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Effects of Nano-Diamond-Coated Milling Bits on Cutting Dental Zirconia

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Abstract: Hard alloy end mills are commonly employed for milling zirconia prostheses in dentistry. Nano-diamond-coated milling bits ensure high processing efficiency, accuracy, extended tool life, and reduced processing costs. This study aims at comparing various effects of cutting dental zirconia with nano-diamond-coated and ordinary milling bits. Two types of milling bits, one with nano-diamond coating and one without, were used to cut the dental zirconia green blanks (Ø98.5 mm, thickness: 25 mm) at three different speeds (1000, 1500, and 2000 rpm) in a dental milling machine. The unsintered and sintered zirconia surfaces were evaluated with glossmeter, optical profilometer for surface roughness, SEM, and EDX. The glossiness of the sintered zirconia block was statistically higher than that of the unsintered block ($p < 0.05$). For sintered zirconia, the nano-diamond-coated milling bit yielded a statistically ($p < 0.05$) higher glossiness in all spindle speeds than uncoated, save for the uncoated milling bit used at 1500 rpm. However, in terms of roughness, only sintering showed to be a statistically significant factor ($p < 0.001$) outweighing other two factors, and sintered zirconia always yielded lower surface roughness than the unsintered counterpart. Overall, the nano-diamond-coated milling bit can be operated at various speeds, resulting in a higher gloss on the sintered zirconia block, while an ordinary, uncoated milling bit can only achieve the same glossiness at a designated speed. The type of milling bits and the speeds have no significant effect on the surface roughness.

Keywords: nano-diamond; coating; milling bit; zirconia; glossiness

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1. Introduction

The tooth is an important organ of the human body; teeth not only perform the chewing function in daily life but also play an important role in pronunciation, language, and maintaining the normal shape of the face. Defects of teeth are a common clinical problem in the oral cavity. Prostheses such as inlays, onlays, veneers, crowns, and bridges are commonly used to solve this problem. ZrO_2 (zirconia), a white zirconium dioxide crystal, is a glass-free polycrystalline ceramic material that is particularly useful in dentistry for prostheses thanks to its high strength, toughness, good biocompatibility, chemical stability, and aesthetic properties [1].

Currently, computer-aided design and computer-aided manufacturing (CAD/CAM) is the dominant method for producing dental prostheses through digital design and computer numerical control (CNC) milling. CAD/CAM technology has revolutionized various industries by enabling the integrated design and manufacture of products. CAD involves creating digital models as 3D representations of products using geometrical parameters, while CAM uses these design data to control automated machinery through CNC systems.

With more than 40 years of development, dental CAD/CAM systems have transformed the traditional handmade approach to digitalized fabrication and brought new vitality to the dental field [2]. CNC machining is a subtractive manufacturing method that employs milling bits to cut and remove material from a flat-surfaced workpiece [3]. It is important to note that milling bits and milling burs are two different types of milling tools that are often confused by readers. In general, milling bits are primarily used for cutting action in dry conditions, such as cutting zirconia, wax, polymethyl methacrylate, polyether ether ketone, metal, etc. Milling burs, on the other hand, are designed and used for grinding in wet conditions [4].

In dental CAD/CAM, hard alloy end mills, specifically tungsten carbide alloys, are commonly employed for milling zirconia prostheses. The milling process can have an impact on both the surface profile of the restoration and the milling tool itself. Surface alterations such as increased surface roughness and critical defects on the ceramic surface may occur during CNC milling process. The defects may act as stress concentrators and lead to crack propagation [5–7] and, finally, failure of the prostheses.

The shape of milling bits as well as the spindle speed, feed rates, and angle not only influence the milling efficiency [8] but also the milled prosthesis, especially in surface defects [9] and roughness [10]. Surface roughness is closely linked to glossiness [11]. A dental prosthesis with higher glossiness mimics the look of natural teeth, which is preferred by the patients. Despite the increased efficiency offered by CNC milling, manual surface finishing is still required after the milling process for zirconia prostheses, which includes polishing and glazing, to enhance light transmittance. Surface finishing is necessary not only for aesthetic purposes but also for functional and hygienic reasons [12]. A smooth surface reduces bacterial attachment and wear on the opposing enamel while also providing long-term color stability, as it is less susceptible to staining compared to a rough surface.

