The effect of disk type and cutting speed on the micro-tensile bond strength of ceramic specimens to resin cement

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The bond strength of dental materials has been evaluated by tensile testing of micro-specimens. The cutting process used to obtain specimens may influence the results. The objective of this study was to investigate the influence of different types of diamond disks and cutting speeds on the bond strength of ceramic specimens and on specimen integrity. Lithium disilicate-based ceramic cubes were bonded with resin cement to composite resin cubes, according to the manufacturers’ instructions. The ceramic/cement/resin blocks thus obtained were divided into two groups to be cut with Buehler® or Extec® disks and then sectioned at cutting speeds of 200 rpm and 400 rpm. The results showed that the bond strength values were affected by the cutting speed and disk/speed interaction (p<0.05). SEM analysis revealed better specimen properties when the blocks were cut at 200 rpm. It was concluded that ceramic specimens must be cut at low speeds.

Keywords: Materials testing, Ceramics, Micro-tensile bond strength, Scanning electron microscopy

INTRODUCTION

In the long-standing endeavor to improve adhesive systems, numerous laboratory studies have been conducted to evaluate the clinical behavior of dental materials in various oral environments. Even though laboratory assays cannot faithfully reproduce clinical conditions, they have become increasingly sophisticated. Current advanced technologies capable of disclosing material failure modes, as well as other mechanical and biological-related issues, have provided the potential to effectively improve bonding to dental tissues.

The variation in reported bond strength values of dentin-bonding agents has been attributed to differences in measurement techniques, dentin surface preparation, and the age of the dentin itself¹-⁴. Exposing smaller areas of the bonded specimens to tensile force was proposed by Sano et al. This modification is known as micro-tensile testing. The tensile bond strength values thus obtained were inversely related to the bonded surface area of the specimens. In these small surface areas, cohesive failure could be prevented and all bond failures were predominantly adhesive in nature. In addition, several measurements could be made on a single tooth⁵.

The micro-tensile test is quite versatile, with the following advantages: specimen fracture occurs predominantly at the adhesive interface and the ability to test smaller areas that can be analyzed using scanning electron microscopy (SEM). Furthermore, the values and means can be calculated for a single tooth and for a smaller number of specimens, and the ability to measure regional differences in the bond strengths of resin/dentin and resin/enamel⁶. This laboratory assay is also a valuable technique for studying the influence of water on the durability of the adhesive interface, because the small dimensions of the micro-specimens require less time for water infiltration than do larger samples, thus accelerating the process⁷-⁹.

However, the micro-tensile specimen preparation technique is laborious. It has been stated that force application must be controlled to prevent premature micro-specimen fracture. The blocks that are made into the micro-specimens must be cut with a concentric diamond disk to prevent damage to the adhesive interface, and cooling is also needed to prevent drying and/or overheating⁸. An adhesive area of 1.0 mm² has been recommended owing to the difficulties involved in preparing smaller bars. Further decreases in bond area below this limit have led to a disproportionate increase in the number of specimen fractures⁹-¹³.

In recent years, micro-tensile strength testing has been increasingly used as a method to assess the true bond strength of an adhesive system to different dental substrates, ceramics, metals or polymers. It allows the appropriate alignment of samples, a more homogenous distribution of force, and has ultimately proven to be a reliable method to compare bond strengths of different commercial brands of adhesive systems¹⁴,¹⁵. The cutting procedure used to prepare micro-specimens for tensile testing is critical and technique-sensitive. The intrinsic fragility of substrates and materials may adversely affect the results¹⁶.

On the other hand, the disadvantages of micro-tensile testing include longer laboratory phase, a meticulous technique and special equipment, specimen sensitivity to manipulation and proneness to quickly dehydrate, and difficulty to measure bond strength, owing to specimen fragility¹⁶,¹⁷. This difficulty is observed especially when the materials submitted to
tension have a high hardness index, a high elasticity modulus, and a low tenacity to fracture (e.g., ceramics). These characteristics lead to quicker propagation of cracks and defects at the adhesive interface[14,15,18].

The disks normally used to cut micro-specimens from larger pieces are impregnated with diamond particles[19], and the friction between each abrasive particle and the surface of the piece contributes to elevating the temperature at the disk/surface interface. This effect may lead to the burning of the specimen, thereby causing microstructural and dimensional changes related to the material’s thermal expansion coefficient, and to the occurrence of cracks[20].

