

RESEARCH AND EDUCATION

## Effect of zirconia surface treatment on its wettability by liquid ceramics



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Rising esthetic expectations have fueled a search for new ways of making metal-free substructures for restorations. Although metal-ceramic restorations have been shown to be clinically successful, they have disadvantages such as imperfect esthetics and poor biocompatibility.<sup>1-4</sup> Ceramic restorations are both esthetically appealing and biocompatible.<sup>5-7</sup>

Since the 1990s, significant advances have been made in computer-aided design and computer-aided manufacturing (CAD-CAM) systems and oxide ceramics, particularly zirconia. Zirconia is used to fabricate post-and-cores, crown copings, frameworks for fixed-partial dentures, complete-contour crowns and fixed-partial dentures, implant abutments, and suprastructures of implant-supported fixed restorations.<sup>8</sup> When used as the framework for fixed prostheses, veneering ceramic (usually feldspathic porcelain) is fired onto the surface at a firing temperature slightly below 1000 °C. The veneering ceramic wets the framework surface and enhances the esthetics of the prosthesis. The clinical performance of a veneered zirconia restoration depends on the quality of the connection between the veneering and framework materials.<sup>7</sup> In clinical trials, problems with the chipping of the veneering porcelain (15% to 62%), cracks (25% to 50%), delamination

### ABSTRACT

**Statement of problem.** The wettability of the framework by liquid ceramics is important in ensuring a suitable bond between veneering ceramics and zirconia.

**Purpose.** The purpose of this in vitro study was to examine the dependence of the wetting angle on temperature to determine the transition temperature from nonwettability to wettability states and to calculate the values of the relative wetting forces of the milled surfaces.

**Material and methods.** Fifty zirconia cylinders were divided into 5 groups (n=10) and subjected to the following treatments: milling, grinding, polishing, and airborne-particle abrasion with Al<sub>2</sub>O<sub>3</sub> or SiC. After treatment, the specimens were rinsed, dried, and examined with respect to their wettability by liquid ceramics by using the automated Thermo-Wet test bench. The results were statistically analyzed by an ANOVA ( $\alpha=.05$ ).

**Results.** The most rapid wettability was obtained through airborne-particle abrasion with Al<sub>2</sub>O<sub>3</sub> at 930 °C. Additionally, the highest relative bond strength (with respect to the machined surface) was obtained with Al<sub>2</sub>O<sub>3</sub> abrasion.

**Conclusions.** Because of variations in the wettability of the zirconia surface after different treatment methods, the firing temperature of the ceramic should also vary depending on the type of surface treatment applied. Thus, it is determined individually according to the chosen method. (*J Prosthet Dent* 2019;122:410.e1-e6)

(>10.7%), and large fractures (3% to 33%) in veneered zirconia restorations have been reported to occur with unacceptable frequency.<sup>9-13</sup>

The quality of the bond depends on the chemical-diffusion bond, the connection resulting from the difference in the shrinkage of both materials, and the mechanical connection provided by microretentions formed by liquid ceramic penetrating the surface irregularities of the substructure. The mechanism of a chemical-diffusion bond has not been fully explained but likely involves the mutual dissolution of ceramics and zirconia.<sup>14,15</sup> Achieving a good bond between the veneering ceramic and zirconia is essential so that the

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## Clinical Implications

The wettability of ceramics depends on both the surface treatment and temperature. Determining the influence of temperature on the wetting angle and selection of the optimal surface treatment for zirconia may facilitate the task of selecting the optimal firing temperature and thus ensure optimal wettability of the substructure by liquid ceramics.

thermal expansion coefficient ( $\alpha$ ) can be adjusted. The veneering material should have an  $\alpha$  value that is equal to or slightly less than that of the framework material to minimize the tensile stresses in the ceramics.<sup>16</sup> Most manufacturers offer veneering ceramics whose expansion coefficient deviates slightly from the value typical for zirconia ( $\Delta\alpha$ ). The value of veneered ceramic is approximately 1 unit of  $\alpha$  lower ( $\Delta L/L \times 10^{-6} \text{ K}^{-1}$ ) than for zirconia, the  $\alpha$  value of which generally lies within the range of 10.5 to 11.0  $\times 10^{-6} \text{ K}^{-1}$ . These values are applied in most ceramic-porcelain systems, as well as in others made purely with ceramics without zirconia.<sup>14</sup> Achieving the proper thermal expansion coefficient is possible, but other factors such as uniform adherence and adhesion between the zirconia and ceramics are also key to a successful outcome.