Diamond coating is a highly effective method for enhancing wear resistance, thereby improving the performance and durability of milling tools. Numerous studies have reported that diamond-coated tools exhibit significantly better cutting performance and longer life expectancy compared to uncoated tools [13]. Moreover, the use of nano-sized diamond particles in the coating further enhances its physical and mechanical properties when compared to macro-sized coatings. This improvement is primarily attributed to the surface effect and small-size effect of nano-diamond particles [14]. By combining the nanotechnology and diamond coating, the application of nano-diamond-coated milling bits ensures high processing efficiency, accuracy, extended tool life, and reduced processing costs [15,16]. While there is a growing trend towards 3D printing of prostheses, which may reduce the need for CNC milling, the high cost and limited availability of this technology still hinder its widespread use in printing ceramic dental restorations [17].

Studies have investigated the use of nano-coated milling bits for milling hard, brittle materials, with a primary focus on tool-wear evaluations [13,18–20]. Given the limited knowledge regarding the impact of nano-diamond-coated CNC milling bits on the glossiness and roughness of zirconia surfaces, our current study aims to address this research gap. The objective of this study is to assess the effects of coating on milling bits at various speeds (2000 rpm, 1500 rpm, and 1000 rpm) when milling zirconia green blanks. Additionally, we aim to investigate how the nano-diamond coating and spindle speed influence the glossiness, roughness, and surface alterations of the zirconia material. The null hypothesis for this study posits that there are no discernible differences in surface roughness and glossiness between zirconia milled using coated and uncoated bits, regardless of the spindle speed employed.

2. Materials and Methods

2.1. Experimental Procedure and Materials Preparation

The experimentation design of this study is illustrated in Figure 1. Six two-flute ball nose milling bits with a diameter of 1.9 mm (SF Technology Ltd., Hong Kong) were used in this study, with three of the bits coated with nano-diamond particles by chemical vapor deposition (CVD) (CVDiam HF60, NeoCoat, La Chaux-de-Fonds, Switzerland). Before nano-diamond deposition, the bits were treated with Murakami solution and Caro's acid, followed by diamond seeding in an ultrasonic bath. Then, a 1%–3% volume concentration of methane with hydrogen was deposited, forming a multilayer structure after 48 h of deposition.

