel
Bruno Alexandre Pacheco de Castro Henriques
Ceramic & Composite Materials Research Laboratory (CERMAT), Mechanical Engineering Department, Federal University of Santa Catarina, Brazil
pedrogriebeler@gmail.com, douglas.df1@gmail.com, m.fredel@ufsc.com, bruno.henriques@ufsc.com

Claudio Abilio da Silveira
Milton Pereira
Precision Engineering Laboratory (LMP), Mechanical Engineering Department, Federal University of Santa Catarina, Brazil
claudio_ards@hotmail.com, milton.pereira@ufsc.br

Abstract. *Ceramic materials are interesting for certain applications due to their 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. However, mechanical and physical properties 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 Yttria stabilized 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). A 1064 nm nanosecond-pulsed Direct Laser Writing (DLW) system was used to produce squared grooves in compact green samples. Laser parameters were varied to evaluate its influence. The material removal behavior, surface characteristic and material behavior were investigated experimentally. It was found that material doped with graphite had higher material removal rates than pure Zirconia material. However, graphite composites showed lower surface quality.*

Keywords: *Zirconia, Graphite, Laser Micromachining, Green Ceramics*

1. INTRODUCTION

Zirconia (ZrO2) has achieved considerable attention in various domains owing to its distinctive properties, such as high strength, toughness, hardness and wear resistance, as well as its high temperatures and corrosive environments performance. These properties make Zirconia interesting for distinguished applications, such as reinforcement of metal-based composites (Parveez et al., 2021) thermal barrier coatings (Liu et al., 2019) and for biomedical applications, as dental application and hip implants (Reveron et al., 2021). Tetragonal Polycrystalline Zirconia stabilized with 3 mol% Y2O3 (3Y-TZP) is the most widely utilized and extensively researched option for biomedical application mostly at dental and hip implants, as it stands as an example of high strength ceramic (up to 1200 MPa) and aesthetics, besides its biocompatibility.

The inefficacy of traditional machining processes on ceramics is primarily attributed to the inherent properties of the material, notably its hardness and mechanical resistance (Kobayashi et al., 1989), leading to higher processing time and costs. Owing to its properties mechanical machining have limitations on ceramics materials, for example: abrasive machining/grinding showed long machining times and high machining costs, besides mechanical weakness, caused by cracks formation, plastic deformation and residual stress (Dahotre). Ultrasonic machining (USM) has typically low material removal rate, tool vibrations and difficulty on parameters optimization. (Dahotre). Due to ceramics inherent brittleness, avoiding material defects is essential to ensure reliability besides good production efficiency, therefore, alternatives machining processes have been discussed, such as electrical discharge machining (EDM), laser beam machining (LBM).

LBM has particularly attracted significant interest since it is a contactless process, eliminating concerns of material contamination, tool wear and machine vibration. Moreover, LBM offers precise laser parameters control, enabling high material removal rate while minimizing material damage. LBM has shown as well to be a flexible process, as it can be easily automated for various machining techniques and its parameters can be easily controlled and replicated. These significant benefits reinforce the advantages of Laser Beam Machining, establishing it as a highly advantageous technique in the realm of material processing. In the laser processing technique, the surface of the workpiece is exposed to intense optical energy of high density, resulting in localized material removal through processes such as melting, dissociation, and/or evaporation (Samant et al., 2009).

Nevertheless, some limitations may arise on Zirconia laser treatment. This includes the risk of excessive heating, laser-induced phase transformation and crack formation (Han et al., 2022). Besides laser parameters, material properties such as thermal conductivity and laser absorption influence laser ablation since higher material removal rate can be achieved when variating these properties. To do so, composites materials are being studied to understand the different results on laser processing in different material compositions (Han et al., 2022) and trying to achieve an optimal pattern as higher thermal conductivity are more susceptible to thermal cracking under excessive laser energy input on the other hand can present higher material removal rate and efficiency.

Zirconia ceramics doped with Carbon based material have been studied aiming to improve electro-thermal properties (Muñoz-Ferreiro et at., 2022). In this work Zirconia (3Y-TZP) doped with graphite powder on different contents aiming at improving optical and thermal properties (higher light absorption and thermal conductivity). Afterwards the samples were processed with different laser parameters, in order to choose the most effective parameters for useful applications and analyzed composite consequences on laser ablation.