An important factor ensuring the quality of the bond is the wettability of the substructure.<sup>17</sup> In dental technology, 2 approaches to wettability are used. The first is wetting by water (hydrophobicity or hydrophilicity, which is relevant when ceramics are applied onto the framework in the form of a water suspension).<sup>7</sup> This ensures a suitable and homogeneous distribution of the ceramic layer on the framework surface, which is one of the factors impacting the quality of the bond. At this stage, the ceramics should penetrate all surface irregularities and concavities. The second approach is wetting by liquid (molten) ceramics at the firing temperature. In this case, wetting by liquid ceramics should be as high as possible to ensure its flow into surface irregularities.

Increasing the adhesive force and ensuring proper treatment of the substructure is essential for achieving microretention of the surface for liquid ceramic penetration. With traditional ceramics, the surface is usually developed by grinding with rotary instruments, airborne-particle abrasion, acid etching, or a combination of these techniques.<sup>18</sup> Unfortunately, the properties of zirconia differ from those of traditional silica-based materials. Its hardness is comparable with that of the abrasive materials used; therefore, zirconia cannot be treated conventionally and requires more aggressive processing. Acid etching is not effective on account of the durability and chemical strength of zirconia.<sup>19,20</sup> Another approach is

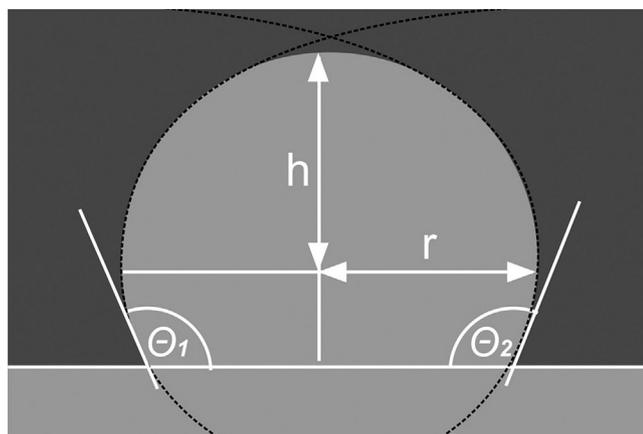
airborne-particle abrasion with abrasives that have similar ( $\text{Al}_2\text{O}_3$ ) or slightly higher (SiC) hardness than that of zirconia. Thus, treatment is possible but is less efficient than with metals. The manufacturers provide no clear guidelines regarding airborne-particle abrasion treatment, and scientific studies have provided inconsistent results. For example, Teng et al<sup>20</sup> reported that airborne-particle abrasion increases the bond strength between ceramics and zirconia.<sup>21,22</sup> However, according to Fischer et al,<sup>22</sup> airborne-particle abrasion is unnecessary for surface treatment. Some scientific studies support the view that airborne-particle abrasion offers no significant benefit to adhesion, regardless of the aluminum oxide grain size.<sup>16,23</sup> Another study reported the negative effects of airborne-particle abrasion on the structure of zirconia.<sup>24</sup>

The purpose of this in vitro study was to examine the wettability of zirconia by liquid ceramics at varying temperatures, depending on the surface treatment method applied, with a view to selecting the optimal ceramic firing temperature. It was hypothesized that different surface treatments would affect the wettability of zirconia by liquid ceramics.

## MATERIAL AND METHODS

Fifty cylinders ( $\phi 8.0 \times 2.0$  mm) of sintered zirconia (Ceramill Zi; Amann Girrbach AG) were divided into 5 groups ( $n=10$ ) and subjected to the following treatments: milling, grinding, polishing, and airborne-particle abrasion with  $\text{Al}_2\text{O}_3$  or SiC particles. Wet milling was carried out using a 5-axis milling unit (Ceramill Motion; Amann Girrbach AG). Cylinders were ground on a rotary grinder (Metasinex; Metasinex Row) by using SiC abrasive paper with a grit size ranging from 220 to 2400 under water cooling. The specimens were treated for 20 seconds with an airborne-particle abrasion process (Microblast Duo; Riley Industries Ltd) by using grain gradation of 110  $\mu\text{m}$ , a stream angle of 45 degrees, and a working distance of 10 mm from the nozzle. The specimens were washed in ethanol for 10 minutes in an ultrasonic washer (Quantrex 90 WT; L&R Manufacturing, Inc) and dried with compressed air. The wettability of the specimens by liquid ceramics (Sakura Interaction; Elephant Dental B.V.) was tested by using the automated Thermo-Wet test bench over a temperature range of 650 °C to 1700 °C. The images were recorded by using a charged coupled device (CCD) camera equipped with a set of vision filters from the Thermo-Wet laboratory stand. To determine surface tension and contact angles, the geometric features of the objects were extracted from these images by axisymmetric drop shape analysis (ADSA).