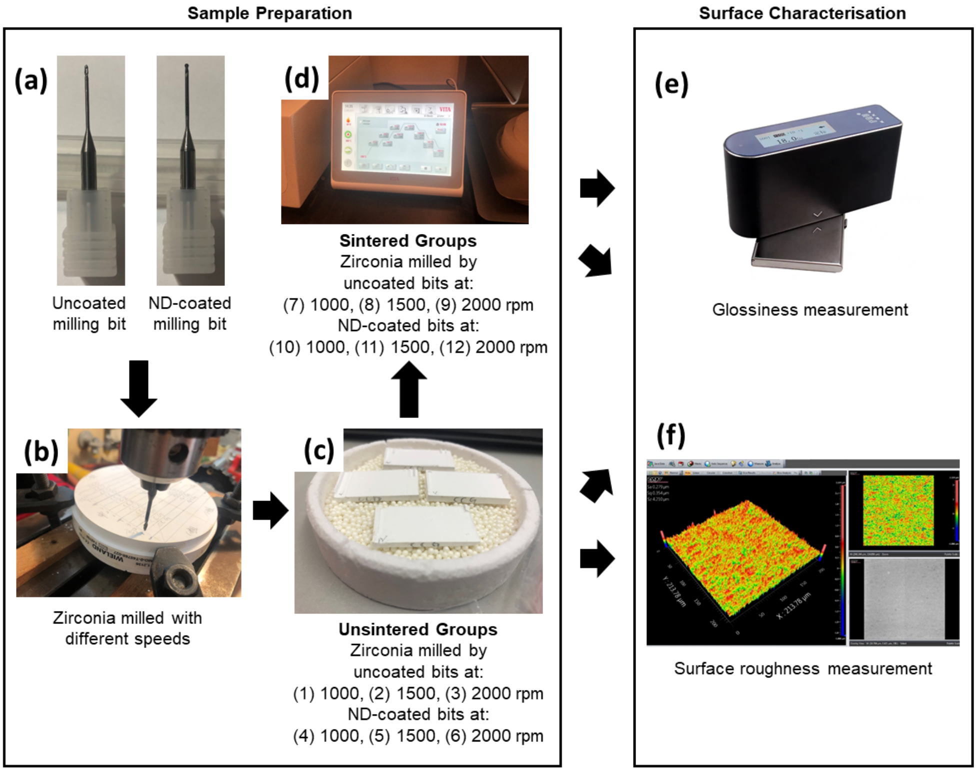


Figure 1. Schematic diagram of the experimentation design: (**a**) uncoated and coated milling bits; (**b**) a zirconia green blank on the milling machine; (**c**,**d**) grouping of specimens: (1)–(6) unsintered zirconia milled by uncoated/ND-coated bits at 1000, 1500, and 2000 rpm and (7)–(12) sintered zirconia milled by uncoated/ND-coated bits at 1000, 1500, and 2000 rpm; (**e**) characterized by a gloss meter; and (**f**) characterized by an optical profilometry system (ND, nano-diamond).

Three pieces of zirconia blocks with a diameter of 98.5 mm and a thickness of 25 mm (Zenostar MO 0, Wieland, Pforzheim, Germany) were used in the experiment. Slice-shaped samples measuring 1.5 × 0.5 × 2.5 cm^3 were cut from a dental micro-milling machine (X2, Shanghai SIEG Machinery Co. Ltd., Shanghai, China) in high (2000 rpm), medium (1500 rpm), and low (1000 rpm) speeds. Coated and uncoated milling bits in 1.9 mm diameter were installed in the milling machine. The sample size was set as six, with each sample having dimensions of 1.5 × 0.5 × 2.5 cm^3. Seventy-two specimens were obtained by cutting three zirconia green blank, of which half were cut using uncoated milling bits with low to high spindle speed; the other half were cut using coated milling bits with low to high spindle speed. Milling bits were changed after milling every 12 specimens.

The milled zirconia green specimens were then sintered in a sintering furnace (Zyrcomat 6000 MS, Vita Zahnfabrik, Bad Säckingen, Germany) following the manufacturer's guidelines. The sintering process involved heating the samples from room temperature to 900 °C at a rate of 600 °C/h, followed by a 0.5 h holding time. The temperature was then increased from 800 to 1500 °C at a heating rate of 200 °C/h, followed by a 2 h holding time. Finally, the samples were cooled to 900 °C and 300 °C with cooling rates of 600 °C/h and 500 °C/h, respectively. The entire sintering cycle took approximately 10 h.

2.2. Roughness

An optical profilometry system (Zygo NexView, Zygo, Middlefield, CT, USA) was used to measure the surface roughness of each sample. A total of 72 specimens were used for roughness measurement, with each group consisting of six samples (n = 6). Three variables were tested: two sintering conditions, two coating conditions, and three spindle speeds. For each sample, three random points on the surface were selected for data measurement and recording [21]. The arithmetic average height (Sa), root mean square height (Sq), and maximum height (Sz) were obtained for each sample from the profilometer's built-in software Mx (Version 6.1.0.4, Zygo, Middlefield, CT, USA).

2.3. Glossiness

For glossiness measurements, the same 72 zirconia samples (n = 6) collected for roughness measurement were used. A universal gloss meter (WG60G, FRU, Shenzhen, China) was utilized for measurement. The gloss meter featuring a 60° projection angle and a measuring hole of 8 mm × 4 mm. To measure each sample, three random points were selected, and the average value of each point was measured internally by the gloss meter.

2.4. SEM and EDX

The zirconia samples together with milling bits surfaces were magnified at 100×, 500×, and 1500× and photographed using a scanning electron microscope (SEM) (SU1510, Hitachi, Tokyo, Japan). In addition, zirconia samples and milling bits were analyzed using an energy-dispersive X-ray spectroscopy (EDX) for surface composition at a magnification of 100×.