2. MATERIALS AND METHODS

In this work were used Zirconia (3Y-TZP) – (99% - Tosoh) with atomized particles with average diameter of 60 µm and Graphite (Nacional de Grafite Ltda. – Micrograf 9905UJ) with an average diameter of 5.5 µm. The mixtures, containing 0.5wt%, 1wt% and 5wt%, were placed in plastic jars and taken to the roller mill, where they remained for 12 hours. The 2,92 g powders were uniaxially pressed in 20 mm steel dies at 1.5 Ton for 30s to obtain the green compacts. The green zirconia compacts were subjected to surface texturing, using an Ytterbium Pulsed Fiber Laser model YLPN-1-1x120-50-M (Figure 1).

A pulse repetition rate (PRR) of 50kHz was used and a wavelength of 1064 nm. Direct Laser Writing (DLW) technique was used to ablate matrices (5x5) of small squares with dimensions of 500 µm x 500 µm with different laser parameters. For each sample, with different graphite concentrations, one matrix was made with different 8ns pulse duration 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 Figure 1.

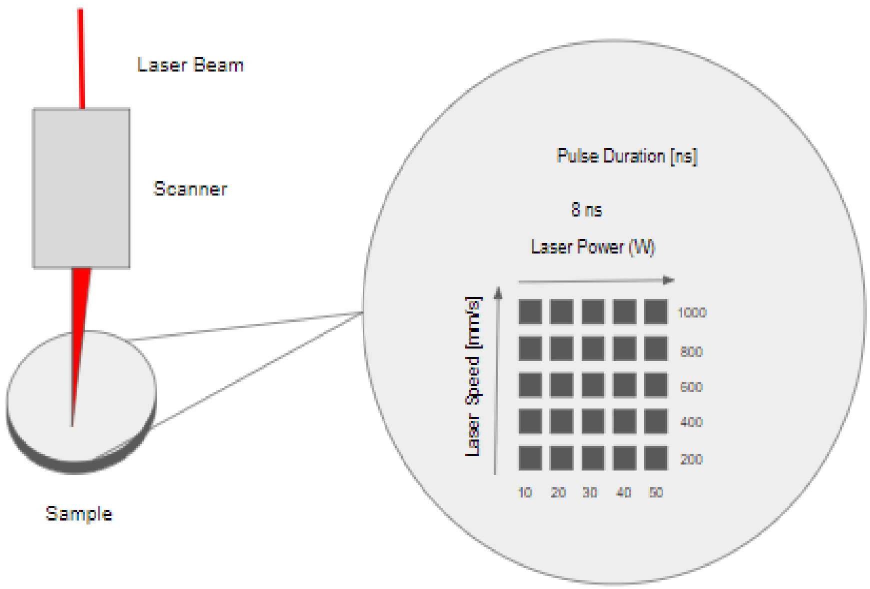

Figure 1. Schematic diagram of DLW parameters.

Afterwards, the green compacts were sintered in a high temperature furnace in air at 1500°C for 2h. For examination of the topography of the ablated grooves, a white light interferometer (Zygo NewView 7300) was used on green and synthesized samples. The morphology of the treated regions was analyzed using a Scanning Electron Microscope (TM-3030, Hitachi) at an operating voltage of 15.0 kV.

3. RESULTS AND DISCUSSION

The heat generated by the different laser parameters 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 2a). It is also possible to notice that, at speeds below 400mm/s other phenomena make 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 2b), 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.

