

Effect of surface abrasion and silica coating on tensile bond strength of a resin cement to zirconia ceramics

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Purpose: To evaluate the effects of different surface treatments on the tensile bond strengths of a resin cement to zirconia ceramics.

Materials and Methods: Zirconia discs (Lava) were fabricated and the surfaces of the specimens were ground with #600-grit SiC paper. Then the specimens were divided into four groups according to the surface treatment: (1) no pretreatment (control); (2) air-abrasion with 110 μm Al_2O_3 ; (3) laboratory tribochemical silica coating (Rocatec) with 110 μm Al_2O_3 and 30 μm silica modified Al_2O_3 ; (4) coating the surface (INT coating) with silica-based porcelain (Vintage ZR). Each group was subsequently divided into two subgroups assigned to be silanated using a ceramic primer (RelyX Ceramic Primer) or not. The bonding procedure was then performed using a dual-cure resin cement (RelyX ARC). After 24 h storage in water, the specimens were thermocycled (5-55°C, 5,000 cycles). The bonding performance was evaluated by the tensile bond strength test. Statistical analyses were performed at the level of $p=0.05$.

Results: The control groups showed the lowest bond strengths. The silanated groups showed significantly higher bond strengths than the non-silanated groups for each surface condition ($p>0.05$). The highest bond strength (27.2 \pm 6.2 MPa) was provided by the INT coating group with silanization. However, no significant difference was obtained between the INT coating and Rocatec groups after silanization ($p>0.05$).

Conclusion: With silanization, the INT and tribochemical silica coatings were the most effective in improving the bond strengths of the resin cement to zirconia ceramics. (Int Chin J Dent 2009; 9: 23-30.)

Key Words: INT coating, resin cement, silica coating, tensile bond strength, zirconia.

Introduction

Since zirconia is tough, has high strength, is a metal-free material, and its color is sufficiently white, it is used today in many dental ceramic systems,¹ and also in biomedical applications.² Due to their high resistance to fracture, full coverage zirconium oxide-based crowns can be cemented conventionally, as recommended by some manufacturers.³ However, the cementation technique is important for the clinical success of a restoration.⁴⁻⁶

Bonding between the tooth substrate and the restoration is advocated for improving the retention, marginal adaptation and inhibition of secondary caries.⁴⁻⁷ Acid etching and silanization are not expected to improve the adhesion of resin cement to high-strength ceramics, such as alumina and zirconia-based materials, because they have little or no silica content.⁸⁻¹³

For zirconia ceramics, airborne-particle abrasion is an alternative method for roughening the ceramic surface.^{12,14,15} A tribochemical silica coating, such as Rocatec system and Cojet system, is another surface abrasion technique, which improves bonding to alumina and zirconia surfaces.^{8,10,16} With the tribochemical silica coating, the surface of the restoration is air-abraded with the silica-coated alumina particles.^{8,10} The blasting pressure results in silica particles becoming embedded in the ceramic surface, rendering the silica-modified surface chemically more reactive to resin through a silane coupling agent. However, this

technique cannot coat and modify the entire surface of a zirconia restoration.^{17,18}

The internal (INT) coating technique has been introduced as a new coating method in a laboratory work.¹⁹ With the INT coating, the internal surface of the zirconia restoration can be fully or partially coated by fusing silica-based ceramics. The goal of the INT coating is to improve the bond strength of resin cement to zirconia ceramics. Additionally, this technique may optimize the marginal fitness of the restorations.

Therefore, the aim of this study was to evaluate the effects of different surface abrasion and silica coating techniques on the tensile bond strengths of a resin cement to zirconia ceramics.

Materials and Methods

Tensile bond strength

The materials used in this study are listed in Table 1. Seventy yttria-stabilized tetragonal zirconia discs (Lava, 3M ESPE, St. Paul, MN, USA) were fabricated according to the manufacturer's instructions. The specimens had a diameter of 15 mm and a thickness of 2 mm. The surfaces of the specimens were ground with #600-grit SiC paper under running water and cleaned ultrasonically in distilled water.

Table 1. Materials used in this study.

