

ANALYSIS OF ZERODUR® MACHINABILITY USING SINGLE POINT DIAMOND TURNING

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Abstract. Some mirrors used in satellite cameras must present a high surface quality. They are usually made of ZERODUR®, a brittle glass ceramic, by means of abrasive processes. It was observed that the surface quality of the material deteriorates some time after the machining, thus requiring rework. The most influential cause of this phenomenon is the crack growth due to stress corrosion. These cracks are generated by the conventional machining processes of lapping and polishing. This paper presents a analysis on the machinability of ZERODUR® using ultraprecision turning with single-point diamond tool as an alternative to these traditional methods. ZERODUR® samples were subjected to indentation and scribing tests in order to study the ductile-brittle transition and material removal mechanisms. After that, the samples were turned with single-point diamond tool using the machining parameters that were defined during the indentation and scribing tests. The surface quality of each sample was evaluated with an interferometric optical profiler. The results demonstrated that ZERODUR® may be machined in ductile regime if an appropriate depth of cut is used. Also, the analysis allowed evaluating the influence of some machining parameters on the resulting surface quality. Thus, estimates for the best values for these parameters are provided.

Keywords: ZERODUR[®], Single Point Diamond Turning, indentation test, nano-scratching test, ultraprecision turning.

1. INTRODUCTION

With the rising growth of industrial production worldwide and the increasing competitiveness based on cost, quality and speed, the companies are continually looking for new materials that can present superior quality, lower costs and mechanical properties that may be relevant to the product performance, such as high electrical conductivity, corrosion resistance, low density, high mechanical strength, thermal stability, among others.

In addition, with the technological advances, new fabrication processes and machines usually surpass traditional ones in terms of efficiency and accuracy. In order to benefit from the advantages of these new materials and use them to manufacture products in small, medium and high scale, it is often necessary to purchase new machinery and measuring equipment, to hire specialized workforce and to develop new production processes.

In the optical industry, technological advances frequently occur. The companies seek more efficient machinery that may depend less on the operator's skills. This may provide competitive advantage to these companies.

The ZERODUR® is lithium aluminosilicate glass-ceramic produced since 1968 by Schott AG, a German manufacturer of industrial glass products. This non-porous glass ceramic has some superior optical properties, such as an extremely low thermal expansion coefficient and an excellent homogeneity of thermal expansion (SCHOTT, 2006; Döhring *et al.*, 2009). Due to these features, this material may be used to fabricate a variety of parts, such as: moulds, measurement standards and gauges, components for high power lasers, telescope mirrors, and so on.

Lapping and polishing are the traditional abrasive processes usually employed to machine ZERODUR®. From practical experiences, it was observed that the surface quality of the material deteriorates some time after it is submitted

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to these machining processes. Thus, the parts need to be rework in order to restore the desired surface quality. Several factors can cause this phenomenon, and the most influential may be the crack growth due to stress corrosion. This occurs when a glass or ceramic glass is subjected to tensile loads in wet environments (SCHOTT, 2009; Kurt *et al.*, 2012). The lapping and polishing processes generate microcracks, and the water that accumulates in these cracks causes corrosion when the material is subjected to stress loads. In these conditions, the speed of crack propagation depends on the combined effect of the stress and chemical attack from water.

Several factors present in the conventional machining processes can cause surface quality degradation, such as the fixture used and the forces applied to the part during the machining process. Thus, the conventional fabrication processes may be not recommended to manufacture high quality optical mirrors, since these processes create favorable conditions to surface degradation after machining. This paper presents a study on the machinability of ZERODUR ® using ultraprecision turning with single-point diamond tool as an alternative to traditional methods of lapping and polishing. Experiments were carried out using DOE techniques in order to observe the influence of the feed rate, the depth of cut and the rake angle of the cutting tool onto the resulting dimensional accuracy and surface roughness of the part. The relationship between these parameters and crack generation is also investigated. As a result of the analysis, it was possible to estimate the best values for these machining parameters, which may be used as a reference in the fabrication of high quality optical elements with ZERODUR[®].

