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# EFFECT OF ANISOTROPY OF ZIRCONIA PINS PRODUCED BY ADDITIVE MANUFACTURING ON THE WEAR RESISTANCE

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*Abstract. Zirconia has a wide range of applications due to its hardness and fracture toughness, which provide high wear resistance. Dentistry applications, such as prosthetic crowns and orthodontic brackets appeal to zirconia motivated by its aesthetics. Brackets are a type of orthodontic accessory that undergoes shear frictional force resulting from the passage of a metallic wire through the bracket slot imposing high tangential force. On the other hand, ceramic additive manufacturing (AM) produces anisotropic parts. This work aims to evaluate the influence of anisotropy on the wear resistance of zirconia. Pins produced by AM (in parallel and perpendicular layer deposition directions) were subjected to shear forces on a "pin-on-disk" dry wear test, applying a normal load of 10 N and a sliding speed of 1 m/s. The wear resistance of pins is classically represented by equations that indirectly involve hardness and fracture toughness, and can be used for an indirect assessment of fracture toughness relative to the adhesion between layers. In the first 500m, the perpendicular direction presented wear rate 2,5 times greater than the parallel one. The wear rate difference starts to decrease and gets similar rates for both samples from 2000m. Therefore, the total mass loss after 3000m of the perpendicular direction was more than 50% higher, evidencing anisotropic wear of the ceramic pins produced by AM.*

*Keywords: Additive Manufacturing, Anisotropy, Zirconia Ceramic, Wear Resistance.*

## 1. INTRODUCTION

Additive manufacturing (AM) is the term used to define the process of producing parts by joining parts layer by layer from a three-dimensional digital model (Zocca et al., 2015). This method is used in polymers, metals, ceramics and composites and allows the production of complex, reduced-size parts while maintaining the physical and mechanical properties obtained by traditional methods (Lovo et al., 2020). AM by vat photopolymerization (VP) has gained ground in recent decades, especially with the digital light projection (DLP) technique, as it is more accessible and affordable (Mozón et al., 2017). In this process, a light source projects the layer onto a mobile platform located in a vat that contains the photopolymerizable monomer. The region reached by the projection undergoes polymerization, forming the first layer of the part. The platform moves downward so that the polymerized layer is submerged in the vat (top-down method) and then one more projection is made and one more layer is cured. Thus, successively, the layers are formed and stacked in order to form the desired three-dimensional piece (Galante et al., 2019).

In ceramics, this method is advantageous in terms of reducing the need to use machinery and allowing the production of parts that are impossible to make using other methods, such as subtractive manufacturing. In addition, AM by VP is one of the few processes that can result in high-performance ceramics, high resolution and low surface roughness (Xing et al., 2017). Zirconia is a ceramic of high hardness, flexural strength and fracture, presenting biocompatibility, thermal stability and desirable aesthetics. These characteristics make zirconia a product used in medicine and dentistry, such as in dental implants, prostheses and dental crowns (Zhang et al., 2020).

During the AM process, the layers are formed in a certain direction, which gives the final part anisotropic behavior. The anisotropy, in the case of ceramic suspensions, is also influenced by the shape of the ceramic powder particles, since the more it deviates from a sphere, the greater the anisotropy (Nakai, H. et al., 2021). Anisotropy had already been observed by Ahn et al (2003) in the melted material extrusion process, which shows the influence of the layer deposition direction on the mechanical characteristics of the part. In jetting technology (Kesy and Kotlinsky, 2016) (Cazón, A., et al., 2014) and stereolithography (Puebla, K., et al., 2012) anisotropy was also investigated. However, for the DLP process, little has been studied about the influence of anisotropy on the mechanical properties of the final parts, mainly regarding the effect of anisotropy on wear.

3Y-TZP zirconia is a ceramic group with high hardness and fracture toughness, generally applied as subject parts to wear such as dentistry, textile wire guides, mechanical seals, shoe bearings, and drawing nozzles (Patil, A., et al., 2023). The action of shear forces caused by the relative movement between two surfaces in contact generates shear stresses parallel to the surface and may be influenced by the anisotropy arising from the additive manufacturing process, due to the deposition of layers where an interface is found between them. This issue may be mitigated by applying the Design for Additive Manufacturing (DFAM) concept (Vaneker et al., 2020), building the parts considering the direction of the expected acting loads.

