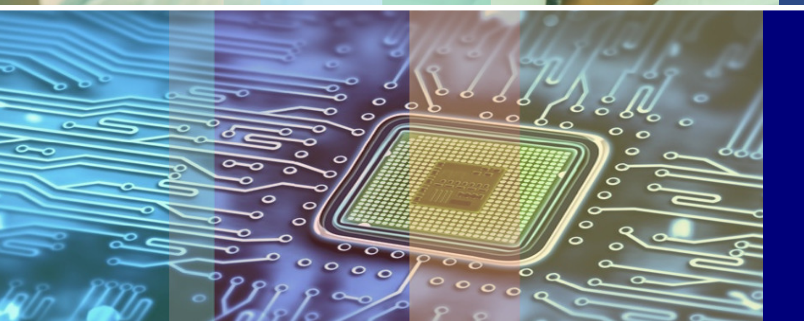
Tokuyama Bond Force II

Tokuyama Dental **TECHNICAL REPORT**



TOKUYAMA BOND FORCE - TECHNICAL REPORT

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1 Introduction

Tokuyama Dental Corp. launched a 7th generation one-bottle one-step bonding agent, Tokuyama Bond Force, in February 2007. Tokuyama Bond Force contains proprietary 3D-SR monomer technology with several functional groups per molecule that interact with tooth calcium.[1, 2] It is a revolutionary all-in-one adhesive that achieves adhesion to tooth substance comparable to two-step bonding agents.[3, 4, 5, 6]

Although many companies have launched various one-bottle one-step bonding agents in recent years, the direction of development has shifted towards more simplicity, such as storage at ambient temperature.

Given these circumstances, we aimed to improve storage stability and ease of handling while preserving adhesion properties of Tokuyama Bond Force. Tokuyama Bond Force II was launched in Japan on January 2014. Features of Tokuyama Bond Force II are listed below.

New Features of Tokuyama Bond Force II:

1) Simple and Quicker Procedure with Reliable Adhesion
2) Ambient temperature storage (0-25°C)
3) Selectable Container (Bottle and Pen)

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2 Adhesion mechanism

2.1 ADHESION MECHANISM

To achieve the enhanced properties outlined above, our patented 3D-SR monomers were improved and the chemistry was optimized. 3D-SR monomers have several functional groups that interact with calcium and polymerizing groups per molecule. They interact with the calcium of the tooth structure at multiple points, causing a strong adhesion to the tooth structural interface, and create a three-dimensional cross-link by reacting with the calcium (*Figure 1*). Cross-linking our adhesive 3D-SR monomers play an important role to intensify the density of three-dimensional cross-linking through copolymerization reactions to form a uniformly thin, very strong and highly adhesive bonding layer.

