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Effect of operating air pressure on tribochemical silica-coating

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Abstract

Objective. Alumina and zirconia are inert to conventional etching and need to be initially conditioned with, for example, silicatization. The aim of the present study was to evaluate the effect of operating air pressure of tribochemical silica-coating method on the shear bond strength of composite resin to ceramic substrates. **Material and methods.** Alumina (Procera Alumina, Nobel Biocare) and zirconia (LAVA; 3M ESPE and Procera Zirconia; Nobel Biocare) were airborne particle silica-coated (CoJet; 3M ESPE) using selected, clinically available air pressures of 150, 220, 300, and 450 kPa. The surfaces were silanized with silane coupling agent (ESPE Sil; 3M ESPE) and coated with adhesive resin (3M Multipurpose resin; 3M ESPE). Particulate filler resin composite (Z250; 3M ESPE) stubs (diameter 3.6 mm, height 4.0 mm) were added onto ceramics and light-cured for 40 s. The test specimens ($n = 18/\text{group}$) were thermocycled ($6000 \times 5-55^\circ\text{C}$) and shear bond strengths were measured with a cross-head speed of 1.0 mm/min. Fracture surfaces were examined with SEM, and an elemental analysis (EDS) was carried out to determine silica content on the substrate surface. **Results.** The highest bond strengths were obtained with the highest pressures. ANOVA showed significant differences in bond strength between the ceramics ($p < 0.05$) and between the specimens treated at various air pressures ($p < 0.05$). **Conclusions.** Clinically, the operating air pressure of silicatization may have a significant effect on bond strength to non-etchable ceramics.

Key Words: Alumina, airborne-particle silica-coating, silanization, Y-TZP, zirconia

Introduction

The increasing demand for biocompatibility and esthetics in restorative dentistry has led to the development of all-ceramic systems in which oxide ceramics and special manufacturing techniques, e.g. stabilization of the crystal lattice, are used to optimize their strength. Aluminum trioxide (Al_2O_3) and yttria stabilized tetragonal zirconium dioxide (Y-TZP) ceramics have significantly improved mechanical properties, thus providing a wide range of clinical applications such as root canal posts, crowns, veneers, fixed partial dentures, implants, and implant abutments [1]. For clinical applications, such as veneers, reliable bonding of composite resin cement to the ceramics is required.

Although hydrofluoric acid etching is effective in improving the silanizability of all-ceramic systems based on glass ceramics [2,3], etching cannot improve the resin composite bond strength to high-purity oxide ceramics [4–6]. It is clinically well known that these ceramic surfaces can be

successfully conditioned with silica-coating [7] followed by silanizing with a silane coupling agent [3,8] and become reactive to consequent treatment with conventional adhesive resins. Nowadays, however, several products are available based on, for example, modern organophosphate resin composites that, unlike the products and methods dealt with in the present study, do not require silicatization or silanization in order to be reactive and effective.

Tribochemistry involves creating chemical bonds by applying kinetic energy [9], e.g. in the form of sandblasting, without any application of additional heat or light. Tribochemical silica-coating or silicatization using CoJetTM at the dentist's surgery is a widely used and accepted conditioning method in ceramic and metal alloy structure reparation and cementing. This is a cold silicatization method, with the energy needed in the silicatization process transferred to the object material in the form of kinetic energy, which generates frictional heat locally

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at the impact focus. Silicatization takes place macroscopically, however, without any change in temperature [7]. The CoJet system is based on airborne micro-blasting sand, which is especially silica-modified aluminum trioxide and provides the ceramics with a reactive silica-rich outer surface prone to silanization and the following resin adhesion for cementing with suitable resin composites.