The study on wear resistance in ceramic-based composites is related to their tribological behavior. Normally, the wear of these materials is verified by a frictional movement resulting from the contact between a ceramic sphere or pin on a disk (Xiong, 1999). In ceramic materials, friction generates cracks around the contact zone, which can be lateral, median and radial hertzian (Arantes, V.L., et al., 2021). The friction and wear values and of the ceramic materials are influenced by the chosen test parameters, such as the load applied to the ceramic specimen, disk speed, temperature, humidity and surface finish (Evans and Wilshaw, 1976). Thus, keeping the same parameters in all tests, it is possible to compare results in samples printed in different directions of layer deposition in order to have a reliable analysis.

In this work, two printing positions were performed and the wear test was directed to verify the effect of the friction force in the longitudinal and perpendicular/transversal directions to the deposited layers. As a counterproof, a Y-TZP Tosoh sphere was tested under the same conditions.

## 2. MATERIALS AND METHODS

The following materials were part of the preparation of the formulation of the ceramic suspension used for the production of wear specimens: Zirconia powder with a specific surface area of $16 \pm 3$ m$^2$/g – (TZ-3Y-E, Tosoh Corporation, Japan ); Photosensitive resins - Poly (ethylene glycol) diacrylate molecule (PEGDA 250, Sigma Aldrich, USA); Photoinitiator – Phenylbis (2,4,6-trimethylbenzoyl) phosphine oxide (PPO, Sigma Aldrich, USA); Dispersants - DISPERBYK - 111 (BYK - Chemie, Germany), Solvent: nmethyl-2-pyrrolidone (Synth, Labsynth, São Paulo).

To verify the influence of anisotropy on the wear of ceramics produced by AM, the pin-on-disk type test was selected, following the ASTM G99 - 05 standard. It is known that the wear rate and friction coefficients can be strongly influenced by the method used for the applied load, sliding speed, surface finish, surrounding environment, and temperature. Therefore, all these parameters were maintained during the tests so that the results are reliable and comparable.

The pins used for the wear test were obtained by additive manufacturing (AM) processes, using a custom-built VP 3D printer (top-down approach), with layer thickness of 50 μm, and exposition time of 10s per layer. The pins were manufactured in two distinct printing directions: perpendicular and parallel to the shear direction (Figure 1). The pins were sintered at 1500ºC for 2 hours and assembled on metallic support adhered with Araldite[TM] adhesive (Figure 2).
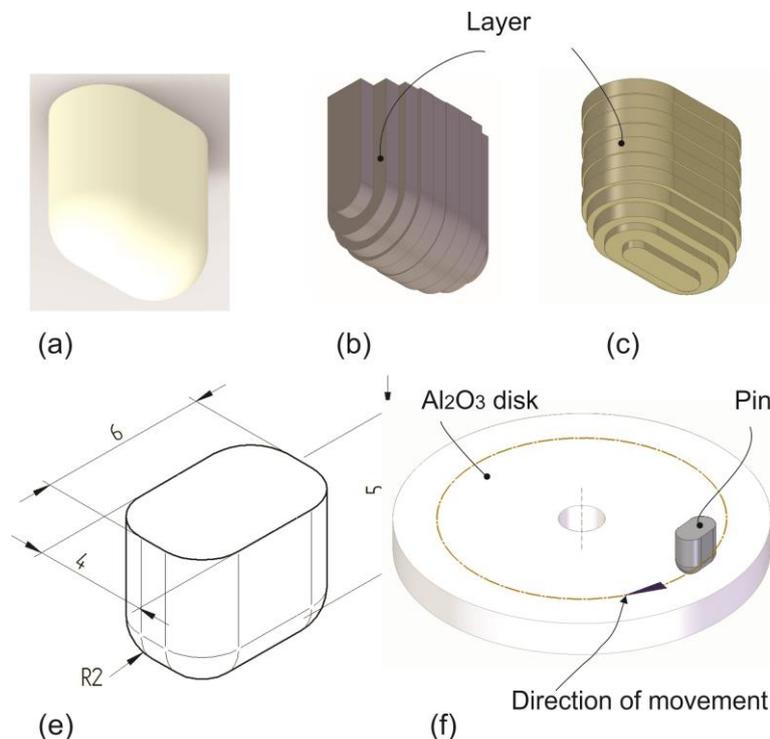