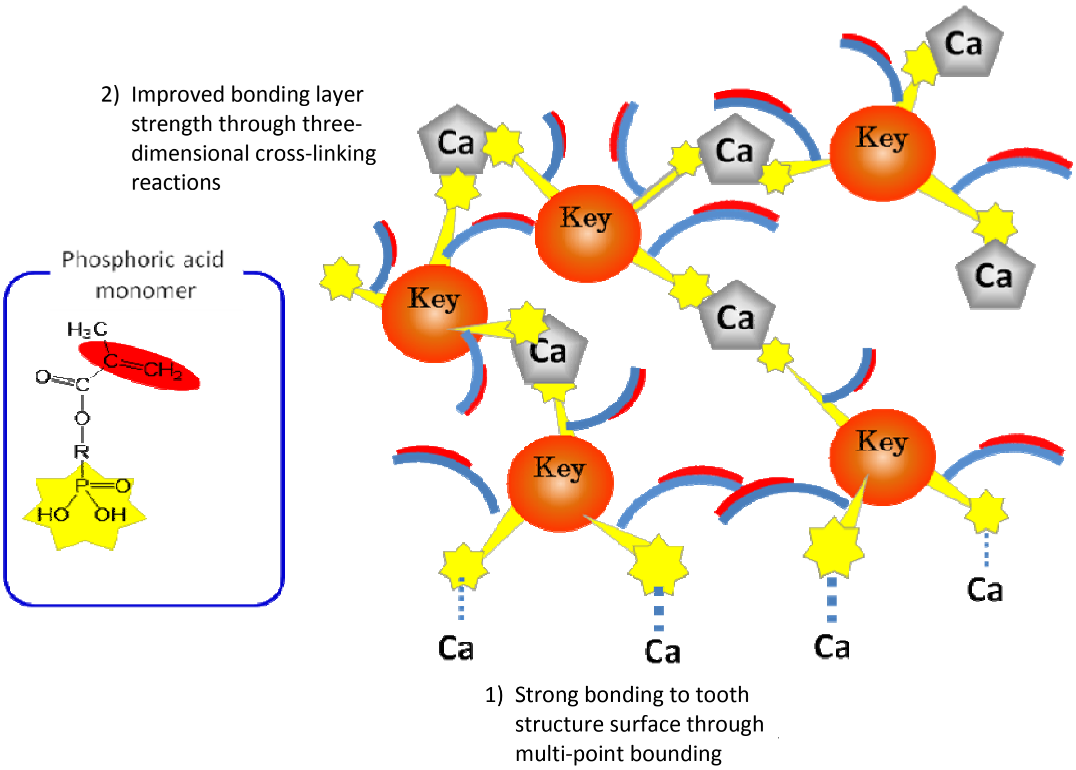


Figure 1 *Three-dimensional cross-linking reactions of adhesive 3D-SR monomers and calcium ions*

In comparison to the previous Bond Force generation, the number of groups interacting with calcium groups and polymerizing groups per molecule of new 3D-SR monomer has been successfully increased. The enhanced chemistry has not only provided a simpler and quicker procedure with storage at ambient temperatures, but has also offered a reliable adhesion. *Figure 2*

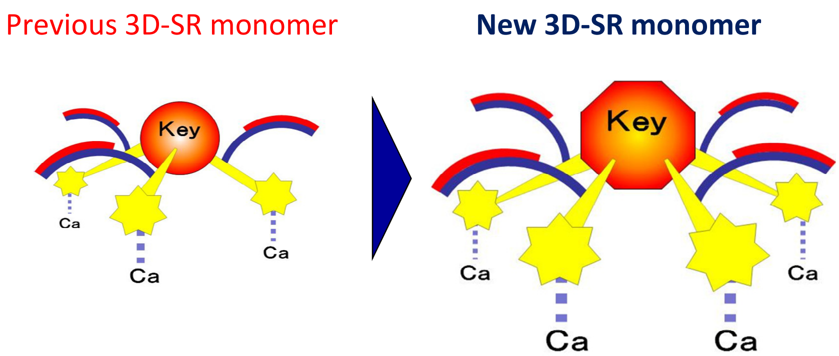


Figure 2 *Structure of new adhesive 3D-SR Monomers*

With the new and improved 3D-SR monomers providing a higher level of interaction with the calcium and enhanced three-dimensional cross-linking reactions, Tokuyama Bond Force II now provides decreased application time from the previous 20 seconds down to 10 seconds. In addition, technique sensitivity has also been reduced by making the bond strength to the tooth structure less dependent on air blowing conditions.

Furthermore, optimization of composition, especially setting bond pH to 2.8, decreases the hydrolysis of the monomers which occurs during storage of Tokuyama Bond Force II. In short, through new adhesive 3D-SR monomers and optimized composition, Tokuyama Bond Force II achieves excellent tooth structure adhesion properties and storage stability, making it a strong bonding agent with excellent handling, that can be stored at ambient temperature.

2.2 COMPOSITION

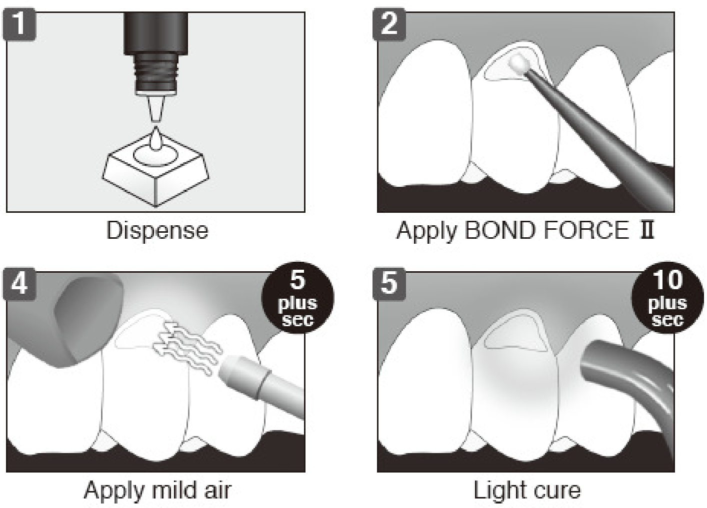
The composition of Tokuyama Bond Force II is as listed in *Table 1*. It includes phosphoric acid monomer, which constitutes the main body of the adhesive 3D-SR monomer and is required to demineralize the tooth substance, various monomers that form bonding layers, alcohol solvent, water, and camphorquinone, which is a photopolymerization catalyst. The bond pH is 2.8.