2. LITERATURE REVIEW

2.1 Material features

ZERODUR[®] is the commercial name for a non-porous glass ceramic developed by SCHOTT AG in 1968. As its main property, it presents an extremely low coefficient of linear thermal expansion (CTE) (Döhring, *et al.*, 2009; Westerhoff, *et al.*, 2010). It is composed of high-quartz nano-crystallites of 30 nm to 50 nm in size, which represent approximately 70 to 78 percent of its weight, embedded in a remaining glassy phase. The crystallites present a negative coefficient of thermal expansion, while the glassy phase has a positive one. The fabrication process is controlled in such a way that the negative expansion of one phase and the positive expansion of the other one compensate each other (size and number of the nano-crystallites are adjusted to achieve that). This balance provides a great dimensional stability to the material when it is subjected to thermal variations (SCHOTT, 2009, Döhring, *et al.*, 2009).

The production process of ZERODUR® is similar to the production process of other optical glass: the glass is melted, refined, homogenized and conformed to the desired shape. In the specific case of ZERODUR®, the initially molten glass is cooled down to room temperature. Then, the nano-crystallite phase is generated by continually tempering the glass over a period of several months. This thermal treatment is called ceramization (Döhring *et al.*, 2008).

As its chemical and physical features are very similar to the features of other optical glass, the machining of ZERODUR® may be done with the same machines and processes used for the precision machining of optical glass (SCHOTT, 2009).

2.2 Ultraprecision machining

The ultraprecision machine tools arose after the Second World War, where viable alternatives for machining special components, such as computer, microelectronic and optical components, were being sought. This kind of machine tool was used to generate high quality surfaces in ductile materials, as an alternative to precision grinding.

Gradually, the area of ultraprecision machine has been further developed. Taniguchi (1983) presented a historical analysis of the machining processes, machines and tolerances evolution, which was later complemented by other authors such as Venkatesh and Izman (2007). This analysis is shown in Figure 1.

According to Duduch (1993), the term ultraprecision machining does not have a static definition, since this technology evolves with time. Thus, the level of tolerances related to the term ultraprecision depends on the historical time and kind of machine considered.



Figure 1. Evolution of the machining processes, machines and tolerances. Source: Venkatesh and Izman (2007)

2.3 Microindentation test

Indentation hardness tests are primarily used to determine the hardness of a material to deformation. In some cases, it is possible to determine tensile strength curves of materials by means of hardness tests, because indentation hardness correlates with tensile strength. The microindentation technique was originally developed to study the mechanical properties of thin films applied on material substrates. In these testes, the load applied is very low when compared to the loads applied in conventional hardness tests. The material hardness is determined by the ratio between the applied load (test load) and the area of impression, as shown in Equation 1.

$$H = \frac{F}{A} \tag{1}$$

Where: H = hardness of the material; F = applied test load; A = impresion area.

The microindentation hardness tests are usually carried out to gather some information about the mechanical properties of the material. These tests may provide specific information about the material removal regime, i.e., ductile regime or brittle regime, allowing to determine the conditions for which the plastic deformation process prevails over cleavage.

In this research, Vickers hardness tests were carried out in order to evaluate the mechanical properties of ZERODUR®. The advantage main advantage of these tests is that the calculations required to yield the hardness value do not depend on the size of the indenter. In addition, as they use a diamond-made indenter, they can be applied to several materials, regardless of their hardness. The scales of Vickers' test is usually wider than the scales of other hardness tests, thus providing a good resolution.

2.4 Nano scratching test

In the nano-scretching test, an indenter or another cutting tool moves with controlled speed over the surface of a given sample while the load applied to the tool is gradually incremented. The test is repeated many times varying the applied speed and load. The scratches are analyzed in order to gather information about the mechanical properties of the materials. Le Houérou *et al.* (2003 apud Wasmer, *et al.*, 2005) investigated the nano-scratching of glasses, presenting the various phenomena that may occur during this test. The model proposed by this author is shown in Figure 2. Wasmer, *et al.* (2005) also considered this model to investigate the nano-scratching of semiconductors.

An important aspect observed in this model is the existence of a region of plastic regime similar to what is found for ductile materials. In this regime, it is possible to machine brittle materials likewise ductile materials.