Figure 1 – Schematic pins for wear test, and (a) pin model, in (b) pin with perpendicular layers, (c) pin with parallel layers, (d) dimensions of the pin and (e) direction of friction.

Figure 2 - Pin assembled on AISI 1045 steel bracket.

After the process of burning organic residues and sintering (350ºC to 1500ºC), the samples were washed with acetone, dried at 100ºC for 1 hour, cooled in a desiccator for 30 minutes before weighing. The samples were kept in a desiccator when outside the test operation.

The experimental conditions were the following: Applied load of 10 N under atmospheric humidity between 45 and 55%; sliding of the zirconia post over an Alumina disk (Sain Gobain Ceramics / Brazil) >99% purity, flat surface grinding, Ra 0,25 +/- 0,08 μm; sliding speed of 1 m/s; a distance of 500 m covered in each step of measuring mass loss; total distance traveled 3 km.

A standard counterproof was performed with a Tosoh zirconia sphere (YTZ® Grinding media), with a diameter of Ø5mm, HV10 1250, Fracture Toughness 6.0 MPam$^{0.5}$, hold tight at metallic support and tested under the same experimental conditions for the AM pins.

Figure 3 shows the running test setup showing the pins on the support, de alumina disk and running wear dry test.
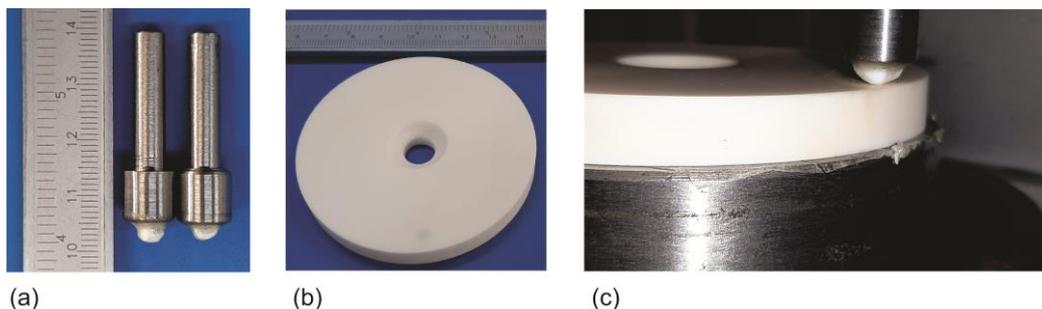


Figure 3 – Experimental setup, (a) pins, (b) $Al_2O_3$ disk and (c) perpendicular layer pin positioned.