Table 1 *Composition of Tokuyama Bond Force II*

BASIC COMPONENTS	FUNTION
Phosphoric acid monomer (3D-SR monomer)	Decalcification of tooth substance, Formation of bonding layer
Bis-GMA	Formation of bonding layer
3G (TEGDMA)	Formation of bonding layer
HEMA	Penetration into the tooth substance, Formation of bonding layer
Alcohol	Solvent
Water	Decalcification of tooth substance
Camphorquinone	Photopolymerization catalyst

2.3 INSTRUCTIONS

The instructions for Tokuyama Bond Force II are illustrated in *Figure 3*. The procedure for use is short: apply with the brush a suitable amount of bonding agent brushing the walls of the cavity to properly wet them, wait for 10 seconds, apply mild air for approximately 5 seconds, and light cure for 10 seconds. As stated above, this product achieves a shorter chair time and simpler handling than Tokuyama Bond Force.



TIPS: while performing this brushing motion, apply new bonding agent in the cavity once or twice to maximize the demineralizing and bonding properties of the dentinal substrate and achieve high adhesion values.

Figure 3 *Instructions for Tokuyama Bond Force II*

3 Features

3.1 ADHESION TO TOOTH STRUCTURE

Tokuyama Bond Force II has excellent properties of adhesion to the tooth structure. These were evaluated in tensile bond strength measurement as shown in *Graphics 1-2-3*. The results of initial bond strength are shown in *Graphic 1*, and those after 3.000 and 10.000 thermal cycles (4°C⇔60°C, dwell time of one minute) in a durability test are shown in *Graphics 2-3*, respectively.

Tokuyama Bond Force II showed excellent adhesive properties with respect to both cut enamel and cut dentin. These results are due to the polymerization of reaction products of the new adhesive 3D-SR monomers (developed as an essential element of Tokuyama Bond Force II) and the calcium, forming a strong bonding layer on the tooth surface.

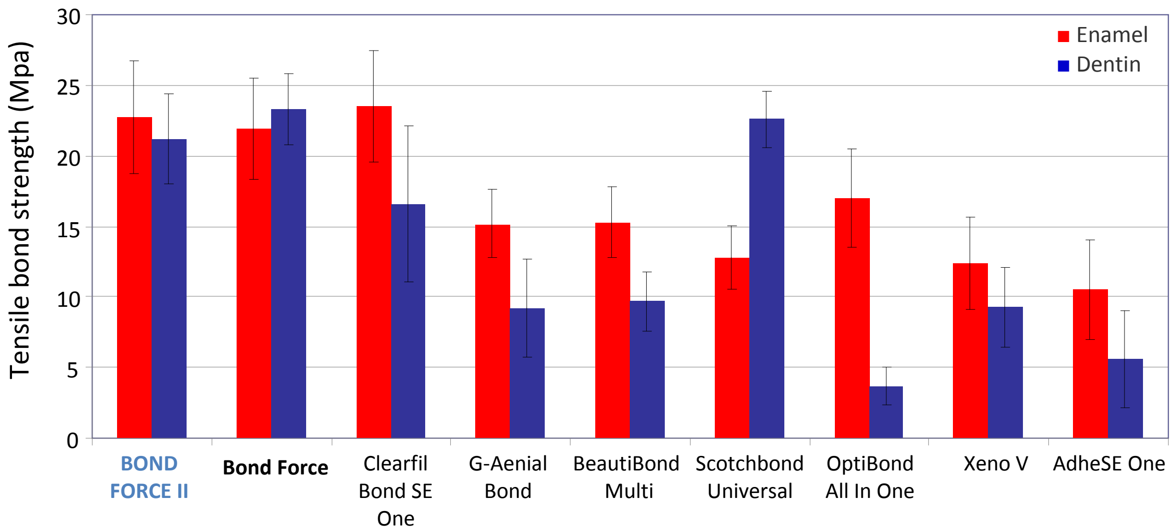
Test method:

1) Grind an adhesive flat surface on the labial surface of an extracted first anterior bovine tooth using #600 Si-C paper. Determine the adhesion area using double-sided tape with 3-mm-diameter holes. Attach a wax sheet with a size of 8 mm in diameter and 0.5 mm in thickness hole to create a simulated cavity.