The influence of cutting speed on the bond strength of human molar dentin to resin was studied by Reis et al. Speeds of 100 rpm, 300 rpm and 500 rpm were used to cut micro-specimens with approximately 0.8 mm² of bond area. It was found that the cutting speed was a significant factor for bond strength values, although not all groups showed the same trend, thus preventing a more conclusive result[16]. Sadek et al. also assessed composite resin specimens bonded to the dentin and enamel of human third molars that were cut using various speeds of 100 rpm, 200 rpm and 400 rpm. It was observed that the bond strength values were significantly smaller for the enamel micro-specimens at 400 rpm, with the presence of numerous cracks next to the adhesive interface. This finding did not occur with the dentin specimens[18]. Hence, cutting fragile structures like enamel can interfere with the integrity of micro-specimens and with the results of bond strength testing[13,21].

The aim of this study was to evaluate the effect of disk type and cutting speed on the micro-tensile bond strength of bonded ceramic to composite resin using resin cement. The null hypothesis tested was that different disks and cutting speeds do not affect the bond strength of ceramic to resin cement or specimen integrity.

**MATERIALS AND METHODS**

Twenty blocks were made by bonding ceramic cubes to resin cubes with a dual-cure resin cement. These ceramic/cement/resin blocks were randomly separated into four groups of five blocks. Then, each group was named according to the different study conditions (disk type and cutting speed), as follows: E1, Extec disk (Extec Corp., Enfield, CT, USA) at 200 rpm; E2, Extec disk at 400 rpm; B1, Buehler disk (Buehler Ltd., Lake Bluff, IL, USA) at 200 rpm; and B2, Buehler disk at 400 rpm. Both disk types had the same size and were saturated with a low concentration of diamond particles.

**Preparation of the ceramic and resin cubes**

Initially, the wax patterns used to prepare the ceramic cubes were obtained by means of a square-shaped acetate Teflon matrix, measuring 6x6x5 mm (Fig. 1). The procedural steps of embedding in investment material, wax burnout, pressing of the ceramic, devesteing and cleansing, were performed following the manufacturer’s instructions (Ivoclar Vivadent AG, Schaan, Liechtenstein). Next, the upper aspect of the ceramic cubes was finished with 220-, 400- and 600-grit silicon carbide-impregnated sandpaper to produce a uniform surface for cementation. After this procedure, the cubes were washed and kept in an ultrasonic cleaner for 10 min in distilled water, air-dried and stored for cementation.

For preparation of the resin cubes, Z100 composite resin (3M ESPE, São Paulo, SP, Brazil) was inserted in the same matrix using the incremental technique. Each 2-mm portion was polymerized for 40 s using a light-curing unit (Demetron Optilux 400™; Kerr Corporation, Orange, CA, USA). The resin cubes were then removed from the matrix and the polymerization was completed with the same light-curing unit for an additional 40 s on all aspects.

**Applying the cement**

The previously prepared ceramic cube surfaces were submitted to treatment with 10% hydrofluoric acid for 20 s (without rubbing), rinsed with water jets for 60 s and dried with an air syringe. A primer (Rely X Ceramic Primer; 3M ESPE, São Paulo, SP, Brazil) and a thin layer of adhesive (Single Bond; 3M ESPE, São Paulo, SP, Brazil) were applied, followed by application of a layer of resin cement (Rely X ARC; 3M ESPE, São Paulo, SP, Brazil) on the etched ceramic. All procedures were carried out according to the manufacturer’s instructions. The resin cubes were then placed over the ceramic cubes. The blocks thus produced were kept under a pressure of 10 N for 10 s (ISO/TS 11405:2003 [E]; Fig. 2). Excess cement was removed with a disposable micro-brush and the ceramic/cement/resin blocks were immediately light-cured for 40 s. After five minutes, each block was immersed in distilled water and kept at 37±2°C (ISO 11405:2003 [E]) for 24 h, until the cutting procedure was initiated.

**Producing the bar specimens**

Each ceramic/cement/resin block was fixed to a 3-mm acrylic cylinder with sticky wax (Asfer; Indústria Química Ltda., São Paulo, SP, Brazil) for the cutting procedure. A layer of wax was placed around most of the block in order to stabilize it during cutting and to minimize the incidence of tensions[19,20] (Fig. 3). The acrylic base/block set was fixed to a precision cutter.
The blocks were then sectioned perpendicularly to the bond area to produce slices with an approximate thickness of 1.0 mm under irrigation with a solution prepared with lubricant for metallographic sections (ER 70100; Erios Ltda., São Paulo, SP, Brazil) diluted in water, according to the manufacturer’s instructions. Immediately following, the blocks were rotated 90° and new sequential cuts were performed perpendicular to the former cuts. This was enabled by using an “L”-shaped support fixed to the machine arm (Fig. 3), which allowed a 90° rotation of the set without displacing it. Once the cuts were completed, the bar specimens obtained were washed under running water and stored in distilled water at 37°C. Nine bar specimens were cut from each of the five ceramic/cement/resin blocks, thus producing a total of 45 specimens per group. A certain number of bar specimens broke during the cutting procedure and the losses were randomly distributed within the groups. After concluding the cutting procedure, the bars of each group were randomly separated into three parts, where one part was submitted to fractographic analysis and two parts were submitted to the tensile test (Table 1).