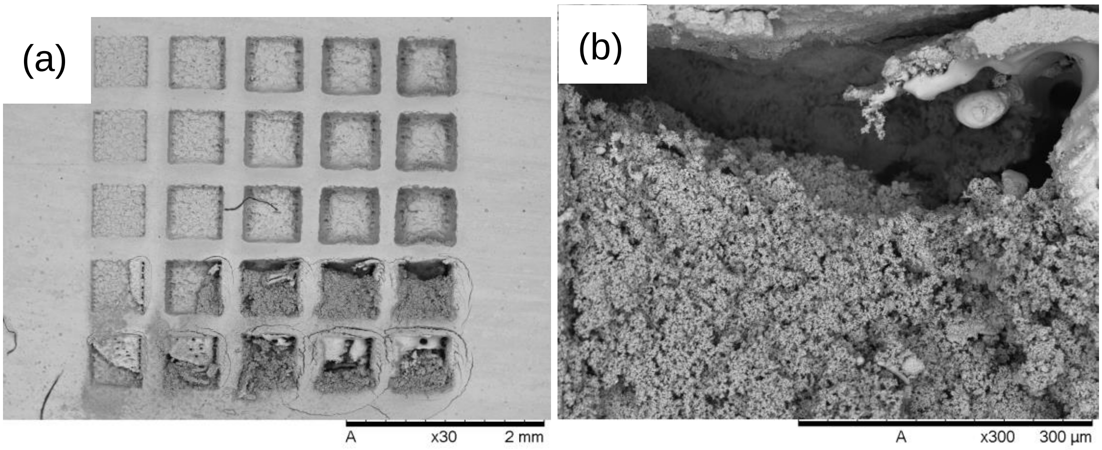

Figure 2. SEM images of (a) 0.5% graphite sample, 30 ns pulse duration, speed of 1000 mm/s – 200 mm/s and power of 10W – 50W 30x magnification and (b) same sample, 300x magnification for 400mm/s and 40W.

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 3), however, the lateral edges showed a greater depth in relation to the remaining surface (Figure 4). This occurred in virtually all parameters, and it is probably justified by the laser trajectory which caused more damage to the extremities. The laser trajectory alters its path along the lateral edges. Such a trajectory modification engenders a deliberate concentration of thermal energy along the edges, thereby amplifying the thermodynamic effects on these extremities. Consequently, this configuration manifests an elevated thermal effect that culminates in a higher material removal rate.

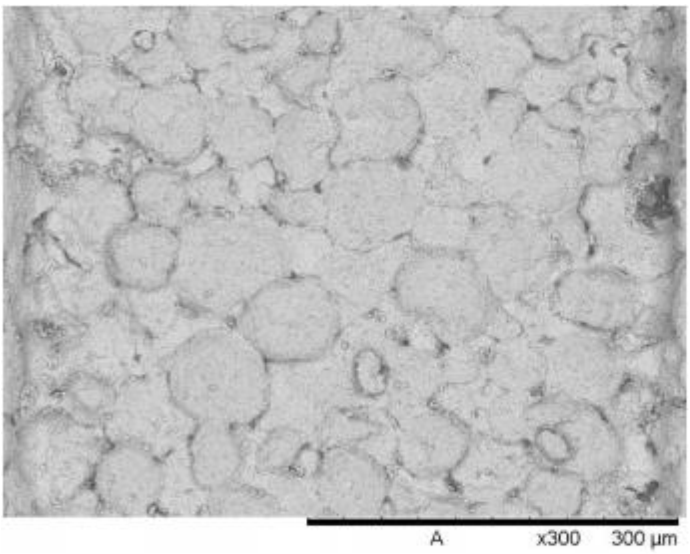

Figure 3. SEM: 1% graphite sample, 30ns pulse duration, groove with 1000 mm/s and 10 W.