There are five key parameters in airborne particle silicatization: nozzle distance from the substrate surface, impact angle to the substrate surface, working time, coverage area, and, in particular, operating air pressure [10]. The CoJet system offers some benefit in direct repairs to bond resin composites to fractured ceramic restorations [11]. Furthermore, the adhesion of a conventional resin to ceramics with airborne particle abrasion without silicatization and silanization is inadequate [12]. Nonetheless, using resin composites with special adhesive organophosphate monomers, a long-term durable resin bond strength to airborne particle abraded alumina ceramic can be achieved [13].

Several studies have shown that these methods might also work for zirconia and alumina ceramics [14–17]. However, there are results suggesting that the coverage area of silica-coating can vary, and not much has been presented in the literature [18]. The answer is likely to be found in the parameters and their variation within the CoJet method.

Clinically, it is quintessential and often mandatory for several reasons to follow the manufacturer's recommendations for the working parameters listed above. Still, it is important to evaluate scientifically, and compare critically, the reliability of various repairing and cementing methods for oxide ceramic constructions. Modern organophosphates have been studied and suggested for cementing in several *in vitro* studies concerning bonding of resin composites to glass ceramics and oxide ceramics. Those studies have mainly dealt with the features of different luting cements [19,20] and with different surface conditioning methods of the ceramic surface [13,15,20–24] as well as with the effect of the presence or absence of silica-coating achieved with various methods [15,18,24–26]. For the time being, bonding procedures based on organophosphates are found superior to silicatization and silanization. However, it is possible that alterations in silicatization methods and novel silanes may change the present situation. As part of the investigation into alternative oxide ceramic bonding methods, the aim of the present study was to evaluate *in vitro* the effect of the operating air pressure of the CoJet airborne particle silicatization on the bond strength of composite resin to sintered alumina and zirconia by using dioxide by using the various operating air pressures available in the dentist's surgery. To our knowledge, information concerning the effect of the operating air pressure used in airborne silicatization systems

has been lacking. The hypothesis is that there will be differences in shear bond strengths when different operating air pressures are used.

Material and methods

Ceramic materials

The materials of the study are listed in Table I. Seventy-two square-shaped samples ($2 \times 10 \times 10$ mm) of each ceramic were embedded within cylinders (diameter 20 mm, height 10 mm) with an acrylic resin to form the bonding substrate. The resin composite stubs were photo-polymerized on to this surface. The surfaces of the ceramic substrates were examined with a scanning electron microscope (SEM; JEOL Scanning Electron Microscope JSM 5500, PGT Prism 2000-Si(Li) EDS detector, JEOL, Tokyo, Japan). The substrate surfaces were initially wet ground (Struer Lappol; Struers, Copenhagen, Denmark) with silicon carbide abrasive paper of 1000 grit (FEPA standard 42-1984 R 1993, Federation of European Producers of Abrasives). The substrates were then randomly divided into four groups for tribochemical silicatization.

Airborne particle silicatization

The operating air pressures selected in the airborne particle silicatization system (CoJet; 3M ESPE, Seefeld, Germany) were 150, 220, 300 or 450 kPa. Duration of the abrasion was a constant 10 s and perpendicular distance between the tip of the airborne-particle hand-piece and the substrate was a constant 10 mm, as given in the CoJet system instructions. Silica-coated aluminum trioxide particles (CoJet Sand, particle diameter 30 μ m; 3M ESPE, Seefeld, Germany) were applied. All the treatments were carried out by the same operator to keep the experiments comparable with each other.

Silane, resin, and resin composite

The substrate surfaces were then silanized with a fresh, unopened silane coupling agent (ESPE Sil; 3M ESPE, Seefeld, Germany) containing 3-methacryloxypropyl-trimethoxysilane as the active agent. One coat of silane was applied with a fine brush onto the substrate surfaces and covered with light-polymerizable adhesive resin (3M Multipurpose resin; 3M ESPE, St Paul, Minn., USA) containing bisphenol-A-glycidyl dimethacrylate (BisGMA) and 2-hydroxyethylmethacrylate (HEMA). The silane coating was allowed to dry for 5 min, as recommended by the manufacturer, and the adhesive resin was light-polymerized for 10 s (Demetron Optilux; Kerr, Orange, Calif., USA). Wavelength maximum of the light was 495 nm and light intensity was 550 mW/cm². A particulate filler resin composite (Z250, 3M ESPE, St Paul, Minn., USA) was applied to form