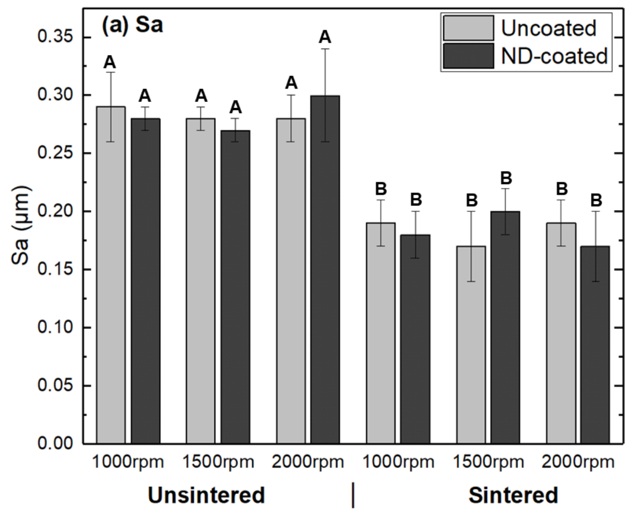
2.5. Statistical Analysis

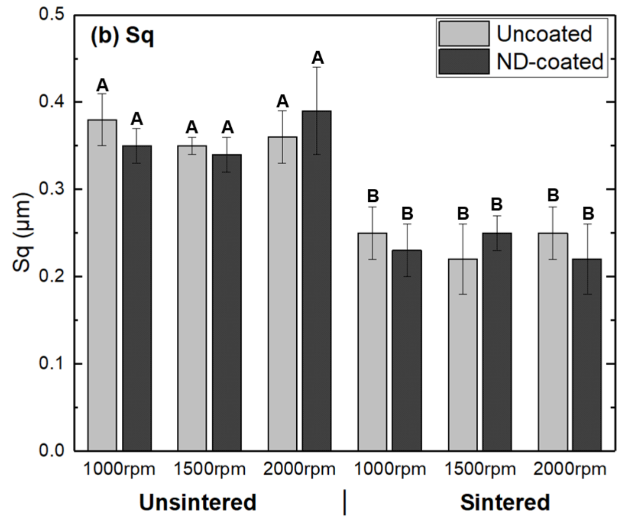
Statistical analysis was conducted using SPSS software (Version 25, IBM, Newark, NJ, USA). One-way analysis of variance (ANOVA) with Tukey's post hoc test was used to determine statistical significance at an α level of 0.05. Additionally, three-way ANOVA was employed to evaluate the effects of sintering, milling bit coating, and spindle speed on the glossiness of zirconia surfaces.

3. Results

3.1. Roughness

Figure 2 shows the results of Sa, Sq, and Sz for all groups. One-way ANOVA showed that the roughness indexes Sa, Sz, and Sq were not statistically different when comparing with different spindle speeds. It could be noted that the sintering process significantly lowers the surface roughness; i.e., the surface of zirconia blocks becomes smoother after sintering.





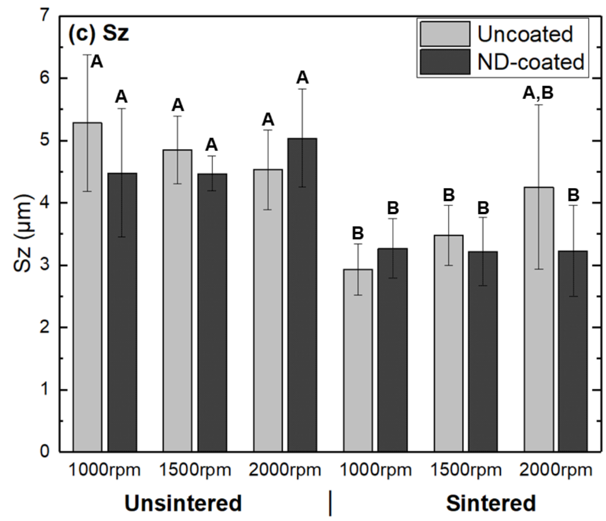


Figure 2. Surface roughness ((**a**) Sa, (**b**) Sq, and (**c**) Sz) results of zirconia specimens milled with different milling conditions (ND, nano-diamond). Same letters denote no statistical significance ($\alpha = 0.05$).