**Tensile bond strength test**

The area of each bar was measured with digital calipers with a resolution of 0.01 mm (Pantec; Instrutemp Ltda, São Paulo, SP, Brazil). The bars were positioned individually in a custom-made testing jig22) with the aid of clinical forceps, and then fixed with a gel form of a cyanoacrylate-based adhesive. Zapit accelerator (Pacer Technology, Rancho Cucamonga, CA, USA) was applied over the adhesive to accelerate hardening and prevent the adhesive from flowing onto the bond interface. After waiting 4 min for the adhesive to harden, the bar/jig set

| Table 1 | Number of bar specimens (n) in each group after the cutting procedure |
|---------|-----------------------------|
|         | Submitted to fractographic analysis | Submitted to the tensile test | Total bars |
| B1      | 13                          | 30                          | 43          |
| B2      | 13                          | 29                          | 42          |
| E1      | 15                          | 29                          | 44          |
| E2      | 09                          | 27                          | 36          |
| Total   | 50                          | 115                         | 165         |
was adapted to an Instron® universal testing machine (Model 5566; Instron Corporation. Norwood, MA, USA). Tensile force was applied using a 50 kgf load cell at a crosshead speed of 1 mm/min until specimen fracture (Fig. 4). The tensile bond strength of each bar in relation to area was calculated according to the following equation:

\[ T = \frac{N}{mm^2} \]

Where:
- \( T \) = tensile strength (in MPa)
- \( N \) = force necessary to fracture the bar (in N)
- \( mm^2 \) = ceramic/cement bond area

**SEM analysis**
The ruptured specimens were analyzed under a scanning electron microscope (Superscan SSX-550; Shimadzu Corporation, Kyoto, Japan), at an approximate magnification of 70×. They were classified as mixed, adhesive and cohesive: mixed, when the fracture occurred between the ceramic and resin cement (cohesive failures of cement and ceramic); adhesive, when it occurred in the interfacial region and in the resin cement (adhesive zone); and cohesive, when it occurred in the composite resin4). The non-fractured bars from each subgroup were qualitatively analyzed for the presence of bubbles, cracks and fissures, at 80×, 150× and 240× magnifications.

**Statistical analysis**
The results of the micro-tensile test were initially submitted to the Golmogorov-Smirnov test at a significance level of 5%. Since the data indicated a normal probability distribution, the ANOVA test was also applied to compare the bond strength values for the bars in each experimental group (<0.05). Since the bar values were not found to be influenced by the ceramic/cement/resin blocks, the analyses were conducted assuming bar independence within each of the four different study groups; hence, the bars were treated as the sample units. Comparisons between the total bond area means, bond strength means, diamond disk and cutting speed influences of each group were performed by descriptive analyses, the Tukey post-hoc test, ANOVA and two-factor ANOVA.

**RESULTS**
First, the mean area of all bars within each group were compared, as shown in Table 2. The Tukey post-hoc test revealed that the mean areas of groups E1, B1 and B2 were homogenous (1.04 mm², 1.12 mm² and 1.08 mm², respectively), but significantly greater than that of Group E2 (0.93 mm², <0.05). Table 3 shows the bond strength means (in MPa) of the study groups, as follows: B1=25.2±9.0, B2=24.2±11.3, E1=28.6±10.4 and E2=20.3±12.4. The bars that did not resist the cutting procedure were assigned a zero value, insofar as the losses occurred irregularly in the different groups, as revealed by the ANOVA test. Although the analysis showed that there were differences among groups, only groups E1 and E2 were found to be significantly different.