Table I. Materials used in the present study

Brand	Manufacturer	Main composition	Batch no.
CoJet™ sand	3M ESPE, Seefeld, Germany	Silica-coated aluminum trioxide particles (30 µm in diameter)	161891
Espe Sil	3M ESPE, Seefeld, Germany	MPS silane diluted in alcohol	140046
Multipurpose resin	3M ESPE, St Paul, Minn., USA	Bis-GMA+HEMA-based resin	4NR 20040315
Filtek™ Z250	3M ESPE, St Paul, Minn., USA	Particulate filler restorative composite	4GP 20040310
Procera Alumina	Nobel Biocare, Gothenburg, Sweden	Aluminum trioxide	–
Procera Zirconia	Nobel Biocare, Gothenburg, Sweden	Yttria stabilized tetragonal zirconia	–
LAVA	3M ESPE, Seefeld, Germany	Yttria stabilized tetragonal zirconia	–

stubs 3.6 mm in diameter and 4.0 mm in height. The stubs were light-polymerized for 40 s.

Thermocycling and shear bond

Thermocycling was used as an artificial ageing method: 18 specimens of each group were thermocycled 8,000 times at between 5°C and 55°C (Thermocycling unit 8I, custom-made; Biomechanical Testing Laboratory, University of Turku, Turku, Finland). After thermocycling, the test specimens were stored in water (37°C) and then immediately placed in the holder of a universal testing machine (Lloyd Instruments LRX Material Testing Machine; Lloyd Instruments, Fareham, UK) measuring shear bond strength of the resin composite stub to the ceramic material. A cross-head speed of 1 mm/min with a parallel knife-edge blade touching the interface of the ceramic and resin composite cylinder was used, and the debonding fracture load was registered. Debonding loads were calculated as shear stress (MPa), i.e. by dividing the failure load (N) by the bonding area (mm²).

Scanning electron microscopy (SEM), failure mode analysis, and energy dispersive spectroscopic analysis (EDS)

Fracture surfaces on the ceramic substrate were examined using a scanning electron microscope (SEM, JEOL Scanning Electron Microscope JSM-5500, PGT Prism 2000-Si(Li) EDS detector; JEOL, Tokyo, Japan), with magnifications 100-, 500- and 2000-fold, in order to classify the failure mode visually. The failure type of de-bonding was defined as ‘cohesive’ when about two-thirds or more of the resin composite cement was left on the ceramic surface, and as ‘adhesive’ when less than one-third remained. Failure mode was designated as ‘mixed’ when it fell between the above limits. Furthermore, an energy dispersive spectroscopic analysis (EDS) was carried out to determine silica content on the substrate surface.

Statistical analysis

Two-way analysis of variance (ANOVA) followed by the Tukey post-hoc test and Weibull analysis were

used as statistical methods. Weibull distribution, designed to compute failure probabilities as a function of applied stress, was used to characterize the nature of the shear bond strength by fitting the statistical distribution to life data in order to estimate the failure probability.

Results

Shear bond strength of the resin composite to each ceramic material is given in Table II for the different operating air pressures. The shear bond strengths of both zirconium dioxide ceramics (LAVA; Procera Zirconia) increased as the air pressure was increased from 150 kPa to 450 kPa. The shear bond strengths of the aluminum trioxide (Procera Alumina) also increased as the air pressure was increased, although the increase was observed only with air pressures above 220 kPa. Two-way ANOVA revealed that both factors, ceramic material ($p=0.03$) and air pressure ($p<0.001$), had significant effects on bond strength values. There was no interaction between factors (ceramic material and pressure) ($p=0.387$).