3.2. Glossiness

As shown in Figure 3, the average gloss of all sintered zirconia samples was significantly higher than that of the unsintered samples. Among the sintered groups, the surfaces of the zirconia milled with uncoated milling bits at medium speed (1500 rpm) and a coated milling bit at high speed (2000 rpm) exhibited significantly higher values of 14.65 GU and 14.85 GU, respectively. By comparing the spindle speed regardless of the other conditions, zirconia milled at medium speed showed the highest statistically significant glossiness.

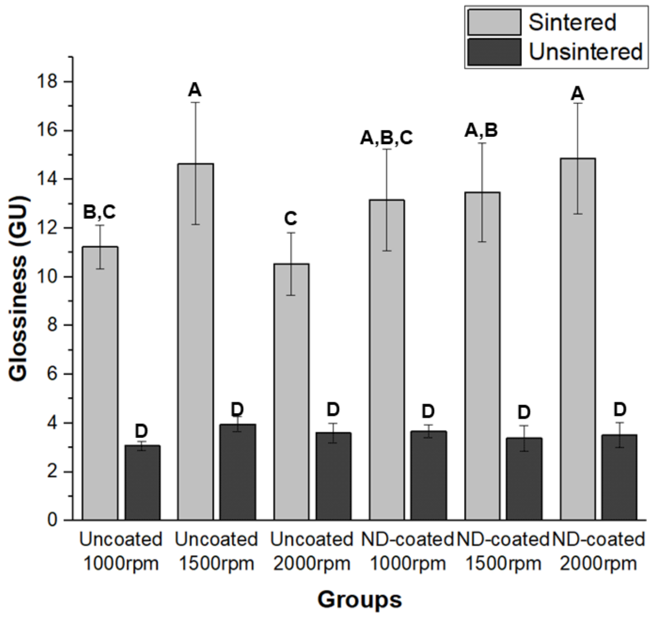


Figure 3. Glossiness of zirconia specimens milled with different milling conditions (ND, nanodiamond). Same letters denote no statistical significance (α = 0.05).

Moreover, according to the three-way ANOVA (Table 1), significant main effects on glossiness were observed for (A) sintering status, (B) milling bit coating status, and (C) spindle speed as well as significant interactions regarding glossiness between sintering and milling bit coating status, between milling bit coating status and spindle speed, and between all three factors with a p-value below 0.05.

Table 1. Three-way ANOVA analysis of glossiness (dependent variable) results.

Source	Type III Sum of Squares	df	Mean Square	Sig. (P)	Partial Eta Squared
Corrected Model	1702.735 [a]	11	154.794	0.000	0.936
Intercept	4908.753	1	4908.753	0.000	0.977
Sintering Status (A)	1606.500	1	1606.500	0.000	0.933
Milling Bit Coating Status (B)	12.417	1	12.417	0.014	0.096
Spindle Speed (C)	14.819	2	7.409	0.027	0.113
A × B	13.090	1	13.090	0.012	0.101
A × C	8.172	2	4.086	0.130	0.066
B × C	28.647	2	14.323	0.001	0.198
A × B × C	19.090	2	9.545	0.010	0.141
Error	116.282	60	1.938		
Total	6727.770	72			
Corrected Total	1819.017	71			

[a] R Squared = 0.936 (Adjusted R Squared = 0.924).

3.3. SEM and EDX

Figure 4 shows the typical surface morphology of milled zirconia with different speed and coating conditions. No distinct difference could be found in the morphology of zirconia milled by coated or uncoated milling bits under same spindle speed. Table 2 shows the EDX results of the zirconia surface after milling by different bits. No obvious difference was identified in zirconia compositions milled at different speeds using either coated or uncoated bits.