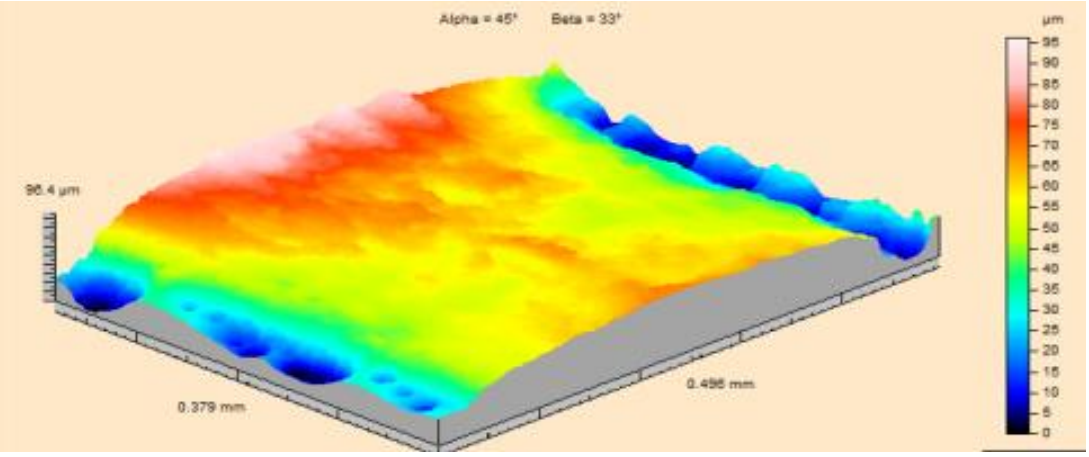

Figure 4. Interferometer image of one groove.

Surface irregularities were noted in morphology analyses of doped graphite samples, distinguishing them from pure zirconia, suggesting that these variations could potentially arise from the intricate process of graphite-zirconia mechanical mixture (Figure 5).

The combination of two different material powders amplifies the differences in particle size and can impede the effectiveness of mixing. This increased variation in particle dimensions can hinder the equal distribution of materials in the mixture, resulting in regions of inconsistent composition.

This phenomenon not only affects the powder mixing process but also has a direct impact on compaction. Inadequate blending leads to areas of the mixture being denser or sparser, causing uneven compaction and suboptimal material distribution during subsequent processing steps. Consequently, this irregular compaction manifests in the final surface quality, with variations in material density and composition often resulting in surface imperfections and compromised structural integrity.

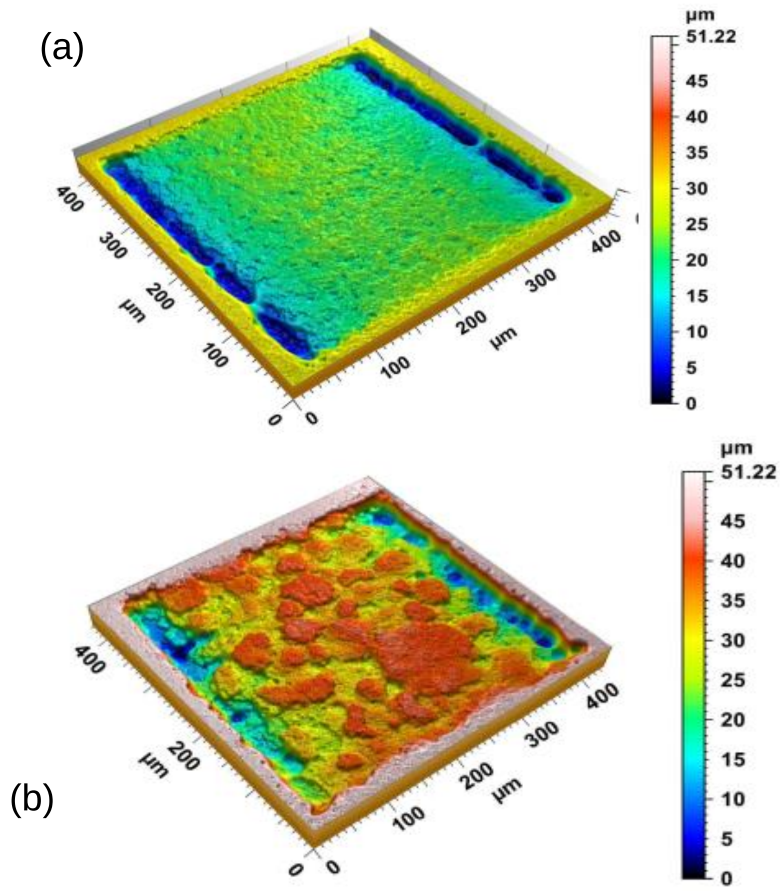

Figure 5. Interferometer image showing samples with different graphite compositions (%wt). (a) 0% graphite (b) 0.5%. Using laser parameters of 800mm/s and 30 W.

The full material ablation, calculated using the average depth area, showed that graphite doped samples had larger material removal, as shown on figure 6.