![Fig. 4 The bar/jig set after tensile bond strength test.](image)

**Table 2** Minimum, maximum and mean areas of bar specimens in each group (mm²)

|     | n | Minimum | Maximum | Mean | Standard deviation | p-value |
|-----|---|---------|---------|------|--------------------|---------|
| B1  | 30| 0.80    | 1.50    | 1.12 | 0.17               |         |
| B2  | 29| 0.80    | 1.30    | 1.08 | 0.15               | 0.000   |
| E1  | 29| 0.90    | 1.40    | 1.04 | 0.16               |         |
| E2  | 27| 0.60    | 1.20    | 0.93*| 0.13               |         |
| Total|115| 0.60    | 1.50    | 1.05 | 0.17               | —       |

*Significantly different by the Tukey post-hoc test (<0.05)
Table 3  Minimum, maximum, and mean bond strength (MPa) values and standard deviation (SD) for bar specimens (n) from each experimental group (p<0.05)

| Group | n  | Minimum | Maximum | Mean   | Standard deviation | p-value |
|-------|----|---------|---------|--------|--------------------|---------|
| B1    | 30 | 0.9     | 45.8    | 25.2*  | 9.0                | 0.036   |
| B2    | 30 | 0.0     | 43.5    | 24.2   | 11.3               |         |
| E1    | 30 | 0.0     | 52.3    | 28.6   | 10.4               |         |
| E2    | 30 | 0.0     | 51.9    | 20.3*  | 12.4               |         |
| Total | 120| 0.0     | 52.3    | 24.6   | 11.1               | —       |

*Significantly different by the Tukey post-hoc test (p<0.05)

Table 4  Influence of disk type and cutting speed factors

| Source of variation            | p-value |
|--------------------------------|---------|
| Area                           | 0.017*  |
| Disk                           | 0.729   |
| Speed                          | 0.012*  |
| Disk/speed interaction         | 0.034*  |

*Significant by two-way ANOVA (p<0.05)

Table 5  Fractographic analysis of the fractured surfaces

| Study groups | Ceramic/Cement (mixed) | Resin/Cement (adhesive) | Composite (cohesive) | Total bars |
|--------------|------------------------|-------------------------|----------------------|------------|
| B1           | 12                     | 18                      | 0                    | 30         |
| B2           | 15                     | 12                      | 2                    | 29         |
| E1           | 06                     | 20                      | 3                    | 29         |
| E2           | 17                     | 09                      | 1                    | 27         |

as revealed by the Tukey test. As can be seen in Table 4, the two-way analysis of variance showed that only the disk factor did not significantly influence the tensile bond strength (p=0.72). In other words, the bar area, speed and disk/speed interaction factors influenced the bond strength values obtained (p<0.05). The structure of the bars from the different groups was compared using SEM analysis. The mode of failure was determined using fractographic principles, and classified as shown in Table 5. It was observed that the number of fractures in the adhesive region was greatest when a 200 rpm speed was used to cut the specimens.

A qualitative SEM evaluation of the non-fractured bars set aside right after the cutting procedure (Figs. 5–8) revealed the presence of cracks beneath the ceramic/cement junction and on the edges of the bars. The presence of bubbles on the resin cement layer was also observed in all groups. However, in the specimens submitted to a speed of 400 rpm, there was a trend towards a greater number and length of fissures and, in

Fig. 5  Bar cut with a Buehler disk at 200 rpm. Note small cracks and external edges with slight imperfections (80x).
Fig. 6  Bar cut with a Buehler disk at 400 rpm. Note wrenched ceramic at the external edges near the cement/ceramic junction, and the presence of cracks (80×).

Fig. 7  Bar cut with an Extec disk at 200 rpm. Note external edges with small imperfections (80×).

Fig. 8  Bar cut with an Extec disk at 400 rpm. Note wrenched ceramic at the ceramic/cement junction and on the external edges (80×).

Fig. 9  Backscatter mode photomicrograph of bar cut with an Extec disk at 400 rpm (80× and 240×).

DISCUSSION

The micro-tensile test is usually regarded as the standard test to assess bond strength between several materials and dental substrates. However, it is very difficult to compare the bond strength results of micro-tensile tests, because, to date, no methodological guidelines have been established. Hence, caution must be taken when confronting the conclusions of different research centers, because the methodological choices made by different authors, such as the substrate or materials, the bond area size and the micro-specimen form employed may not be comparable.

Various shapes of micro-specimens have been used to test the adhesion of dental materials to different substrates. The cutting technique is a very sensitive process, and it induces additional stress. The bar specimen form of 1.0 mm² has yielded higher values than circular and hourglass-shaped specimens in tensile tests. An inverse exponential relationship between tensile bond strength and bonded cross-sectional area has been reported. This has been explained in the light of Griffith’s Law, which deals with stress distribution in solids, where the tensile strength of a fragile material decreases with the increase in transversal bond area, and where imperfections act as points of tension concentration. Thus, the cross-sectional bond area size of choice for adequate testing should not be higher than 1.5 mm², nor lower than 0.5 mm², because larger bond areas result in a greater number of defects and in group E2 also showed the greatest number of losses during the cutting process (Table 1).
smaller bond strength values\(^4,10,20\). Hence, a reduction in the number and size of defects in the adhesive zone is thought to decrease bulk cohesive failures and increase the tensile bond strengths observed during tensile testing regardless of the specimen cross-sectional shape\(^4,9\).