Material	Brand name	Manufacturer	Batch no.	Composition
Zirconia	Lava	3M ESPE, St. Paul, MN, USA	282361	(ZrO ₂ , HfO ₂)<95%, Y ₂ O ₃ <5%, Al ₂ O ₃ <0.25%
Resin cement	RelyX ARC	3M ESPE	FRGM12-08	Bis-GMA, TEGDMA, Functional DMA, Silica filler, Zirconia filler, Peroxide, Initiator, Amine, Pigment
Silane coupling agent	RelyX Ceramic Primer	3M ESPE	6XR	A silane, Ethanol, Water
Air-abrasion	Rocatec-Pre	3M ESPE, Seefeld, Germany	246433	Al ₂ O ₃ particles (110 μm)
	Rocatec-Soft		257956	Silica-coated aluminum trioxide particles (30 μm)
INT coating	Vintage ZR (Shade B4B)	Shofu, Kyoto, Japan	070704	SiO ₂ , Al ₂ O ₃ , K ₂ O, Na ₂ O, CaO, B ₂ O ₃ , Pigment, Fluorescence

Bis-GMA, Bisphenol-A-diglycidylmethacrylate; TEGDMA, Triethylene glycol dimethacrylate; DMA, Dimethacrylate

They were then randomly assigned to one of the following four groups: (1) no pretreatment (negative control); (2) air-abrasion with Al₂O₃ only; (3) laboratory tribochemical silica coating (Rocatec system, 3M ESPE, Seefeld, Germany); (4) INT coating. The details of each surface treatment are described in Fig. 1. For the Al₂O₃ sandblasting group, the surfaces of the specimens were air-abraded with 110 μm Al₂O₃ (Rocatec-Pre, 3M ESPE) at 0.28 MPa for 15 s. For the Rocatec group, the surfaces were firstly air-abraded with 110 μm Al₂O₃ (Rocatec-Pre) in the same manner as described for the Al₂O₃ sandblasting group. The surfaces were then air-abraded with 30 μm silica-modified Al₂O₃ (Rocatec-Soft, 3M ESPE) for 13 s at 0.28 MPa. Air-

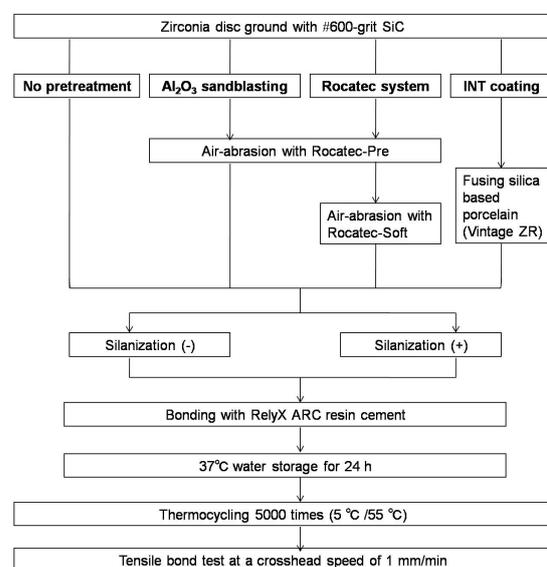


Fig. 1. Flowchart of sample preparation for tensile bond strength test.

abrasion with Rocatec Sand (Rocatec-Pre and Rocatec-Soft) was performed using a laboratory type of air abrasion device (Rocatec Junior, 3M ESPE). During blasting, the tip of the nozzle was rotated perpendicularly to the zirconia surface at a distance of 10 mm from the surface.

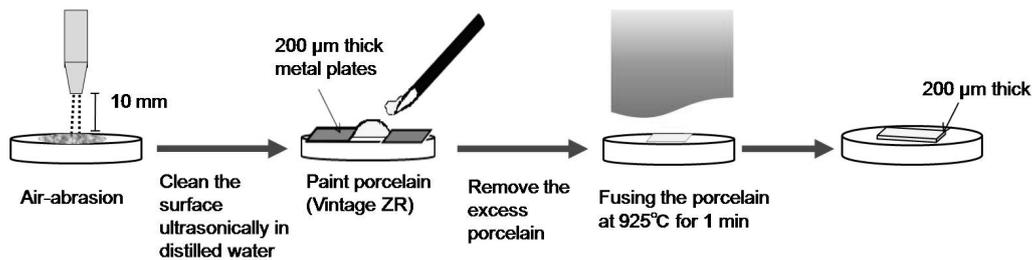


Fig. 2. Illustration of procedure for the INT coating.