The contact wetting angle was determined from the 3-phase point of tangency according to the Young equation. The geometric parameters of the specimen



**Figure 1.** Measured geometric parameters of specimen.

shown in [Figure 1](#) were as follows:  $r$ , the maximum drop radius;  $h$ , the drop height over the equatorial plane; and  $\theta_1$  and  $\theta_2$ , the left and right wetting contact angles. Contact angles were calculated based on images made at different temperatures after various surface treatments and used to prepare a graph representing the influence of temperature on contact angle  $\theta$ .

The relative wetting force was calculated by using one of the tests (surface after milling), which was set as the reference treatment. This test was selected as the reference treatment because such a state is exhibited by the surface of the prosthetic elements after they have been cut from blocks. The wetting force  $F_c$ , which takes into account surface tension  $\sigma_{LV}$ , wetting contact angle  $\theta_0$ , and specimen circumference  $O_p$ , was obtained from the following relation:

$$F_c = O_p \sigma_{LV} \cos \theta_0 \quad (1)$$

Assuming that the specimens have similar circumferences, the relative wetting force could be determined as the ratio of the wetting contact angles to the extreme wetting contact angles obtained in the previous experiments:

$$\frac{F_2}{F_1} = \frac{O \cdot \sigma_{LV} \cdot \cos \theta_2}{O \cdot \sigma_{LV} \cdot \cos \theta_1} = \frac{\cos \theta_2}{\cos \theta_1} \quad (2)$$

Statistical analyses of the results were conducted by using the R statistical software. A 2-factor ANOVA and a post hoc Tukey test were conducted ( $\alpha = .05$ ).

## RESULTS

The conducted tests made it possible to determine the influence of temperature on the wetting contact angle for particular specimens. Images of drops formed during the melting of a ceramic bond on various substrates at a temperature of 930 °C are shown in [Figure 2](#). The shape of the drop changes depending on the treatment, and

this in turn is related to wettability. A graphical representation of the dependence of temperature and surface treatment methods on contact angle  $\theta$  is shown in [Figure 3](#).