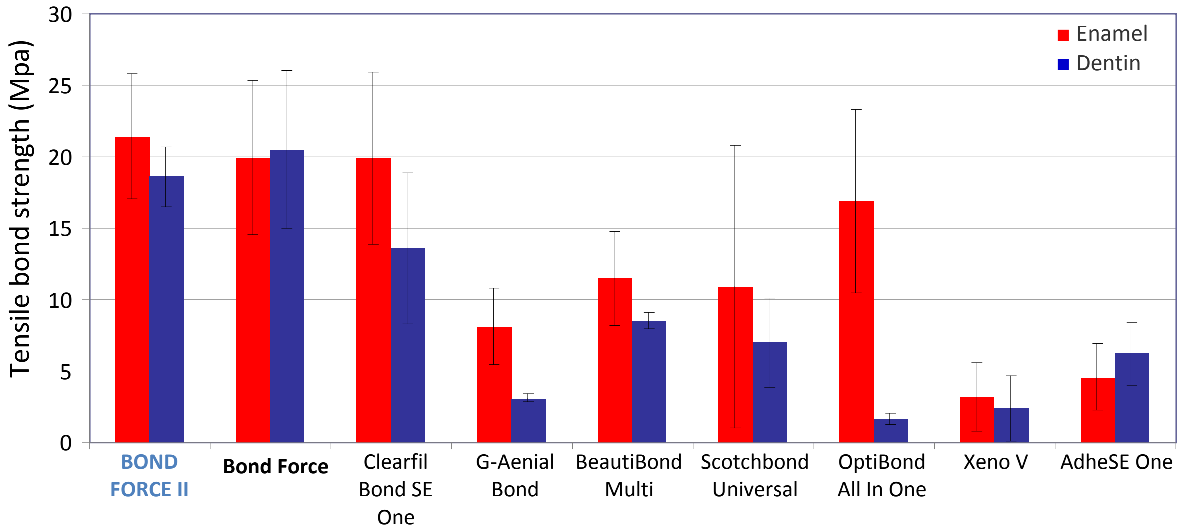
2) Apply Tokuyama Bond Force II, wait for 10 seconds, and apply mild air. Then, light cure for 10 seconds. Follow the manufacturer's instructions when using products manufactured by other companies.

3) Fill the above-mentioned simulated cavity with resin composite (Estelite Posterior Quick/Tokuyama Dental Corp.).

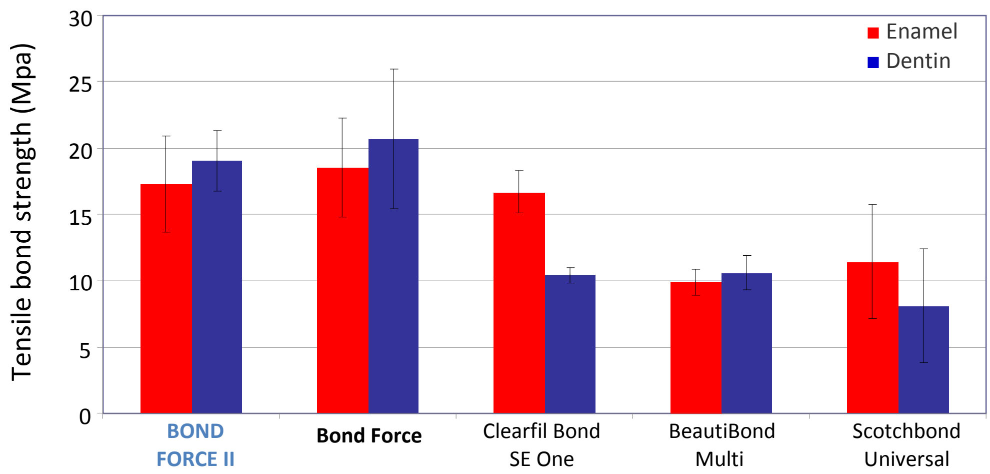
4) After immersing the specimens in water at a temperature of 37°C for 24 hours, or after performing a durability test, carry out a tensile test (n=4) at a crosshead speed of 2 mm/min using a universal machine (AG-5000D, Shimadzu Corp.).