According to the scanning electron microscope and visual evaluation, all the fractures in every group were in the adhesive. Figures 1–3 show the fracture probability of the bond between the ceramic material and resin composite with different shear stress levels. The curves of the fracture probability of both LAVA and Procera Alumina in the groups with the operating air pressures of 150 kPa and 220 kPa cross each other, indicating apparently higher shear bond strength of the group treated with lower air pressures. The groups treated with 300 kPa and 450 kPa air pressure show higher shear bond strengths than the other two groups.

Table II. Mean shear bond strength (SD) for each material and air pressure

	LAVA	Procera Alumina	Procera Zirconia
150 kPa	12.61(2.64) ^a	18.37 (2.35) ^a	11.21 (2.09) ^a
220 kPa	13.39 (3.98) ^{ab}	15.56 (3.00) ^{ab}	15.01 (3.00) ^{ab}
300 kPa	17.13 (4.92) ^{ab}	27.09 (3.31) ^{ab}	22.76 (2.31) ^{bc}
450 kPa	18.22 (3.11) ^b	30.49 (3.00) ^b	26.43 (2.39) ^c

Superscript letters indicate homogenous subsets (Tukey, $p<0.05$) between used air pressure within ceramic material.

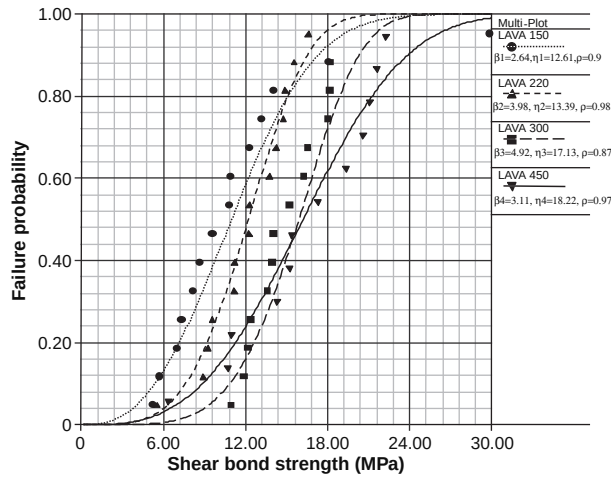


Figure 1. Weibull graphs of shear bond strengths of composite resin to zirconium dioxide (LAVA) as a function of air pressure of the CoJet™ system.

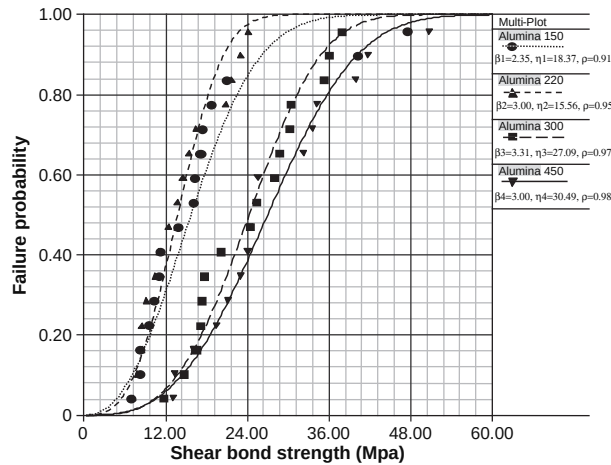


Figure 2. Weibull graphs of shear bond strengths of composite resin to aluminum trioxide (Procera Alumina) as a function of air pressure of the CoJet™ system.