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Figure 2. Model scratching test typical of vitreous materials proposed by Le Houérou et al. (2003). Source: Wasmer, *et al.* (2005)

The nano scratching is considered an intermediary process between indentation and single-point diamond turning. The nano scratching becomes very similar to the turning processes when the indenter reaches a given depth so that the material suffers plastic deformation. Therefore, by means of nano-scratching it is possible to infer important information about the turning process of a given material, such as depth of cut, crack formation, cutting speed, among others.

2.5 Design of Experiments (DOE)

In an experiment, purposeful changes are made to input variables of a process or system aiming to observe the changes caused on the output variable and to indentify the reasons for these changes (Montgomery, 2005). The output variable of the experiment may be also called response variable. If the influence that each input variable exerts on the response variable is understood, it is possible to improve a process, optimizing its time or decreasing its costs, and so on.

According to Montgomery (2005), Statistical Design of Experiments (DOE) is "the process of planning the experiment so that appropriate data will be collected and analyzed by statistical methods, resulting in valid and objective conclusions".

Different experimental designs are available in the literature, such as simple comparative experiments, randomized blocks, latin squares, factorial designs, fractional factorial designs, among others. The choice for a specific design will depend on the kind and number and of factors in study, the time and resources available for the experiment, the sample size, the presence of randomization restrictions, among others.

Factorial designs are applied when the experiments involve several factors and the experimenter desires to study the joint effect of the factors on the response. In the 2^k design, *k* factors or input variables are considered, and each factor is examined at only two levels, a "high" level and a "low" level. This model allows evaluating the influence of each single factor on the response, as well as the joint effect of the factors, but does not allow the evaluation of second order effects (Montgomery, 2005). Figure 3 shows the geometric representation and the design matrix for a 2^k experiment, where k = 3.





Figure 3. a) geometric view of a 2^3 design b) design matrix of a 2^3 experiment

3. EXPERIMENTS

The development of this research basically comprised three phases. In the first one, indentation and nano-scratching tests were carried out, in order to investigate the mechanical properties of the material and define appropriate machining parameters. In the second phase, a set of experiments was designed and executed. The material was machined in a Rank Penumo ASG 2500 ultraprecision turning with a single point diamond tool. Finally, in the third phase, the results were analyzed aiming to observe the effect of different machining parameters combinations on the resulting surface quality of the samples.

3.1 Microindentation tests

As previously said, Vickers microindentation hardness tests were carried out in order to evaluate the mechanical properties of ZERODUR[®]. The tests were performed on a Leica VMHT HOT machine from the Materials Engineering Laboratory, in the School of Engineering of São Carlos (USP). Three ZERODUR[®] samples from the same material blank were used. In order to minimize the measurement errors, the working faces of the samples were polished until they reach a surface roughness (Ra) of approximately 10 nm.

Twenty five indentations were performed in each sample, split into 5 groups with 5 indentations which. The test loads applied to each group are of 5g, 10g, 25g, 50g and 100g, respectively. The average results found for the Vickers hardness of the samples are shown in Table **1** and Figure 4.

	5g	10g	25g	50g	100g
Sample 1	304.8	383	481.4	539	625.4
Sample 2	293.6	420.4	459.4	544	592.2
Sample 3	284.8	433.4	465.6	546.6	629.2
Total average					
(HV)	294.4	412.3	468.8	543.2	615.6

Table 1. Average results for the Vickers hardness (HV) of each sample.

After the indentation tests, a Wyco NT-1100 interferometric optical profiler was used to measure the depths of the impressions, to evaluate the surface quality of the samples and to observe other phenomena related to the material removal process, such as pile-up behavior. The analyses showed the occurrence of cracks when loads of 50g and 100g were applied. This indicates that the depth of cut should be smaller than the depth of the impressions done with loads of 50g. Test loads of 25g did not cause cracks on the surface, however, a pile-up of 30% of the total depth of impression was observed, as shown in Figure 4. Thus, the depths resulted from the impressions performed with loads of 5g and 10g should be considered as possible depths of cut for turning. For these test loads, depths of 0.46 μ m and 0.59 μ m, respectively, were obtained. These results were compared to the ones found in the nano-scratching test, which will be presented in the next section.



Figure 4. Analysis of the depth of the indentation using an interferometric optical profiler. The profile shows a pileup of the material with aproximately 1/3 of the total indentation depth

3.2 Nano scratching tests

For the nano-scratching tests, the ZERODUR® samples were prepared as for the indentation test, that is, the working faces were polished to reach a surface roughness (Ra) of approximately 12 nm.