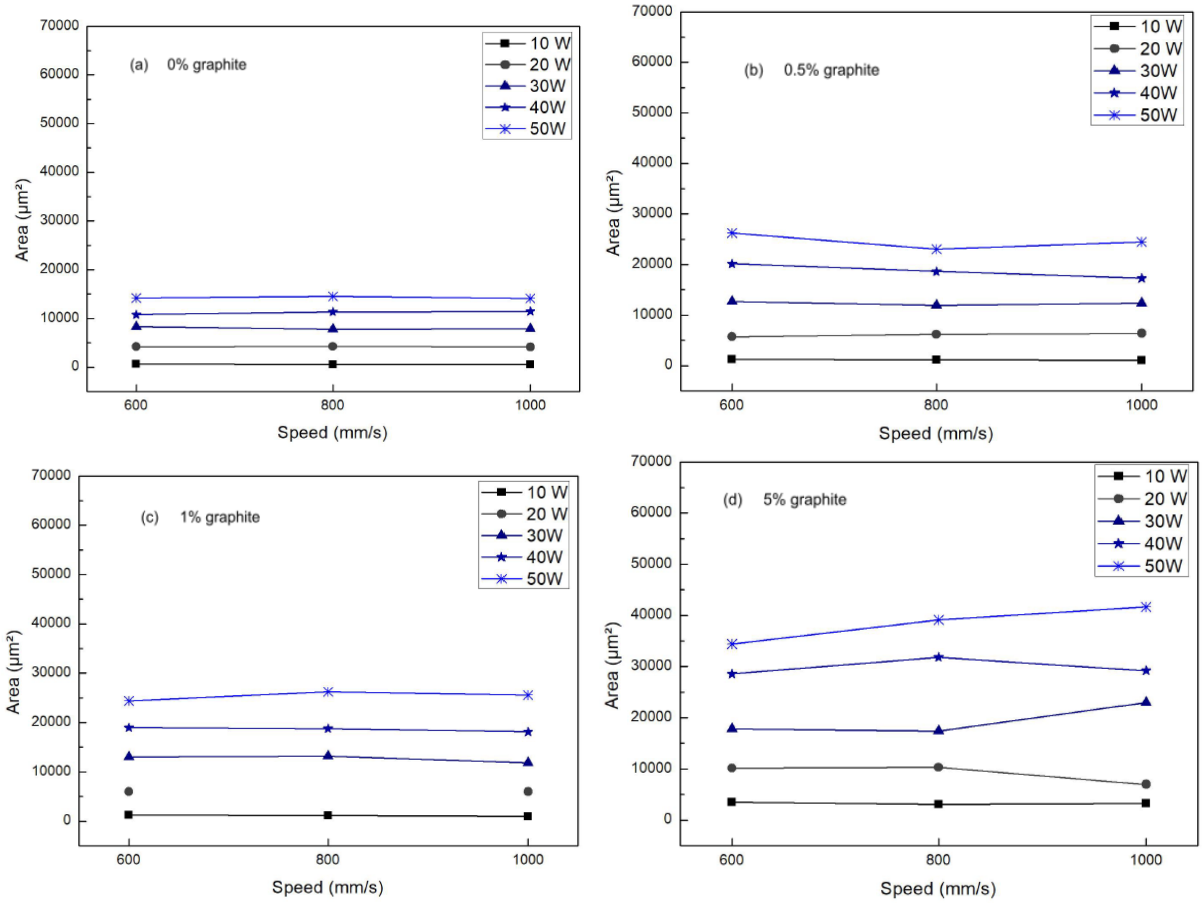

Figure 6. Grooves removal area measurement, for different laser parameters, changing on speed (1000 mm/s - 600 mm/s) and power (10 W – 50 W) with constant pulse duration of 8 ns. (a) 0% graphite (b) 0.5% graphite (c) 1% graphite (d) 5% graphite.

The notably higher material removal rate observed in samples doped with graphite can likely be attributed to the enhanced heat absorption characteristics inherent to graphite. Graphite's distinct thermal properties, including its superior heat transfer and optical attributes, set it apart as a material capable of absorbing and retaining heat more effectively than pure Zirconia. This increased heat absorption translates into a heightened thermal energy reservoir that can be efficiently utilized in material removal processes. As a result, the elevated material removal rate in graphite-doped samples can be reasonably explained by the advantageous heat management properties of graphite, which enable more efficient and effective machining.