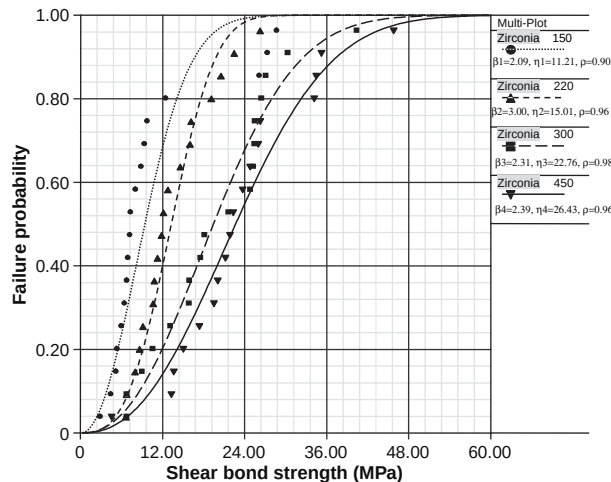


Figure 3. Weibull graphs of shear bond strengths of composite resin to zirconium dioxide (Procera Zirconia) as a function of air pressure of the CoJet™ system.

The results of the EDS analysis are given in Table III. The silica content remained the same or even diminished on the LAVA zirconia surfaces when the air pressure was increased. However, the content increased on the Procera Alumina and Procera Zirconia surfaces when air pressures higher than 220 kPa were used.

Discussion

Our study suggests that the higher the air pressure used in the CoJet silica-coating system, the more reliable and higher the shear bond strengths that can be obtained between the resin composite and the ceramic material. SEM imaging revealed that higher operating air pressure may have caused increased surface roughness of the ceramic material. This can be explained by higher kinetic energy causing at the same time a higher embedding rate of the silica-coating particles. That being the case, the deeper particle penetration causes a more stable attachment of the silica particles to the ceramic material [27]. Furthermore, one can assume that the number of particles blasted and attached to the surface has been higher per time unit with higher operating air pressures. This, for its part, increases the silicon content on the ceramic surface. A similar tribochemical method called Rocatec™ (3M ESPE, Seefeld, Germany) used in dental laboratories has been studied previously, but particle size of the Rocatec system is essentially different (110 μm) [7].

It is important to note that the increased amount of silica and its probable higher embedding rate led to a considerable increase in the shear bond strengths as the operating air pressure was increased. The trend between the operating air pressure and the shear bond strengths was evident for both ceramics in this study. Aluminum trioxide, however, implied higher air pressures than 220 kPa, because at this level no increase in the bond strength could be observed compared with the 150 kPa air pressure level. Differences in the behavior of the materials are due to different kinds of surface properties affecting their ability to receive the silica-coating and can partially explain it. Aluminum trioxide surfaces have larger grains, as one can see in the SEM images (Figures 4 and 5). Alumina grains on the ceramic surface probably provide the adhesive agents with more microretention. In the SEM images (Figures 6 and 7), zirconium dioxide surface appears to be more homogenous. Zirconia also has the ability to transform into another crystallographic form due to mechanical stress, e.g. airborne particle abrasion [28], which causes volume changes in its crystal lattice. The demand for higher operating air pressure seemed to be essential especially for aluminum trioxide, which is a very hard and rigid material [29]. Zirconium dioxide is again a more ductile

Table III. Results of the Weibull analysis for shear bond strength and the EDS analysis for silica content on the surface of ceramics. Values in parentheses represent standard deviations

Air pressure (kPa)	3M-ESPE LAVA (Y-TZP)				Procera Alumina (Al ₂ O ₃)				Procera Zirconia (Y-TZP)			
	m		s		m		s		m		s	
	r		SiO ₂ (wt%)		r		SiO ₂ (wt%)		r		SiO ₂ (wt%)	
150	2.64	12.61	0.94	6.60 (0.20)	2.35	18.37	0.91	6.63 (0.25)	2.09	11.21	0.90	3.59 (1.19)
220	3.98	13.39	0.98	6.52 (0.47)	3.00	15.56	0.95	6.52 (0.47)	3.00	15.01	0.96	3.93 (0.51)
300	4.92	17.13	0.87	6.64 (0.78)	3.31	27.09	0.97	8.19 (0.67)	2.31	22.76	0.98	4.66 (0.53)
450	3.11	18.22	0.97	5.80 (0.42)	3.00	30.49	0.98	10.82(0.40)	2.39	26.43	0.96	4.99 (0.59)

material than alumina and its surface hardness is lower [30].