The samples were pasted with beeswax in an aluminum base, which was fixed into a sine table. After that, the sine table was mounted on the table of a Romi D 1250 CNC machine center. The face of the sample was leveled along the longitudinal direction of the aluminum base with aid of a dial indicator and standard shims. Along the transversal direction of the base, the sample was tilted by 0.01146°, which corresponds to a slope of 0.005 mm in 25 mm. This procedure is shown in Figure 5. The reference point for the cutting edge of the tool was set on the lowest point of the slope, thus, the tool cuts the face of the sample from the lowest to the highest point.



Figure 5. Execution of the nano-scratching tests: a) tilting procedure along the transversal direction; b) leveling procedure along the longitudinal direction; c) displacement of the tool

In the first sample, a set of three scratches were performed with tool speed of 0.3 m/s. After that, three additional tests were performed at 0.8 m/s. The scratches were regularly spaced at 1 mm. The same procedure was repeated for the remaining samples. A Wyco NT-1100 interferometric optical profiler was used to analyze the scratches and generate images of them.

The results found are aligned with previous findings. More specifically, they show that the material behavior follows the phases of the model presented by Le Houérou *et al.* (2003 apud Wasmer *et al.*, 2005), for brittle materials, which are: elastic regime; plastic regime; subsurface cracking regime; subsurface and surface cracking regime; and micro abrasive regime. These phases are directly related to the applied loads and the material removal regime. The comparison between the obtained images and the mentioned model is shown in Figure 6.





The results of the nano-scratching test showed that the transition from the ductile regime to cleavage occurs between the phases of plastic regime and subsurface cracking regime. In terms of depth of cut, this transition occurs between 0.315 μ m and 0.45 μ m. In addition, it could be seen that the region of subsurface cracking presented pile-ups of 2/3 of the average total depth. Thus, based solely on the results of the nano-scratching tests, the depth of cut for single point turning should not exceed 0.315 μ m in order to avoid pile-up.

The speeds of used in the tests seemed not to significantly influence the results.

3.3 Indentation and nano scratching tests combined analysis

The results of the indentation tests suggest that a depth of cut of less than 0.59 μ m should be used. On the other hand, the results of the nano-scratching tests show that the depth of cut to be used in ultraprecision turning should be smaller than 0.315 μ m. At first, values for depth of cut closer to 0.315 μ m should be chosen, since the ultraprecision turning process have more similarity to the nano-scratching process in terms of material removal mechanism. However, some

Blake (1988) proposed a model for the material removal in ductile regime for ultraprecision turning of brittle materials with single-point diamond tool. According to this model, the chip thickness varies from zero at the tool center up to a maximum at the top of the uncut shoulder. There is a gradual decrease in the severity of the damage along the shoulder. The contact between the tool and the workpiece occurs on a limited area where the tool faces the uncut shoulder, since part of the workpiece surface has already been cut. In the nano-scratching tests, however, the contact between the tool and the workpiece area comparing to the turning process. Thus, it is reasonable to suppose that the cutting forces involved in the nano-scratching test are bigger than the ones involved in the single point turning. For this reason, a value for depth of cut bigger than 0.315 μ m was chosen for the tests. Thus, two values for depth of cut were tested, 0.4 μ m and 0.2 μ m, and also two values for feed rate, 0.3 μ m/rev e 0.1 μ m/rev.

3.4 Experimental design (DOE)

In the investigation of the machinability of ZERODUR[®], three factors of influence or three input variables were considered: the rake angle of the cutting tool, the feed rate and the depth of cut. As the objective was to firstly carry out an exploratory research, these variables were tested at two levels, as shown in Table 2. Thus, the 2^3 factorial design was chosen as the most suitable for this case. As previously said, the definition of these levels was based on the results of the indentation and nano-scratching tests.

Factor	low level	high level
rake angle (°)	-20	-5
feed rate (µm/rev.)	0.1	0.3
depth of cut (µm)	0.2	0.4

Table 2. Factors of the experiment and their respective levels.

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The design matrix of the experiment is shown in Table 3.