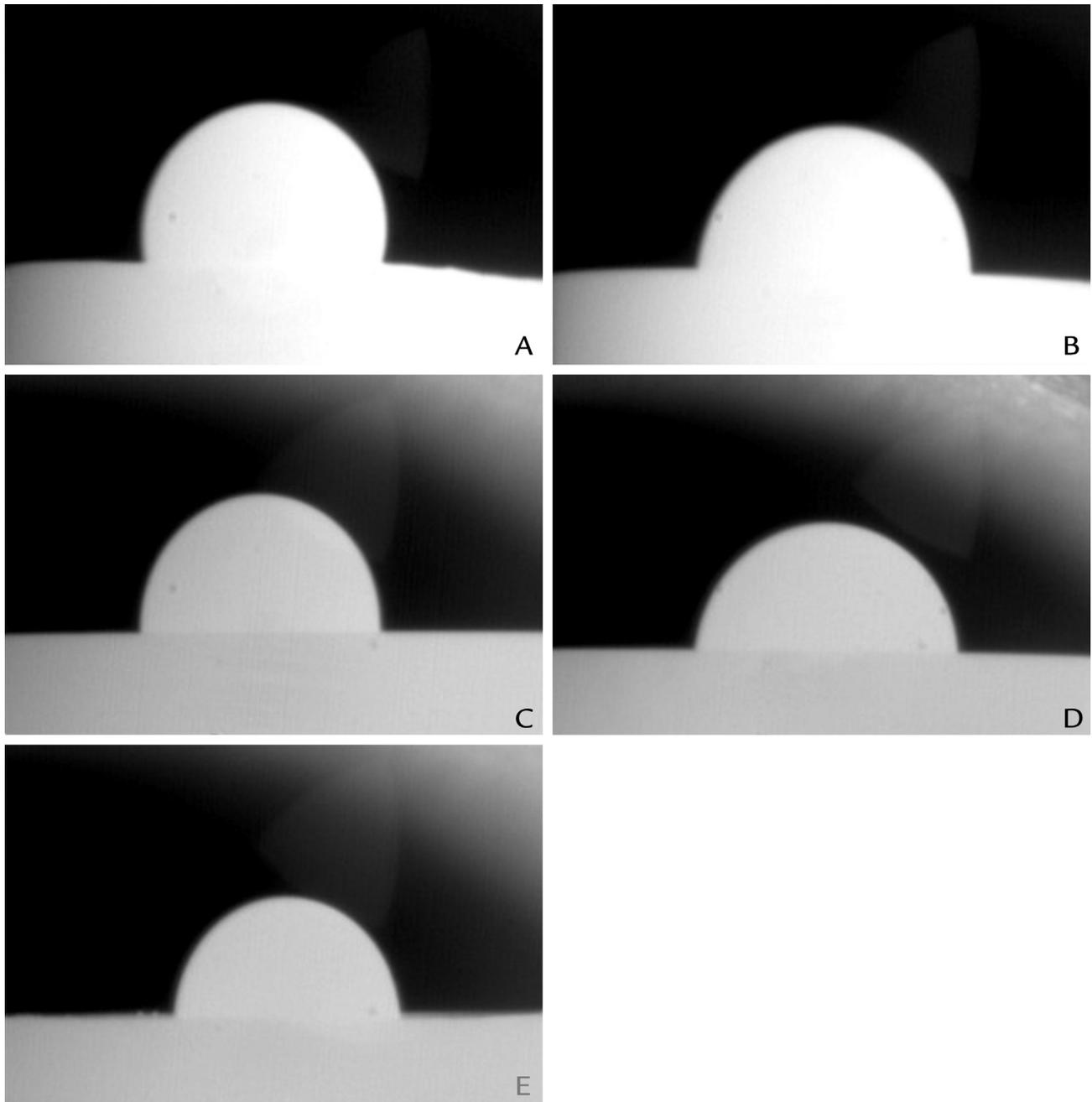
As the temperature increased, the wetting contact angle decreased, while the liquid ceramic wettability of the treated material increased ([Fig. 3](#)). Additionally, depending on the type of surface treatment, differences were observed in the wettability of the substructure. The diagram shown in [Figure 3](#) indicated the temperature at which liquid ceramics began to wet the treated substructure.

Results from each treatment method differed significantly from the others ([Table 1](#)). The results with the highest wetting contact angles were observed after polishing, followed by grinding, milling, and airborne-particle abrasion with SiC and Al<sub>2</sub>O<sub>3</sub>. Significant differences were also noted as a result of temperature. The highest contact angles were observed at a temperature of 875 °C and declined steadily with each subsequent 10 °C increase. The correlation between the treatment method and the temperature played an important role. The decline in results accompanying an increase in temperature varied depending on the treatment method used ([Fig. 4](#)). The flattest course was observed after milling, when the drop-off in results accompanying a temperature rise was the least severe, indicating that the angle decreased when the temperatures were low. In turn, the decrease in angle  $\theta$  was relatively low at higher temperatures, and the highest angle  $\theta$  results were noted after milling. Regarding the other treatment methods, the decrease in the  $\theta$  angle when the temperature increased was greater, and the results were similar in all cases.

The influence of temperature on the relative wetting force, calculated according to equations (1) and (2), is presented in [Figure 4](#). The greatest force was observed in the specimens abraded with SiC or Al<sub>2</sub>O<sub>3</sub> as well as in the ground specimens, and these values were significantly higher than the wetting force of the milled specimens. In comparison with milled specimens, a slight decrease in the relative bond strength was noted in polished specimens. A decrease was observed in both the relative wetting force and the temperature in all the milled specimens.

## DISCUSSION

The results of this study demonstrated that milled zirconia elements without additional surface treatment did not provide the best wettability; therefore, it was difficult to ensure a proper bond between the ceramics and the zirconia framework. These findings support the hypothesis that different surface treatment methods affect the wettability of zirconia by liquid ceramics.