It has recently been demonstrated that shear bond strength values of resin composite luting cement to Procera Alumina substrates are relatively low after thermocycling [3]. However, surfaces were silica-coated with the Rocatec system, in which 110 µm sized silica-coated aluminum trioxide particles are used compared to the 30 µm particles of the CoJet system. The use of larger-sized abrasive particles causes more intensive surface abrasion, because the wear of the surface increases in proportion to the square of the diameter of the abrasive particle [27]. Surface roughness of the substrate treated with various airborne particle abrasion systems therefore differs, leading to differences in surface bonding properties as well [31]. The kinetic energy carried by the coating particle creates focally high temperatures (up to 1200°C) as the particle hits the surface. However, airborne silica-coating is still a cold silicization method and thus thermal stress or distortion within the metal framework or the ceramic matrix during the process can be avoided [7].

From the point of view of the clinician, it is noteworthy that the CoJet device can be connected to the dental unit by various types of connector. If the Kavo™ type connector is used for the CoJet hand-piece, it has to be taken into account that the operating air pressure of a standard dental unit is typically limited up to 250 kPa. This might mean that optimal shear bond strengths cannot be achieved with standard clinical equipment. The compressed air for the airborne particle abrasion device can also be taken before the pressure throttle of the dental unit or directly from the compression pump unit to achieve sufficient operating air pressure. However, care has to be taken not to increase the air pressure to a level that could damage the surface of the restoration or lead to crack propagation in the bulk material. According to the present studies, the effects of airborne particle abrasion have been reported variously as an increase or decrease in flexural strength, depending on the surface damage introduced by the abrasion method used [32]. Thus, in future studies, the matter will be of the utmost importance, but within the present study impossible to estimate.

Another key point of the tribochemical treatment taking place at the dentist's surgery is the aspect of preventing saliva or any other contamination from getting on to the prosthodontic structure during fitting, checking, and handling. This is often difficult to carry out and to avoid at least partial destruction of the silica-coated surfaces [33]. In many cases, the dental laboratory conducts the tribochemical treatment on the ceramic surfaces, but the idea of relying wholly on the laboratory pretreatment contains an error source. In addition, all the steps in the surface treatment, including the sufficient operating air

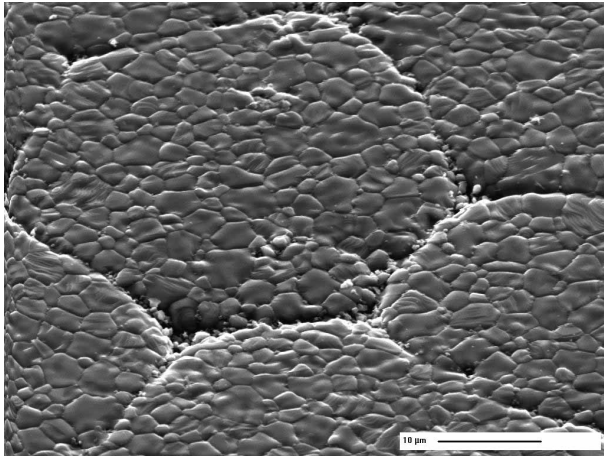


Figure 4. The intaglio surface of Procera Alumina (2000 $\times$, tilted). The horizontal bar denotes 10 μm .

pressure, must be carried out meticulously because silicatization does not cover the entire treated surface and the silica content rises only moderately [34].