An illustration of the procedure for the INT coating group is shown in Fig. 2. The surfaces of specimens were air-abraded with $70\ \mu\text{m}\ \text{Al}_2\text{O}_3$ (Hi Aluminas, Shofu, Kyoto, Japan) at 0.5 MPa for 5 s and then coated with a medium fusing porcelain (Vintage ZR, shade B4B, Shofu), which is a ceramic veneering material designed exclusively for use with the zirconia framework. The porcelain powder was stirred in an excessive amount of water and immediately painted on the ceramic surfaces and fired at 925°C for 1 min with a vacuum to make a coating with a thickness of $200\ \mu\text{m}$. Following this, the specimens were cleaned ultrasonically in distilled water for 10 min and air-dried, except the Rocatec group, which was just air-blown strongly according to the manufacturer's recommendations. Then they were further divided into two subgroups according to whether they would be silanized or not. For the silanization groups, two coats of a silane coupling agent (Ceramic Primer, 3M ESPE) was applied with a fine brush onto the specimen surface, and gently air-blown according to the manufacturer's instructions. A piece of polyethylene tape with a circular hole of 4.0 mm in diameter was positioned on the specimen surface to demarcate the area of bonding.

A dual-cured resin cement (RelyX ARC, 3M ESPE) was then applied to the treated surface with a stainless steel rod as a handle for the subsequent tensile bond testing. After the specimens were kept in water and left at room temperature for 40 min without light curing, they were stored in water at 37°C for 24 h. Following this, they were subjected to thermal cycling of 5,000 times in water ($5\text{-}55^\circ\text{C}$, dwell time of 30 s each). The tensile bond strengths were then measured using a universal testing machine (Autograph AGS-J, Shimadzu, Kyoto, Japan) at a crosshead speed of 1 mm/min.

Failure mode analysis

After tensile bond testing, the failure mode of each specimen was examined under a light microscope (Olympus OCS 912042, Olympus, Tokyo, Japan) at $\times 40$ magnification. The failure modes were classified into the following five categories: a - adhesive failure between zirconia and resin cement; b - mixed failure, including adhesive failure between zirconia and resin cement and cohesive failure in resin cement; c - cohesive failure in resin cement; d - adhesive failure between porcelain and resin cement; e - mixed failure, including adhesive failure between porcelain and resin cement, cohesive failure in resin cement and cohesive failure in porcelain.

Scanning electron microscopic (SEM) observation

The treated surfaces of each group and typical fracture surfaces after tensile testing were examined using a SEM (JSM-5310LV, JOEL, Tokyo, Japan) after the specimens were desiccated and gold-sputter coated.

Statistical analysis

Number of the specimens was seven for each group. The data for the tensile bond strengths were analyzed

using two-way ANOVA except for the no pretreatment groups. The two factors analyzed were surface treatment and silanization. Bonferroni and student's t-test were used as post-hoc test for multiple comparisons. The failure mode data were analyzed using Fisher's exact test (Extended). P values less than 0.05 were considered to be statistically significant for the above-mentioned tests.

Results

Tensile bond strengths

The results of the tensile bond strengths of resin cement to zirconia ceramics after 5,000 thermocycles are summarized in Table 2. Since the control specimens (no pretreatment group) without silanization debonded spontaneously during thermal cycling, the bond strength values could not be detected. Therefore the statistical analyses were performed for all groups, except for the no pretreatment groups. Two-way ANOVA revealed that the tensile bond strengths were influenced by surface treatment ($F=38.159$, $p<0.001$) and silanization ($F=182.663$, $p<0.001$). There was a significant interaction between surface treatment and silanization ($p<0.001$).

The no pretreatment groups showed the lowest bond strengths among the groups regardless of silanization. Silanization significantly improved the bond strengths for each surface condition ($p<0.05$). With silanization, the Rocatec and INT coating groups showed significantly higher bond strength than the Al_2O_3 sandblasting group ($p<0.05$). The highest bond strength (27.2 ± 6.2 MPa) was obtained in the INT coating group with silanization. However, there were no differences between the INT coating and Rocatec groups when they were silanized ($p>0.05$).

Table 2. Tensile bond strengths of a resin cement to zirconia after 5,000 thermocycles.