## 3. RESULTS

The weight loss evaluation results of the samples printed in the perpendicular and parallel direction and the sphere are shown in Figure 4. The sample subjected to wear in the perpendicular direction of its layers in relation to the disk showed a higher loss peak in the first 500 meters. This loss was more than 2,5 times greater than the mass loss observed in the pin with layers parallel to the disk, the layers in the perpendicular mode present an aspect-like shave where the layers approach the blades that suffer efforts from opening in $K_{IC}$ mode and provided the edges to break that occurred until the smoothing of the contour of the surface. Only after 2500 meters traveled, the sample of layers positioned parallel to the disk showed wear 1.41 times higher. As the wear test progresses, it was observed that there is a tendency for both groups (parallel and perpendicular layer pins) to stabilize the mass loss.
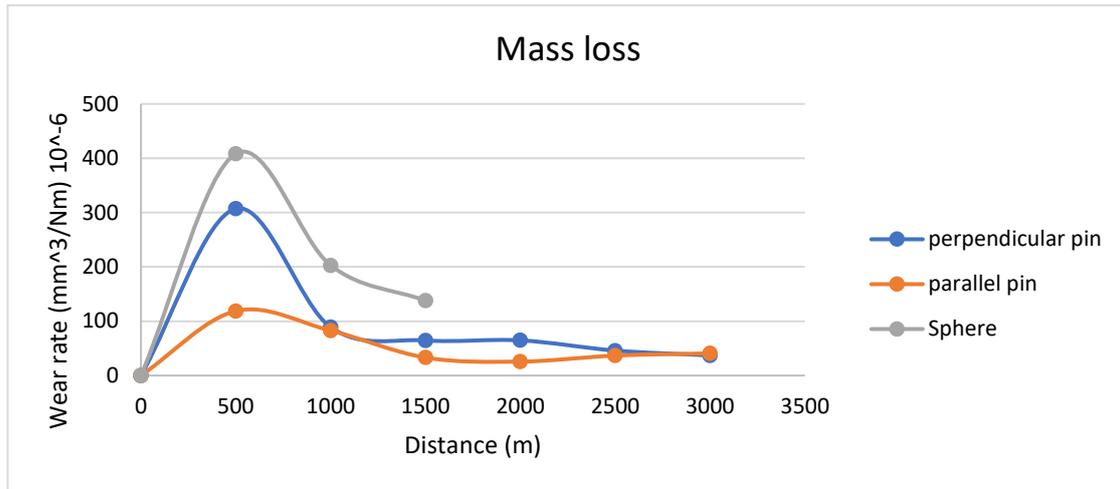
Figure 4 – Mass loss over distance traveled.

The zirconia sphere, tested as a reference group, showed even greater losses and, in this case, it was not possible to measure the mass loss beyond 1500 meters. Table 1 presents the volumetric percentage loss of the 3 analyzed groups (perpendicular, parallel and sphere pin) at the end of the 3000 meters route. Despite the initial area of the sphere being smaller than the area of the pin, it shows that the AM process does not cause deleterious damage in terms of wear.

Table 1 - Percentage volumetric loss of the sample after 3000 m traveled

| Sample | initial volume (cm³) | final volume (cm³) | Loss (%) |
|---|---|---|---|
| Perpendicular pin | 0,1074 | 0,1005 | 6,47 |
| Parallel pin | 0,1109 | 0,1064 | 4,12 |
| Sphere | 0,0644 | 0,0583 | 9,53[1] |

[1] After 1500 m.

According to the values presented in Table 1, the pin with layers parallel to the disk presented the lowest percentage of volumetric loss at the end of the 3000 m course, which corroborates the data obtained in Figure 4. These results may show, in terms of material loss, a supposed advantage of manufacturing dental products, such as orthodontic brackets and dental crowns, parallel to the region where wear occurs.

The results of the friction force evaluation of the samples printed in the perpendicular and parallel direction and the sphere are shown in Figure 5, Figure 6 and Figure 7.
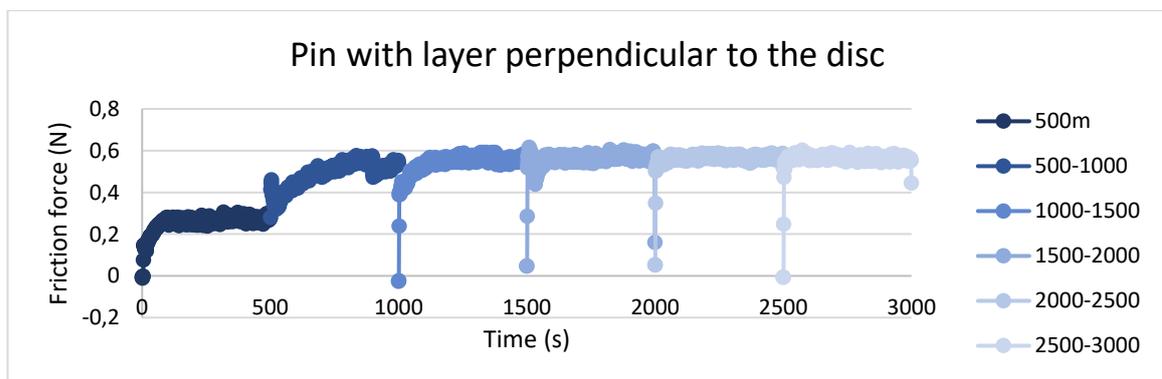