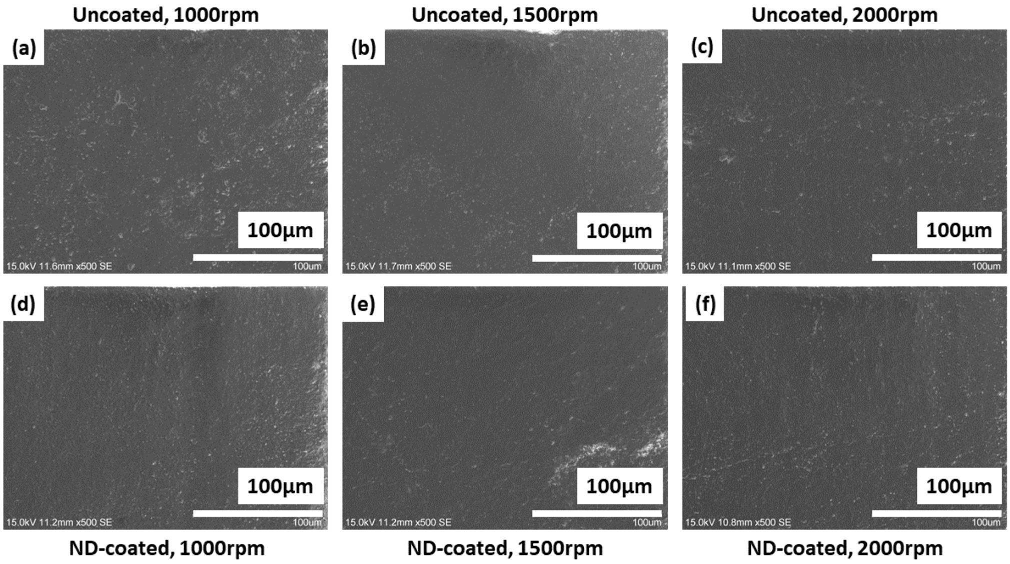


Figure 4. SEM images of sintered zirconia surface after milling with different milling conditions: (**a**) uncoated bit with 1000 rpm spindle speed; (**b**) uncoated bit with 1500 rpm spindle speed; (**c**) uncoated bit with 2000 rpm spindle speed; (**d**) ND-coated bit with 1000 rpm spindle speed; (**e**) ND-coated bit with 1500 rpm spindle speed; and (**f**) ND-coated bit with 2000 rpm spindle speed (ND, nano-diamond).

Table 2. EDX results of zirconia specimens milled with different milling conditions (ND, nano-diamond).

Elements	Uncoated (Atomic %)			ND-Coated (Atomic %)		
	Low Speed	Mid Speed	High Speed	Low Speed	Mid Speed	High Speed
C	11.012	10.099	12.124	11.369	12.804	11.315
O	35.040	35.721	35.442	32.900	34.585	36.392
Al	3.140	3.247	3.019	3.193	3.098	3.054
Y	4.575	4.756	4.707	4.454	4.505	4.411
Zr	41.039	41.772	39.597	43.163	40.367	40.133
Hf	5.195	4.404	5.111	4.922	4.641	4.695

Figure 5 shows the SEM images of the uncoated and coated milling bits. A comparison between the two revealed that the surface of the coated milling bit exhibited small, convex, spherical particles, whereas the surface of the uncoated milling bit appeared relatively flat.

Table 3 illustrates the EDX results of milling bits before and after milling. The main component of both the coated and uncoated milling bits was carbon (C), but the surface of the coated milling bit had a higher carbon content, reaching up to 90%. In contrast, the carbon content on the surface of the uncoated milling bit was approximately 40%. The uncoated bits were mainly made from C, Sr, and W, representing sums over 60% of the composition.