Combination	Factor	Factor	Factor		Combination	Rake angle	Feed rate	depth of cut
Comonation	Α	В	С		Comonation	(°)	(µm/rev.)	(µm)
1	-1	-1	-1		1	-20	0.1	0.2
2	1	-1	-1	→	2	-5	0.1	0.2
3	-1	1	-1		3	-20	0.3	0.2
4	1	1	-1		4	-5	0.3	0.2
5	-1	-1	1		5	-20	0.1	0.4
6	1	-1	1	→	6	-5	0.1	0.4
7	-1	1	1		7	-20	0.3	0.4
8	1	1	1		8	-5	0.3	0.4

Table 3. Generation of the design matrix for the 2³ ZERODUR[®] turning experiment.

Two replications for each combination of factors were done, that is, two ZERODUR[®] samples were used, and each sample was turned with all the 8 combinations of parameters. The tests were performed in random order (8, 1, 3, 7, 2, 4, 5 and 6), aiming to avoid the influence of other variability sources on the results.

The response variable of the experiments is the Ra surface roughness of the turned surfaces.

3.5 Machine and cutting toll

The turning tests were carried out on the Aspheric Surface Generator Rank Pneumo ASG 2500. This ultraprecision turning machine is provided with an aerostatic spindle and hydrostatic bearings. Also, it has a Computer Numerical Control (CNC) ALLEN BRADLEY series $8200^{\text{®}}$ integrated with a HP laser interferometer, which allow the simultaneous control of the displacements in the x and z axes. Due to this features, the machine presents a maximum positioning error of 10 nm and may generate surfaces with Ra roughness of up to 75 Angstroms in many materials.

In this research, a round nose single crystal diamond tool model C020LG from Contour Fine Tooling[®] was used. This tool has a nose radius of 0.507 mm and a rake angle of 0°. As shown from the figure 7, a customized fixture was built to allow tilting the tool by specified angles and, thus, to generate negative rake angles of -5° e -20°, as defined in the experimental design.



Figure 7. Adjustable tool fixture: a) for 5°, b) for 20°, c) isometric view.

3.6 Sample preparation and machining

Each of the two samples was fixed into a aluminum base with wax for optical components. This base was fixed on the machine spindle by means of a vacuum board. The face of the samples was turned to generate a stepped profile, as shown in Figure 8. Each step corresponds to each of the combinations shown in Table 3, performed in a random order.



Figure 8. Views of the machined sample

4. RESULTS AND DISCUSSIONS

The experimental results are shown in Table 4, with the combinations in standard order. As previously said, however, the tests were performed in random order (8, 1, 3, 7, 2, 4, 5 and 6).

					Response variable -				
								Roughnes	s Ra (nm)
Combination	Factor	Factor	Factor		Tool angle	Feed rate	Depth of	Replication 1	Replication
Combination	А	В	С		(°)	(µm/rev.)	cut (µm)	(sample 1)	2 (sample 2)
1	-1	-1	-1		-20.0	0.1	0.200	273.7	302.5
2	1	-1	-1	→	-5.0	0.1	0.200	479	479.95
3	-1	1	-1		-20.0	0.3	0.200	213.3	215.09
4	1	1	-1		-5.0	0.3	0.200	413.85	430.84
5	-1	-1	1	→	-20.0	0.1	0.400	277.83	301.83
6	1	-1	1		-5.0	0.1	0.400	518.94	540.67
7	-1	1	1		-20.0	0.3	0.400	193.17	213.8
8	1	1	1	→	-5.0	0.3	0.400	537.84	570.23

Table 4. Experimental results of ZERODUR[®] single point diamond turning.

For the analysis of the experiment, the values of the main effects generated by each factor (E_A , E_B and E_C) as well as the values of the interaction effects (E_{AB} , E_{AC} , E_{BC} and E_{ABC}) were calculated, as shown in **Table 5**. In addition, the graphical analysis of the factors and the Analysis of Variance (ANOVA) of the experiment were also carried out with the aid of the open statistical software Action 2.5 (Excel add-in). These analyses are shown in Figure 9 and Table 6, respectively. The main objective of the analysis is to find the factors that are most relevant to minimize the surface roughness.

Table 5. Calculated main effects and interaction effects of the factors.