Graphic 1 *Tensile bond strength (initial, self-etch mode)*



Graphic 2 *Tensile bond strength (after 3,000 thermal cycles, self-etch mode)*

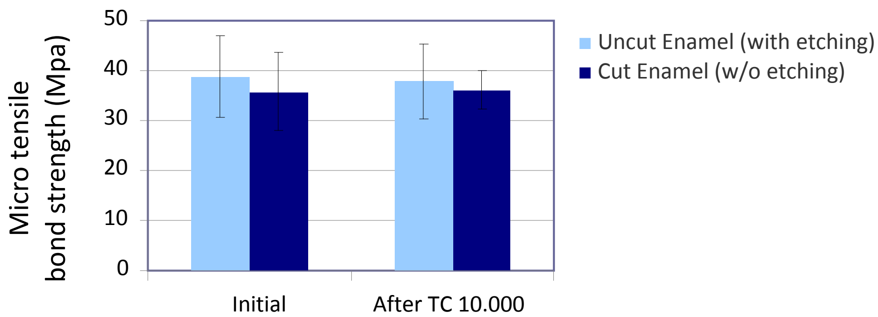


Graphic 3 *Tensile bond strength (after 3,000 thermal cycles, self-etch mode)*

Following this, the bond strength of Tokuyama Bond Force II to the uncut enamel (margin area) were evaluated, i.e., the initial bond strength and those after 10,000 thermal cycles (4°C ⬄ 60°C, dwell time of one minute) were evaluated by a microtensile bond strength measurement. Since Tokuyama Bond Force II has an increased pH of 2.8, phosphoric acid etching is required for uncut enamel. The properties of adhesion to the cut enamel (without phosphoric acid etching) were also evaluated for comparison. The results show that Tokuyama Bond Force II exhibits excellent adhesion to the uncut enamel with phosphoric acid etching, equivalent to those for cut enamel, as shown in *Graphic 4*.

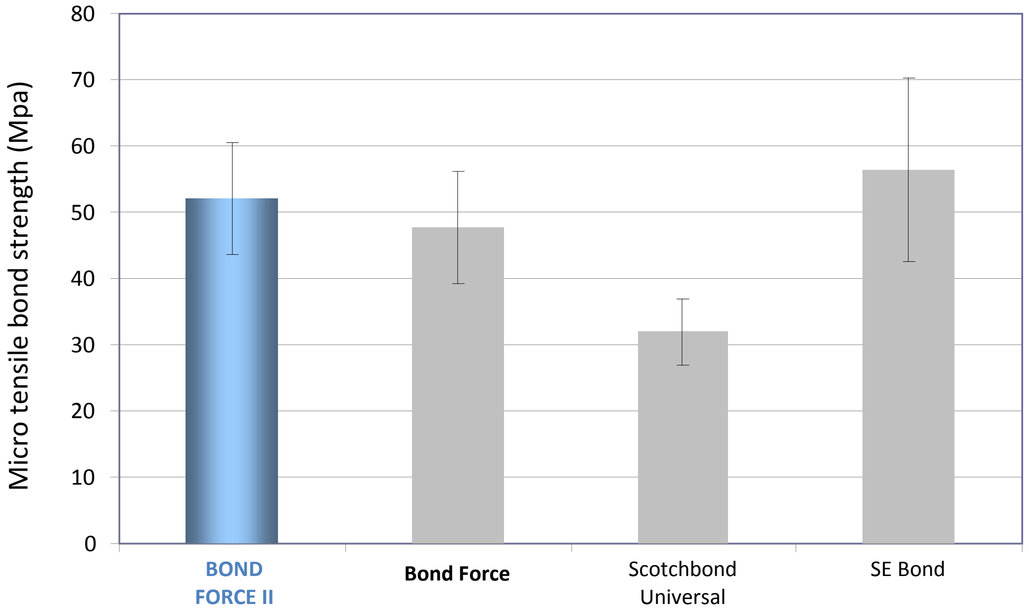
Test method:

1) Use Pressage (Shofu Inc.) to clean the uncut enamel on the labial surface of an extracted bovine first anterior tooth, and rinse with water. Next, apply Tokuyama Etching Gel HV, wait 15 secs rinse with water, and dry. Use #600 Si-C paper to polish the cut enamel on the labial surface of the extracted bovine first anterior tooth, rinse with water, and dry.
2) Apply Tokuyama Bond Force II on the adhesive surfaces.
3) Fill with resin composite (Estelite Posterior Quick/Tokuyama Dental Corp.) in layers.
4) Immerse the specimens in water for 24 hours (initial), or perform 10,000 thermal cycles (TC, 4°C ⬄ 60°C, dwell time of one minute), and then cut to 1 mm × 1 mm stick using a diamond cutter.
5) Perform a tensile test using a universal machine (Ez Test, Shimadzu Corp.) at a crosshead speed of 1 mm/min.