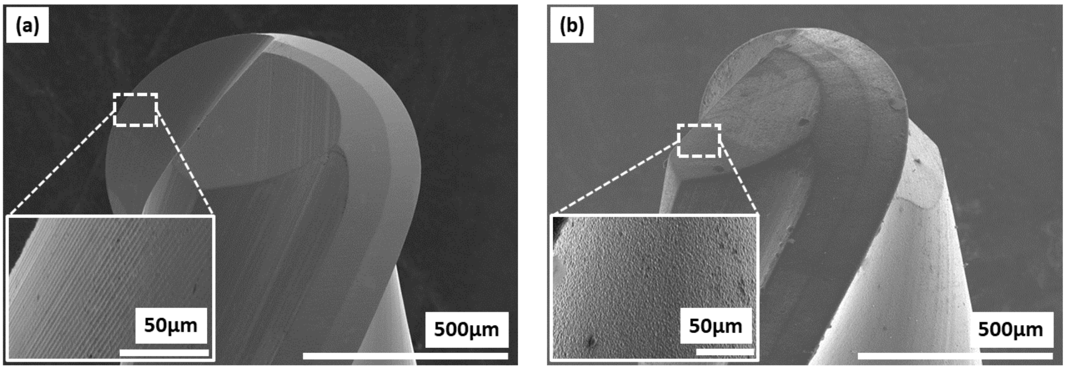


Figure 5. SEM images of (**a**) uncoated and (**b**) coated milling bits.

Table 3. EDX results of milling bits before and after milling.

Elements	Noncoated (Atomic %)		Coated (Atomic %)	
	Before Milling	After Milling	Before Milling	After Milling
C	39.104	20.354	96.005	87.301
O	5.519	10.243	2.380	6.734
Al	3.535	17.800	0.272	0.386
Cr	5.451	3.643	0.213	0.348
Co	8.690	9.410	0.160	0.308
Se	5.909	4.895	0.220	0.298
Sr	12.648	5.365	0.034	0.162
Y	0.376	1.004	0.129	0.386
Zr	3.326	9.802	0.351	3.533
Hf	4.763	5.849	0.197	0.520
W	10.678	11.635	0.040	0.026

4. Discussion

In this study, zirconia milled with uncoated milling bits at medium speed (1500 rpm) and coated milling bits at high speed (2000 rpm) exhibited a higher average glossiness after sintering. Nano-diamond-coated milling bits achieved high gloss regardless of spindle speed, while only the medium speed produced better gloss for uncoated milling bits. In other words, the medium speed of 1500 rpm can be utilized for different milling bits. This finding suggests that nano-diamond-coated milling bits are less sensitive to spindle speed, while ordinary milling bits require optimization. The nano-diamond coating on the milling bits is believed to extend the tools' longevity, while this new finding revealed that zirconia milled with coated bits requires less polishing after sintering. It is worth noting that any polishing on hard ceramics can pose an occupational hazard (e.g., pneumosilicosis) to dental technicians [22]. If the polishing can be confined within a controlled environment, such as within the CAD/CAM equipment, this can help reduce the risk of such hazards. Further parameter refinement is necessary to achieve high-gloss, polishing-free milling.

This study found that spindle speed and coating did not produce a significant difference in surface roughness. Currently, there is no uniform standard for the acceptable threshold value of ceramic surface roughness and glossiness in clinical work. However, Jones et al. [23] suggested that patients are able to distinguish between differences in roughness values of at least 0.5 µm; thus, 0.5 µm is considered to be clinically acceptable. The surface roughness of ceramics is influenced by various factors, including the properties of ceramic materials, the production process, and the grinding and polishing of the restoration [24,25].

The milling of zirconia prostheses typically involves the use of ball end mills, while milling freeform dental restorations with thin walls can be challenging due to the potential for scratches that may compromise the final outcome. Diamond coating is often preferred for milling tools due to its excellent physical and chemical properties, particularly its low coefficient of friction [26]. Therefore, the current nano-diamond-coated tool can potentially improve the process and serve as a one-stop "cut and polish" tool. This makes it an ideal option for processing zirconia, as it can provide both cutting and polishing capabilities in a single tool.