**Figure 2.** Selected images of drops at 930 °C. A, Polished. B, Milled. C, Ground. D, Abraded with Al<sub>2</sub>O<sub>3</sub>. E, Abraded with SiC. (Original magnification ×20).

Previous studies have focused on wetting by metals during soldering, and information on the wetting of prosthetic substrate materials by liquid ceramics is lacking. The present discussion is therefore limited to the authors' own research.<sup>25</sup> The optimal firing temperature was determined by various factors such as wetting the substrate with liquid ceramics during firing, which means achieving a contact angle below 90 degrees. Tests were conducted at temperatures similar to firing temperatures to achieve an optimal contact angle.

Once the framework has been fabricated, the zirconia should undergo further surface treatment. Ceramics begin

to wet the substructure at temperatures above 930 °C, which is higher than the firing temperature recommended by manufacturers (a maximum of 920 °C).<sup>17</sup> Therefore, in practice, the firing process is not performed at a temperature that is optimal for wettability.

The wetting force parameter is used to determine the suitability of materials for a specific technology processing method. This parameter is described as the product of the surface tension and the cosine of the wetting contact angle. In the present study, it was established that when the specimens were polished, abraded with either SiC or Al<sub>2</sub>O<sub>3</sub>, and then ground, the

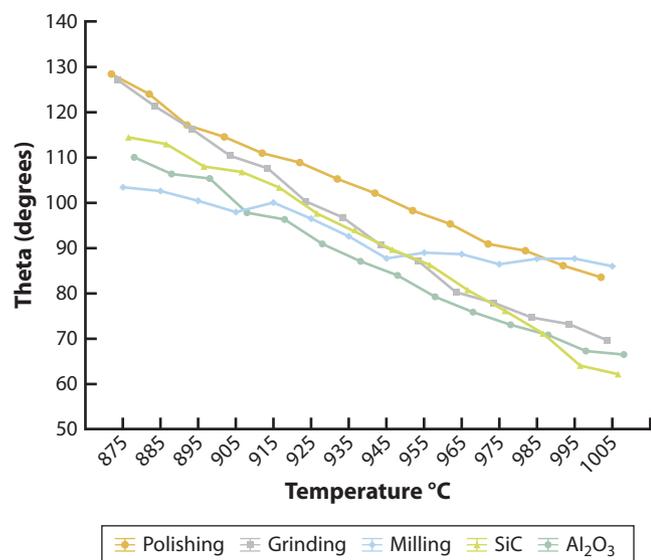


Figure 3. Effect of temperature on wetting angle.

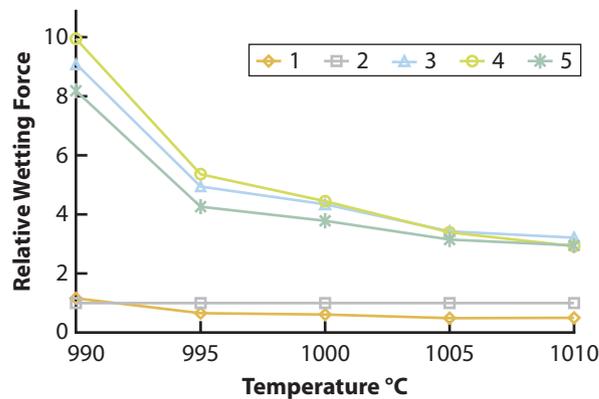


Figure 4. Relative wetting force for specimens 1, 3, 4, and 5 in relation to reference specimen 2, depending on temperature: 1—polished, 2—milled, 3—abraded with SiC, 4—abraded with Al<sub>2</sub>O<sub>3</sub>, 5—ground.