In the present study, some defects were detected in the results of the Weibull analysis for zirconia. The analysis was not as uniform and curve-fitted as it was originally thought to be, and might partly have been due to the high standard deviation in the results and

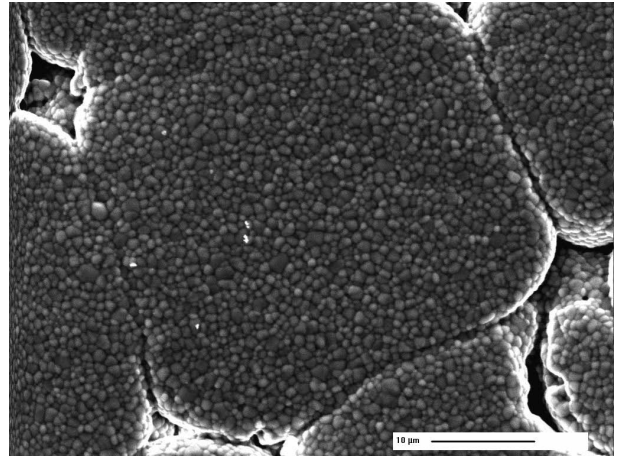


Figure 6. The intaglio surface of Procera Zirconia (2000 $\times$). The horizontal bar denotes 10 μm .

thus operator sensitivity in silicatization, silanization, or bonding the resin composite to zirconia. Weibull analysis was to be used to characterize the nature of the shear bond strength by fitting the statistical distribution to life data from each of 12 groups and to estimate the probability of failure [35]. Originally, Weibull distribution was designed to compute failure probabilities as a function of applied stress, and

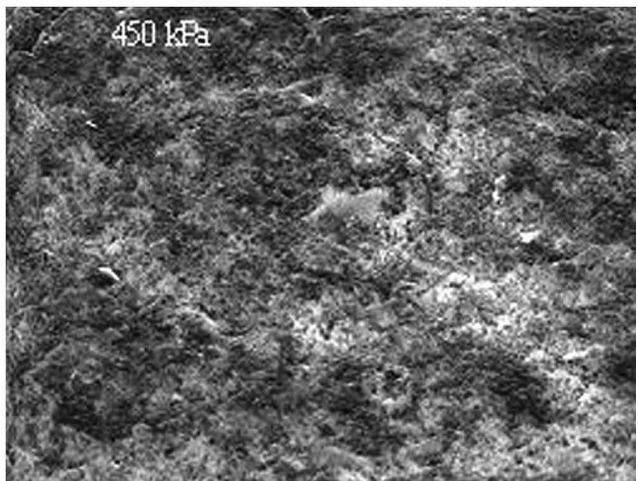
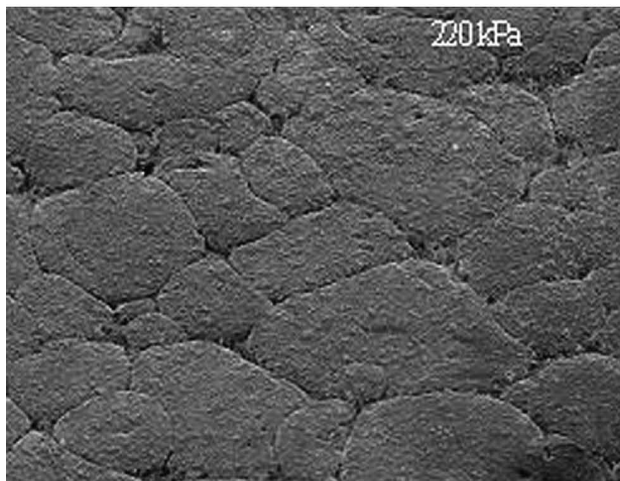
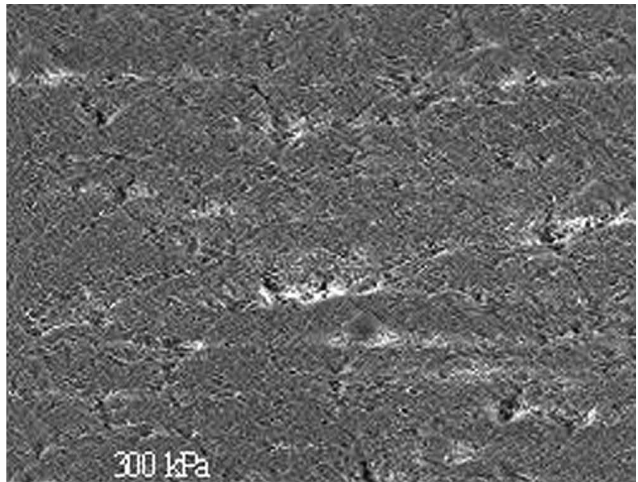
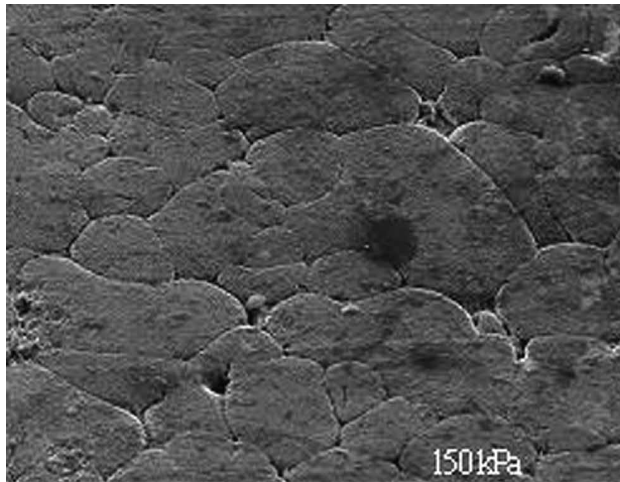