Silanization	Surface treatment			
	No pretreatment	Al_2O_3 sandblasting	Rocatec system	INT coating
Without	n. d.	4.1 ± 1.3^a	10.0 ± 2.3^b	1.9 ± 0.5^a
With	2.9 ± 0.9	8.1 ± 2.2^A	24.1 ± 4.7^B	27.2 ± 6.2^B

$n=7$, Mean \pm standard deviation.

Statistical analysis was performed in all groups, except for the no pretreatment groups.

Within the same row, means with the same superscript letter demonstrate no significant differences ($p<0.05$).

Failure mode analysis

The failure mode distribution is summarized in Fig. 3. Fisher's exact test (Extended) revealed a relationship between silanization and failure mode for each surface condition ($p<0.05$). The Al_2O_3 sandblasting, Rocatec and INT coating groups with silanization demonstrated significantly higher percentage of failure modes b, c and e, respectively ($p<0.05$). The no pretreatment groups with/without silanization and the groups without silanization except INT coating mainly showed adhesive failures between zirconia and resin cement, whereas the INT coating groups mainly showed adhesive failure between porcelain and resin cement. Moreover, the failure patterns were significantly different among the silanated groups and also among the non silanated groups ($p<0.05$).

SEM observation

Fig. 4 shows the SEM images of the surface textures after each treatment. Roughened surfaces were observed in the Al_2O_3 sandblasting and Rocatec groups (Figs. 4B and 4C). For the INT coating (Fig. 4D), a smooth surface was observed since the surface was not air-abraded. Fig. 5 shows the SEM images of the fractured surfaces of the zirconia after debonding. Failure mode a was shown in A, B, C, and E. Failure modes b, c, d, and

e were observed in D, F, G, and H, respectively.

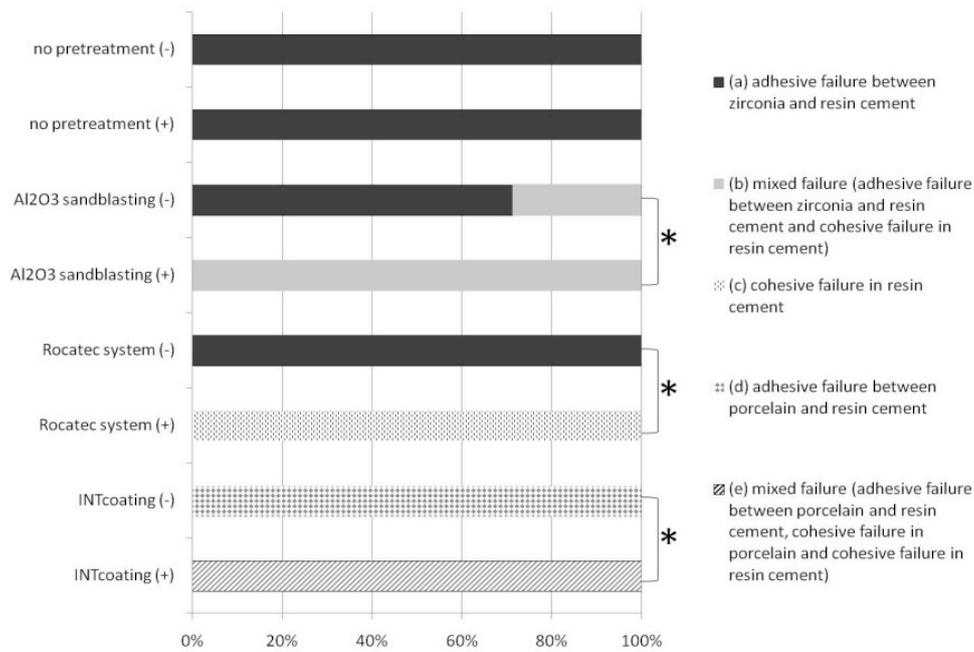


Fig. 3. Failure mode distribution of each group. An asterisk (*) indicates significant difference in failure mode between the groups with (+) and without (-) silanization in each surface condition ($p < 0.05$).