This study showed that the sintering process increases the smoothness and glossiness of zirconia. Previous research has investigated the effect of sintering on ceramic materials. Due to the different structural characteristics of various types of ceramic materials, different materials exhibit different effects after sintering at varying temperatures and conditions. Specifically for pre-sintered zirconia blocks, they are made from yttria-stabilized, tetragonal zirconia powder that is pressed under static pressure and are pre-sintered at a specific furnace temperature. Pre-sintered zirconia has low hardness and excellent cutting performance, making it suitable for the fast and easy processing of personalized dental prostheses with complex shapes. A study by Ding et al. [27] examined the impact of semi-sintering temperature on zirconia processing performance. They found that zirconia green blank can be easily machined but with insufficient hardness when the pre-sintering temperature is below 1000 °C, while the best pre-sintering stability is around 1000 °C. Other sintering parameters, such as heating rate, sintering temperature, and holding time, also affect zirconia's microstructure and semi-permeable properties [28]. Different studies have produced varying results regarding the effect of holding time on zirconia's semi-permeability.

Upon comparing the roughness results and glossiness results, an interesting observation is the negative correlation between these two factors. A lower surface roughness tends to enhance the reflection of incident light, resulting in higher glossiness. Conversely, when the surface has higher roughness, the incident light is more easily diffused, leading to reduced glossiness. Heintze and Zimmerli [11] suggested the presence of a surface roughness threshold (Ra) of 0.3 μm. When the Ra value exceeds 0.3 μm, the glossiness remains low. However, there is a significant increase in surface glossiness when the Ra value falls below 0.3 μm. Additionally, surface roughness plays a crucial role in the formation of oral biofilms [29]. Decreasing roughness increases surface energy, resulting in a smoother surface that discourages biofilm formation and reduces the adhesion between bacteria and the zirconia ceramic surface. Thus, achieving smoothness is not only important for aesthetic and bonding purposes but also for enhancing the user experience and prolonging the service life of zirconia, as the presence of bacteria on the surface can degrade its mechanical properties [30].

The durability of milling bits tends to decrease with repeated use, as wear and coat peeling occur. The continued use of defective milling bits can have a negative impact on surface roughness and the adaptation of CAD/CAM restorations, leading to the formation of surface microcracks and critical defects [15]. High-speed and prolonged milling can accelerate the wear of milling tools, further compromising the quality of the prostheses [31]. The wear patterns of micro-milling bits are typically concentrated in small areas near the tool tip, with the tool tip experiencing micro-cracking. This is different from the wear observed in conventional-sized carbide milling bits, where wear primarily occurs on the flank. When micro-diameter milling bits are used to mill aluminum alloy, the main types of wear include coating peeling, diffusion wear, tool tip breakage, and bonding wear [15]. Tool wear in milling bits follows similar patterns to other types of tool wear. As the cutting force increases, so does the cutting temperature and friction force, which can cause damage to the tool and its coating. Unfortunately, such damage is not easily detectable through visual inspection. Therefore, relying solely on human inspection to ensure processing quality and tool life can be challenging.

Over the past 40 years, dental CAD/CAM technology has significantly reduced the workload for clinicians and technicians by automating processes such as scanning, designing, and milling. However, manual polishing still remains a crucial step in the surface finishing process despite the availability of various polishing tools. This study serves as a proof of concept, demonstrating the potential for achieving lower surface roughness and higher glossiness on zirconia surface through proper coating techniques and selecting an appropriate CNC spindle speed. It also suggests the possibility of utilizing the CNC milling machine to perform the polishing task, leading to a fully automatic workflow in the design and manufacturing of dental restorations.

Although milling bits can be used interchangeably by commercial dental CAD/CAM machines, milling parameters such as cut depth, cut width, cut force, feed rate, and spindle speed as well as the properties of the milling bits, such as their surface coating, can vary significantly. The current experiment only explored the influence of spindle speed and milling bit coating on the surface characterization of dental zirconia. Other milling parameters need to be examined to gain a more comprehensive understanding of their impact. Additionally, the experiment used new milling bits to minimize potential errors resulting from milling with abrasive bits. Therefore, it is important to investigate the lifespan of the coating and milling bits to ensure accurate results over time.

5. Conclusions

Under the conditions of the present study, it can be concluded that the nano-diamond-coated milling bits can be operated at various speeds, resulting in a higher gloss on the sintered zirconia block, while an ordinary, uncoated milling bit can only achieve the same glossiness at a designated speed. The type of milling bits and the speeds have no significant effect on the surface roughness.

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Conflicts of Interest: The authors declare no conflicts of interest.

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