Figure 5. The surface of Procera Alumina abraded with air pressure of 150, 220, 300 and 450 kPa (1000 $\times$).

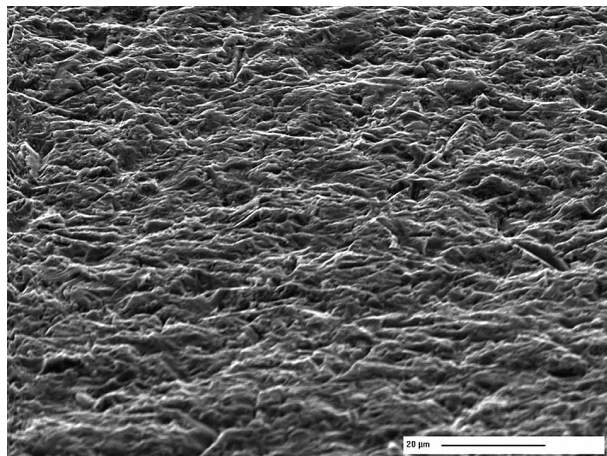


Figure 7. The surface of Procera Zirconia abraded with air pressure of 450 kPa (1000 $\times$, tilted). The horizontal bar denotes 20 μ m.

Weibull analysis data have been published dealing with the mechanical or adhesive properties [36–38] of ceramic materials. However, to our knowledge, no data have been published dealing with the effect of operating air pressure on shear bond strength. Again, the hardness of alumina compared with zirconia may have had an influence on the entire conditioning process as well. Thus, careful attention is needed when selecting statistical methods. Based on the present study, and within its limitations, the following conclusions might be drawn:

1. Silica content on an alumina ceramic surface increases as the operating air pressure increases, whereas results on zirconia surfaces are contradictory.
2. When increasing the air pressure of airborne particle silica-coating, bond strength of the resin composite to zirconia and alumina ceramics increases.
3. Careful consideration needs to be given to the analysis selected for evaluating bond strength values and standard deviations.

Acknowledgments

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