Figure 5 – Curve referring to the friction force per time obtained for the pin perpendicular to the disk measured in the intervals of 0-500 m, 500-1000 m, 1000-1500 m, 1500-2000 m, 2000-2500 m and 2500 – 3000 m.
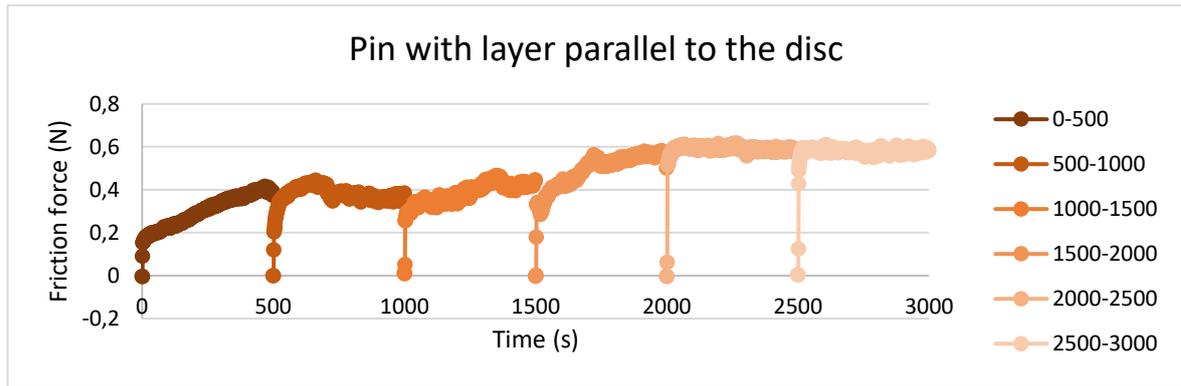
Figure 6 – Curve referring to the friction force per time obtained for the pin parallel to the disk measured in the intervals of 0-500 m, 500-1000 m, 1000-1500 m, 1500-2000 m, 2000-2500 m and 2500 – 3000 m.
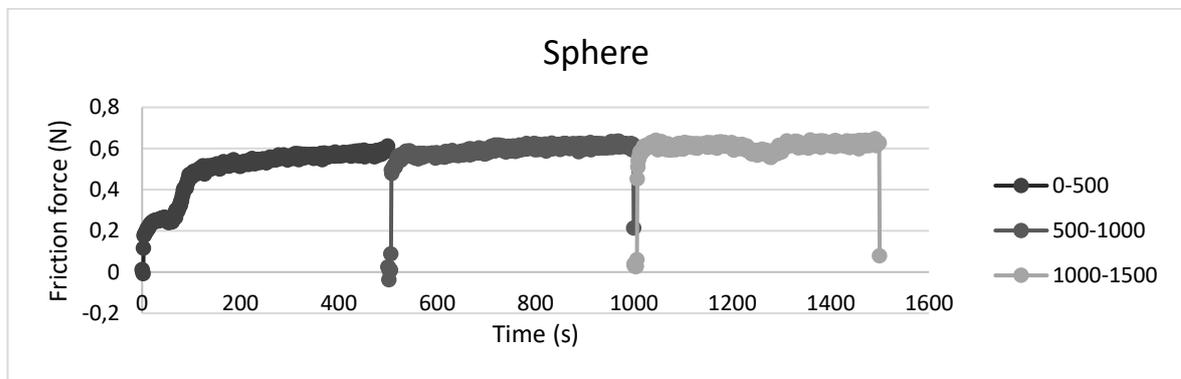


Figure 7 - Curve referring to the friction force per time obtained for the sphere measured in the intervals of 0-500 m, 500-1000 m, 1000-1500 m.