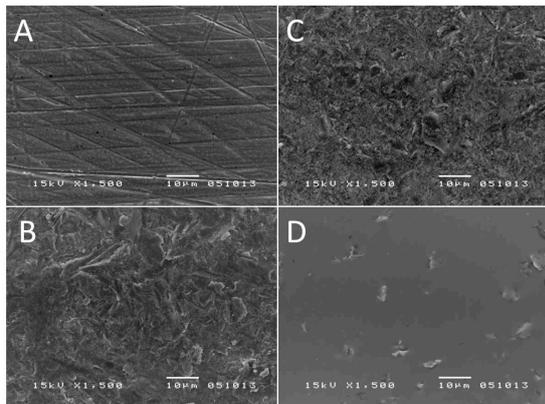


Fig. 4. Upper left SEM images of the surface textures after pretreatment (x1,500, A-D). A, #600-grit SiC paper; B, 110 μm Al_2O_3 sandblasting; C, tribochemical silica coating with Rocatec system; D, INT coating. Roughened surfaces were observed in the Al_2O_3 sandblasting and Rocatec groups (B and C). For the INT coating (D), the smooth surface was observed since the surface was not air-abraded.

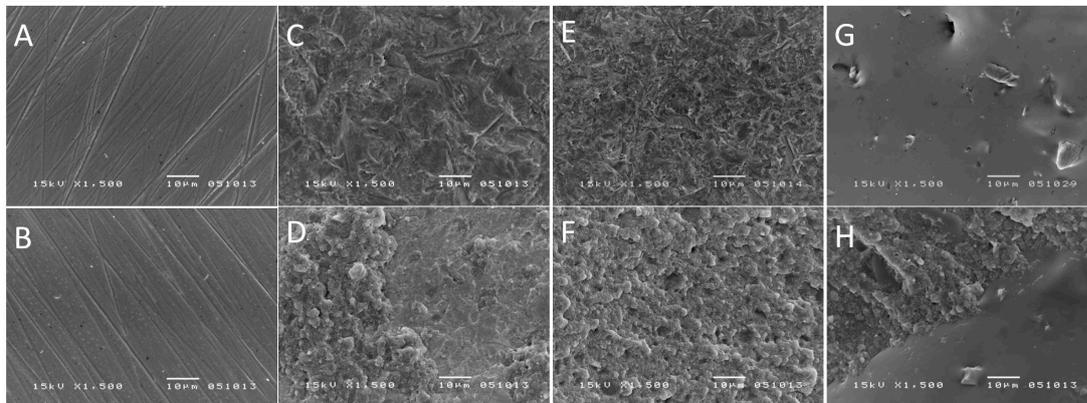


Fig. 5. SEM images of the debonded surface of each group (x1,500, A-H). A, no pretreatment without silanization; B, no pretreatment with silanization; C, 110 μm Al_2O_3 sandblasting without silanization; D, 110 μm Al_2O_3 sandblasting with silanization; E, Rocatec system without silanization; F, Rocatec system with silanization; G, INT coating system without silanization; H, INT coating system with silanization. Failure mode a was shown in A, B, C, and E. Failure modes b, c, d, and e were observed in D, F, G, and H, respectively.

Discussion

Thermocycling is one of the methods most widely used to test the durability of resin bonds and results in the highest clinically relevant stress.^{20,21} It was reported to have a much higher impact on the durability of resin bond strength to zirconia than did water storage at a constant temperature alone.²¹ In the present study, 5,000 thermocycles were performed to represent an aging of the bond of approximately 6 months in vivo.²⁰

Airborne-particle abrasion with alumina particles is the preferred surface treatment method for high-strength ceramic materials, such as alumina and zirconia ceramics, which creates high surface energy and promotes micro-retention.^{8,9,12,15,17,21} Roughening the substrate surface promotes adhesion, since it allows the polymer (resin composite) to flow into the surface and forms irregularities on the substrate surface.²² The present study also demonstrated that the Al₂O₃ sandblasting groups showed higher bond strength than the no pretreatment groups regardless of silanization.