However, the cutting process used to obtain specimens with smaller areas has also led to extensive losses. Premature losses during the cutting of 1.0 mm\(^2\) bar specimens in enamel were reported to be three to four times greater than in dentin\(^13\), and these losses could reach 20% of the specimens when bonding ceramic to resin with resin cement\(^2\). This figure could be even greater considering that some studies have omitted data on losses altogether. In the present study, the greatest number of losses occurred when the Extec disk was employed at 400 rpm. Considering the limitations of this research, one possible explanation is that the Extec disk was submitted to eccentric motion at 400 rpm, probably because it is made of a very flexible metal, thus compromising specimen stability during cutting. This observation was also made by Reis et al., who reported vibratory motion of the disk during cutting, although they did not identify at which speed or speeds such motion occurred\(^10\). In the present study, the eccentric motion observed with the Extec disk at 400 rpm may have also caused a significant decrease in bar mean area compared to the other groups. This hypothesis, however, should be further investigated.

Analyzing the influence of both factors — disk and cutting speed — in the present study, we found that the area of the bars was a source of variation influencing the tensile bond strength results. Table 4 shows that the speed and the disk-speed interaction factors influenced the bond strength values \((p<0.05)\). The null hypothesis tested in this study was rejected, namely, that no significant differences in specimen integrity would be observed when obtaining the specimens by cutting lithium disilicate-based ceramic bonded to resin cement at different cutting speeds. These results could be due to the fragility of this material. The specimens submitted to tensile testing are very sensitive to edge or surface machining damage, and crack propagation is determined by varying levels of stress intensity\(^18,20\).

Upon assessing the photomicrographs of the bars taken with the scanning electron microscope, it was observed that the fissures and cracks produced at a speed of 200 rpm were smaller and distributed mostly along the entire ceramic/cement bond area. The outside edges showed only small fractures at this speed. In contrast, at the speed of 400 rpm, there was a greater trend toward the occurrence of larger fissures and greater structural loss at the edges. These imperfections led to higher stress points on the ceramic during the micro-tensile test, and were more evident for the Extec disk (Figs. 7, 8). Fractographic analyses have shown a relationship between fracture strength and elastic modulus\(^18,21\). The effect of a failure induced on the edges of specimens is “fatal,” whereas a fissure or fracture in the middle of a specimen could be better tolerated. Furthermore, the greater the length of the defect, the greater the fracture tension, particularly when the direction of the defect is perpendicular to the direction of the tensile stresses\(^15,18\).

The influence of the cutting process on composite-to-enamel bond strength has been reported in the literature. Enamel has been shown to present cracks, structural loss on its edges, and a significantly lower bond strength compared to dentin when specimens are cut at high speeds\(^12\). In the present study, a significant difference in the bond strength between ceramic and a dual-cure resin cement was observed. By comparing enamel and ceramic regarding their physical properties of hardness (4.1 GPa and 5.5 GPa, respectively for enamel and ceramic) and elastic modulus (87±5 GPa and 95±5 GPa, respectively), it could be stated that the cutting process should be conducted at lower cutting speeds, when performed in materials with high hardness and elastic modulus\(^15,18\).

In the present study, the standard deviation of the bond strength results was very high; however, significant differences were observed for the Extec disk at speeds of 200 rpm versus 400 rpm, and it seems that bar specimens have a higher standard deviation compared to hourglass specimens\(^21\).

In the present study, the choice of disk brand to perform the ceramic cutting procedure had no influence on the bond strength values. This research was planned with the objective of collecting data that could be useful for standardizing specimen preparation procedures. Procedure standardization should be sought to ensure that the methodology applied is reliable, and to allow comparison of the results obtained by different research laboratories. This investigation revealed that the cutting speed factor had a significant influence on the bond strength and integrity of ceramic micro-specimens, probably owing to specimen incapacity to absorb stress during the cutting procedure. The cutting of specimens should therefore be conducted in an accurate fashion, and should respect the mechanical properties of the dental materials involved.

Conducting a fractographic analysis should be considered in conjunction with micro-tensile testing, insofar as it allows fractures to be classified and also serves the purpose of critically evaluating the effectiveness of the method employed and the conclusions drawn\(^1,18,20\).

CONCLUSIONS

Considering the limitations of this study, it could be concluded that the metallographic cutter should be used at low speeds during ceramic specimen cutting procedures, in order to avoid producing cracks and interfering with the micro-tensile bond strength values obtained.

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