On the same analyses, it can be observed that power parameter is more determinant for ablation than laser speed as at all process variations, depth volume grows with power increase while it remains virtually constant with speed increase. Graphite doped samples showed irregular results as the graphite % grows, making the process less predictable.

Extracting a useful area from ablated grooves, the surface average (SA) was used to analyze the surface roughness, this analysis reinforces the irregularities caused by the graphite mixtures, reaching a maximum value of 8.35µm on 50W e 600 mm/s groove of 5% graphite samples, meanwhile the minimum SA value was measured on pure zirconia samples, of 0.576 µm on 10W e 800 mm/s groove.

After sintering the material, due to graphite reduction on air atmosphere and material contraction, differences on the material morphology could be shown, so the same roughness analysis was made. Similar results can be found when compared with green samples, considering the contraction of the materials.

4. CONCLUSIONS

In this work, laser ablation of pure 3Y-TZP and doped graphite material using a Direct Laser Writing system equipped working at a wavelength of 1064 nm was investigated. The influence of laser pulse energy, pulse repetition rate and scanning speed on the ablated grooves profile and morphology for both materials was analyzed. The following conclusions can be drawn from this work:

- Laser ablation with the parameters used in the present work showed unwanted phenomena for speeds below 400mm/s
- The increase of the power has the tendency to increase the unwanted removal of material, mostly for pulse durations higher than 30ns
- The addition of graphite to the compacts have shown no influence on the laser precision.
- Laser power strongly increases the depth groove, meanwhile laser speed has few influences on the results.
- Graphite doped materials were more damaged by laser ablation, resulting in higher material removal.
- Sintered material had similar morphology of the grooves, keeping similar roughness properties.

5. ACKNOWLEDGEMENTS

This study was supported by the Capes-Humboldt Research Fellowship Programme (Ref: 88881.197684/2018-01), FCT-Portugal (through the reference projects UIDB/04436/2020 and UIDP/04436/2020)), by the Capes/DAAD PROBRAL Programme (Ref: 88887.628082/2021-00), FINEP-Brazil (Project BIOMIO – 01.22.0180.00 (0027/21)), by Foundation for the Support of Research and Innovation of the State of Santa Catarina – FAPESC (ref. 2021TR001461) and by National Council for Scientific and Technological Development – CNPq.

6. REFERENCES

Kobayashi, A., 1989. Precision Machining Methods for Ceramics. In *Advanced Technical Ceramics* (pp. 261–313). Elsevier.

Liu, Q., Huang, S., He, A., 2019. Composite ceramics thermal barrier coatings of yttria stabilized zirconia for aero-engines. *Journal of Materials Science & Technology*, 35(12), 2814–2823.

Liu, Y., Liu, L., Deng, J., Meng, R., Zou, X., Wu, F., 2017. Fabrication of micro-scale textured grooves on green ZrO 2 ceramics by pulsed laser ablation. *Ceramics International*, 43(8), 6519–6531.

Muñoz-Ferreiro, C., López-Pernía, C., Moriche, R., Gommeringer, A., Kern, F., Poyato, R., Gallardo-López, Á., 2022. Highly efficient electrical discharge machining of yttria-stabilized zirconia ceramics with graphene nanostructures as fillers. *Journal of the European Ceramic Society*, 42(13), 5943–5952.

Parveez, B., Wani, M. F., 2021. Tribological behavior of nano-zirconia reinforced iron-based self-lubricating composites for bearing applications. *Tribology International*, 159, 106969.

Reveron, H., Chevalier, J., 2021. Yttria-Stabilized Zirconia as a Biomaterial: From Orthopedic Towards Dental Applications. In *Encyclopedia of Materials: Technical Ceramics and Glasses* (pp. 540–552). Elsevier. Samant, A. N., Dahotre, N. B., 2009. Laser machining of structural ceramics—A review. *Journal of the European Ceramic Society*, 29(6), 969–993.

7. RESPONSIBILITY NOTICE

The authors are the only responsible for the printed material included in this paper.