For the tribochemical silica coating (Rocatec) groups, the surface of the specimens was air-abraded with 110 µm Al₂O₃ followed by air-abrasion with 30 µm silica modified Al₂O₃ (Rocatec-Soft), according to the Rocatec protocol by 3M ESPE. The improved bond strengths found in the present study with the Rocatec groups compared with the Al₂O₃ sandblasting groups may be due to an increased surface area resulting from the additional treatment with Rocatec-Soft, irrespective of silanization.²³

Silane molecules react with water to form three hydroxy-silyl groups (-Si-OH) from the corresponding methoxy-silyl groups (-Si-O-CH₃).²⁴⁻²⁸ The silanol groups then react further to form a siloxane (-Si-O-Si-O-) network with the silica deposited on the silica-based ceramics. The methacryloyl groups of the silane molecules react with the methacryloyl groups of adhesive resins in a free radical polymerization process. In summary, a chemical bond is formed via the silica layer on the surface, silane coupling agent, and resin cement. The pretreatment with RelyX Ceramic Primer significantly increased the bond strength for each surface condition. Silane coupling primers have been reported to increase the wettability of the surface, which could explain the improved bond strengths in the control and alumina-abrasion groups.¹¹ However, no chemical reaction would be expected for these groups, thus the bond strengths were very low and likely to decrease more when subjected to severe aging conditions. For the Rocatec group, silanization significantly improved the bond strength. During tribochemical silica coating, alumina particles modified with silica are blasted, producing high heat, which, together with pressure, cause the silica-modified alumina particles to be embedded within the ceramic surface. This allows for an effective reaction of silane agents with zirconia ceramics.¹⁷

Regarding the INT coating group, the bond strength without silanization was very low (1.9 MPa) due to the absence of a chemical bond between the resin cement and the porcelain coated on the surface of the zirconia. On the other hand, silanization drastically improved the bond strength in the INT coating group, as was the case with the Rocatec group, because a chemical bond was created via the silane coupling agent between the resin cement and the silica-based porcelain coated on the surface of the zirconia. We expected that the INT coating is more effective than the tribochemical silica coating in terms of coating the surface with silica since the surface of zirconia can be fully coated with silica-based ceramics in the INT coating, suggesting that a greater amount of silica is available for silanization in the INT coating group. However, there was no significant difference in the bond strengths between the Rocatec and the INT coating groups with silanization after 5,000 cycles of thermal cycling. Since the size and the amount of silica as well as the surface roughness is different between the INT coating and the Rocatec groups, the bonding durability needs to be further evaluated in severe aging conditions.

In the INT coating group, there were no adhesive failures at the interface between zirconia and porcelain, which indicates that the bonding of porcelain to zirconia exceeded that of resin cement to porcelain. Although the INT coating and the Rocatec groups showed high bond strengths with silanization, they differed in failure modes. With silanization, the Rocatec group showed cohesive failure in resin cement, whereas the INT coating group showed mixed failures including cohesive failure in cement/porcelain and adhesive failure between the cement and the porcelain coated on the surface of zirconia. The bond strength of the INT coating could be enhanced by improving the mechanical properties of the porcelain for coating. For the INT coating, the surface of zirconia was coated with a 200 μm thickness of porcelain to standardize the thickness by a dental technician in the present study. Beuer et al.²⁹ reported that three CAD/CAM systems of zirconia fabrication showed marginal gaps below 120 μm which were considered clinically acceptable. On the other hand, Reisch et al.³⁰ reported that the marginal gaps and internal fitness of zirconia fabricated FPDs varied between 8 μm and 272 μm , and between 39 μm and 502 μm , respectively. In clinical situations, the coating should be thin when the gap is small. On the other hand, after sintering or adjustment with burs, a zirconia frame with a large marginal or internal gap can be repaired fully or partially with a silica-based ceramic using the INT coating in the laboratory.

It must be considered that *in vitro* testing is simpler than the *in vivo* situation. In the present study, thermal cycling was used as means of aging, however, teeth in the oral environment are continuously subjected to different types of stresses that may impair the bonding effectiveness of the cement. Therefore, further studies should be carried out to evaluate the long-term durability of the resin bond strength to zirconia ceramics. In addition, the application of the INT coating technique in the laboratory should be improved. Moreover, clinical evaluations of zirconia restorations are also required to establish reliable application methods.

Acknowledgments

This work was supported by the grant from the Japanese Ministry of Education, Global Center of Excellence (GCOE) Program; International Research Center for Molecular Science in Tooth and Bone Diseases. The authors gratefully thank Mr. Norio Ishimatsu at TENSEI systems Co., Ltd for assisting in the technical procedures.

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Accepted June 9, 2009.

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