It was observed, through Figures 5, 6 and 7 that the friction coefficient tends to stabilize at an average value of 0.6 N for all groups. This value, however, was only reached for the pin with layer parallel to the disk after 1500 seconds of testing. This result is in agreement with the data referring to the lowest mass loss that this sample showed, showing that this is the most resistant sample to wear. The sphere, on the other hand, already presented friction coefficient of 0.54 in the first 500 s of travel, which was the least wear-resistant sample.

The results of the analysis of material loss in volume and coefficient of friction indicate that the sample that presented the greatest wear resistance was the pin printed with layers parallel to the disk. Thus, it can be assumed that, when printing dental parts, knowing the forces that will be applied on them, one chooses to manufacture parts by additive manufacturing in the direction of deposition layers parallel to such forces.

The fracture toughness of materials can be indirectly predicted by the classical equation (Eq. (1)) proposed for Evans and Wilshaw (1976).

$$V = \alpha \frac{W}{K_{IC}^{3/4}.H^{1/2}}.l \qquad\qquad (1)$$

Where [V] is the worn volume, [α] is proportionality constant, [W] is the normal load, [$K_{IC}$] is fracture toughness, [H] is hardness, and [l] is the sliding distance.

As long as the other parameters (H, W and l) are kept the same for all samples made of the same material, larger values of worn volume indicate smaller values of toughness. Thus, considering that the volumetric loss was grater for the perpendicular pin, it can be predicted that its fracture toughness will be lower than the parallel pin.

Figure 8 shows the wear surfaces of the pins and the direction of shear. In Figure 8 a), layers perpendicular to the disc are observed from the top view; in Figure 8 b) parallel layers with slightly inclined wear are observed showing the interface and the subsequent layer; finally, Figure 8 c) shows the surface of the sphere, homogeneous and uniform.
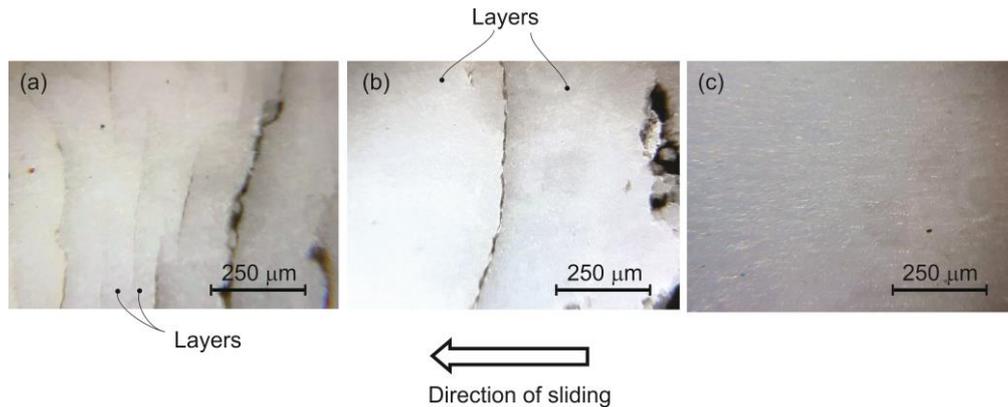
Figure 8 - Wear surfaces from the top view: a) pin with layers perpendicular to the disc; b) parallel layers with slightly inclined wear; c) sphere, homogeneous and uniform.

## 4. CONCLUSIONS

The results obtained in the wear test really show that there is anisotropy in the manufacture of a part. There was also no change in the coefficient of friction and this, although it could be expected, reports that the formed debris did not change the sliding performance.

The anisotropic effect was detected and the highlight is pointed to the parallel placement which had very low wear until the first layer was consumed and the interfaces presented themselves. This aspect reinforces the importance of DFAM for parts subject to wear, such as dental brackets, where a metal wire must allow relative movement on a bracket slot support surface.

## 5. AKNOWLEDGEMENTS

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## 7. RESPONSIBILITY

The author(s) is (are) the only responsible for the printed material included in this paper.