Figure 9. Plots of the main effects of the factors (E_A , E_B and E_C)

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Figure 10. Plots of the interaction effects EAB, EAC and EBC respectively

			ANOVA table		
Factors	D.O.F	Sum of squares	Mean square	F statistics	P-value
Α	1	245049.7506	245049.7506	1075.632153	8.14661E-10
В	1	9326.730625	9326.730625	40.93916159	0.000209496
С	1	7485.7104	7485.7104	32.85810645	0.00043785
A:B	1	4054.505625	4054.505625	17.79702531	0.002921187
A:C	1	9120.25	9120.25	40.03282646	0.000226086
B:C	1	1187.4916	1187.4916	5.212427855	0.051822061
A:B:C	1	2199.61	2199.61	9.655064872	0.014505501
Residuals	8	1822.5543	227.8192875		

Table 6. Analysis of Varia	nce for the factorial	experiment wit	h replication.
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The analysis of the main effects of the factors firstly shows that the rake angle of the cutting tool exerts the most significant effect on the response variable (Figure 9 a). The variation of this factor from the low level (-1, i.e., -20°) to the high level (+1, i.e., -5°) causes an average increase in the surface roughness of 247.91 nm (Figure 10). Thus, the experiment showed that lower values of rake angle (more negative values) provide better surface quality, and this factor is much more relevant for the result than the other tested factors. On the other hand, the sign of the main effect E_B demonstrates that an increase in the feed rate causes a decrease in the surface roughness. In other words, for the range analyzed, higher feed rates result in better surface quality. In the third graph of Figure 10, it is also possible to see that an increase in the depth of cut results in a higher surface roughness, however, this effect is much less relevant than the main effect of tool angle.

As can be seen in Table 5, the effect E_{AC} has the biggest magnitude in comparison to the other effects of interaction. In Figure 10b it is also possible to observe the difference between the slopes of each line. This means that the effect of varying the rake angle at the lower (-1) level and at the higher (+1) level of the depth of cut is significantly different. On the other hand, as the *p*-values for the interactions between BC and ABC are larger than ± 1 %, then they may be considered not significant for the experiment at a level of significance of 99%. The remaining factors and their respective interactions are significant for building a model to explain the relationship among the independent variables and the dependent variable (i.e., the surface roughness) at the mentioned level of significance. These are the main conclusions that can be drawn from the ANOVA table.

The element BC in the table concerns the interaction between the feed rate and the depth of cut. Thus, it is possible to assert that the effect of varying the feed rate is approximately the same, independently of the level taken for the depth of cut. In summary, there is a relevant interaction between the rake angle of the tool and the depth of cut (AC), but there is no significant interaction between the angle of the feed rate and the depth of cut (BC). The value of the interaction between the angle of the tool and the feed rate is intermediary in comparison to the remaining interactions between two factors. Finally, the interaction among all factors (ABC) may be also considered not relevant, as already said.

In addition, it is relevant to note that the magnitude of the main effects, in general, is bigger than the magnitude of the interaction effects. Nevertheless, there is one exception for this: the effect of the interaction between the rake angle and the depth of cut (E_{AC}) is slightly higher than the main effect of the depth of cut (E_C). This aspect may suggest that, in order to obtain lower values of surface roughness, it is more beneficial to set a bigger depth of cut in combination to a lower rake angle for the turning process than to use a lower depth of cut.

5. CONCLUSIONS

This research investigated some parameters of the ZERODUR[®] single point diamond turning process. Based on statistical evidences, it was possible to see that the rake angle of the machining tool is the factor that most affected the resulting surface roughness of the material. In addition, the ANOVA results showed that the interaction between this

angle and the depth of cut must also be considered, as well as the individual influence of the feed rate. These latter aspects, though, influence the response in a much lower degree than the first mentioned factor.

For the ranges of variation covered in the experiment, the rake angle of the tool should be set to the lowest level (20°), while the depth of cut and the feed rate should be set on the highest level ($0.4 \,\mu\text{m}$ and $0.3 \,\mu\text{m/rev}$, respectively) in order to achieve lower values of surface roughness, i.e., to achieve better surface quality.

This study was exploratory. Thus, based on these preliminary results, additional experiments may be carried out to move the investigated ranges of variation in the directions that showed to optimize the response variable.

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