Table 1. Wetting angle (degrees) depending on treatment method and temperature

Temperature (°C)	Treatment (Mean ±Standard Error)					Total
	Polishing	Grinding	Milling	SiC	Al <sub>2</sub> O <sub>3</sub>	
875	128.39 ±0.4	127.17 ±0.47	103.41 ±0.32	114.43 ±0.44	110.07 ±0.28	116.69 ±1.40 <sup>a</sup>
885	124.06 ±0.33	121.31 ±0.44	102.63 ±0.37	112.97 ±0.33	106.36 ±0.42	113.47 ±1.19 <sup>b</sup>
895	117.12 ±0.38	116.18 ±0.39	100.5 ±0.43	108.05 ±0.4	105.35 ±0.34	109.44 ±0.92 <sup>c</sup>
905	114.51 ±0.4	110.34 ±0.28	97.99 ±0.48	106.89 ±0.41	97.76 ±0.41	105.50 ±0.97 <sup>d</sup>
915	110.95 ±0.36	107.61 ±0.24	100.12 ±0.44	103.36 ±0.46	96.29 ±0.35	103.67 ±0.76 <sup>e</sup>
925	108.9 ±0.41	100.3 ±0.32	96.5 ±0.35	97.69 ±0.32	91.00 ±0.31	98.88 ±0.85 <sup>f</sup>
935	105.25 ±0.34	96.79 ±0.31	92.6 ±0.3	94.07 ±0.24	87.12 ±0.24	95.17 ±0.86 <sup>g</sup>
945	102.09 ±0.31	90.67 ±0.42	87.77 ±0.47	89.62 ±0.44	84.00 ±0.34	90.83 ±0.88 <sup>h</sup>
955	98.27 ±0.37	87.07 ±0.25	89.03 ±0.3	86.39 ±0.24	79.23 ±0.29	88.00 ±0.88 <sup>i</sup>
965	95.38 ±0.35	80.25 ±0.39	88.65 ±0.29	80.75 ±0.34	75.89 ±0.39	84.18 ±1.00 <sup>j</sup>
975	90.89 ±0.37	77.9 ±0.23	86.51 ±0.36	76.28 ±0.35	73.06 ±0.44	80.93 ±0.97 <sup>k</sup>
985	89.4 ±0.35	74.68 ±0.3	87.72 ±0.37	71.14 ±0.33	70.72 ±0.37	78.73 ±1.17 <sup>l</sup>
995	86.16 ±0.27	73.19 ±0.37	87.68 ±0.24	64.12 ±0.39	67.35 ±0.44	75.70 ±1.38 <sup>m</sup>
1005	83.53 ±0.39	69.75 ±0.37	86.07 ±0.39	62.20 ±0.4	66.53 ±0.24	73.62 ±1.36 <sup>n</sup>
Total	103.92 ±1.16 <sup>a</sup>	95.23 ±1.56 <sup>b</sup>	93.37 ±0.54 <sup>c</sup>	90.57 ±1.45 <sup>d</sup>	86.48 ±1.23 <sup>e</sup>	—
ANOVA	Factor	F	P	Partial eta <sup>2</sup>	Power	
	Treatment	4518	<.001	0.9663	1.0000	
	Temperature	7890	<.001	0.9939	1.0000	
	Treatmentxtemperature	213	<.001	0.9461	1.0000	

wetting force increased at a temperature of 990 °C with respect to the milled specimens. The wetting force for polished specimens was 15% greater than that of the milled specimens and significantly higher for the remaining specimens, reaching a maximum value with specimens abraded with Al<sub>2</sub>O<sub>3</sub>.

This is also illustrated in Figure 3, where the wetting contact angle theta is minimal in a particular test in relation to the strongest wetting. The optimal bond, given the energetic state of the surface, was obtained for specimens that had undergone the following surface treatments:

airborne-particle abrasion with SiC (specimen 3) or Al<sub>2</sub>O<sub>3</sub> (specimen 4) and grinding (specimen 5).

As the contact angle varies with the treatment method used, this would suggest that it depends on various parameters such as roughness and free surface energy, which affect the condition of the surface after application of various treatments. These parameters differ from one another; therefore, further research should aim at determining the effect of these parameters on wettability.

Changes in the wetting contact angle over time should also be identified. Studies conducted thus far

indicate that such a dependency exists when airborne-particle abraded surfaces are wetted with liquid metals.<sup>25</sup> A similar situation should be expected in the case of liquid ceramics. In addition, wettability is only one of the factors ensuring the suitability of a ceramic fused to a framework and is not the only parameter that should be considered. The higher the temperature, the better the wettability of the substructure.<sup>26</sup> However, limitations exist regarding the maximum firing temperatures that can be used. Therefore, wettability should be just one of the factors considered, and it does not necessarily have to be the most important. Different recommendations and standards indicate the required minimum coping-ceramic bond strength. Another area that requires further research is the effect of wettability on this bond strength.

## CONCLUSIONS

Based on the findings of this *in vitro* study, the following conclusions were drawn:

1. The type of surface treatment of zirconium oxide affects the wettability of its liquid ceramic surface.
2. The firing temperature should be determined individually, according to the chosen method.

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