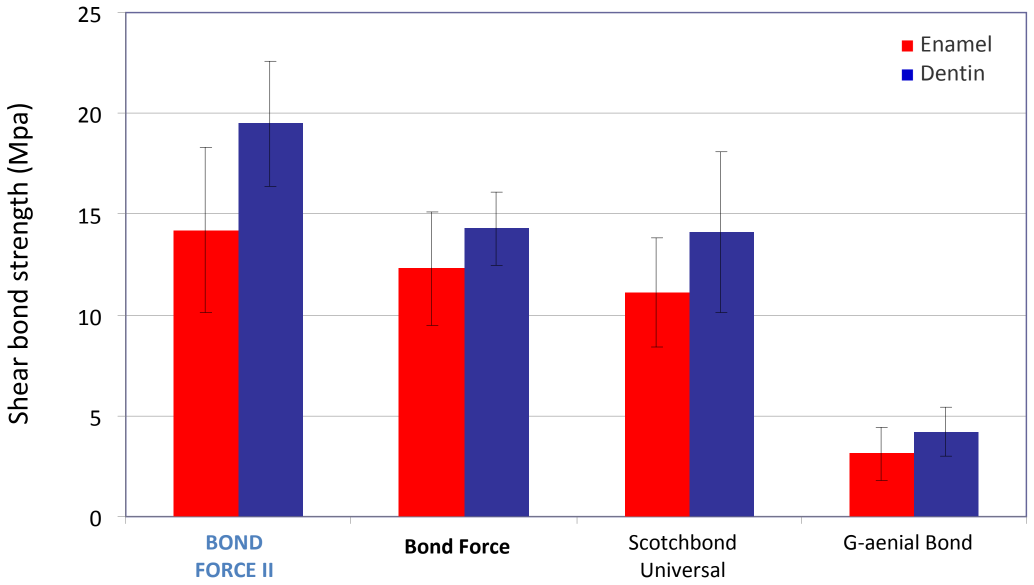


Graphic 4 *Adhesion to uncut enamel*

The adhesion of Tokuyama Bond Force II to dentin was evaluated using a microtensile bond strength measurement, and its properties of adhesion to the tooth structure were evaluated using a shear bond strength measurement. The results of the microtensile bond strength measurement are shown in *Graphic 5*, and those of the shear bond strength measurement in *Graphic 6*. With both methods, Tokuyama Bond Force II showed excellent adhesion to the tooth structure.



Graphic 5 *Microtensile bond strength to dentin*



Graphic 6 *Shear bond strength*

3.2 RELIABLE ADHESIVE PROPERTIES

Figure 4 shows an image of Tokuyama Bond Force II one minute after dispensing. As with Tokuyama Bond Force, Tokuyama Bond Force II also did not exhibit phase separation. This property of no phase-separation is believed to contribute to the reliability of the adhesive properties of these products, i.e., it decreases technique sensitivity.

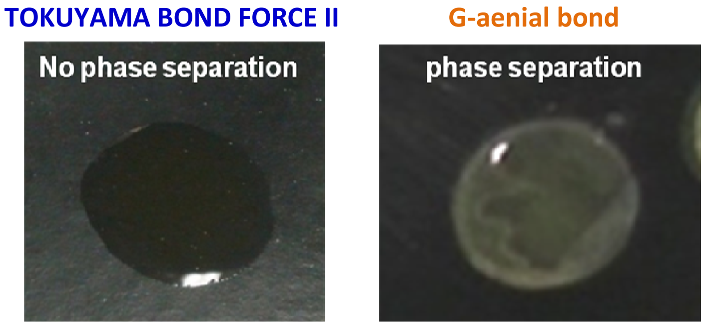


Figure 4 *Images after dispensing*

In previous one-bottle one-step bonding agents, there have been problems with technique sensitivity, since bonding strength was affected by the air blowing condition, which is performed in order to vaporize the solvent. Hence, for the original Tokuyama Bond Force, it is recommended that air blowing be performed in two steps, i.e., weak air flow for five seconds and then strong air flow for five seconds. In comparison, Tokuyama Bond Force II is less likely to be affected by air blowing since it utilizes enhanced adhesive 3D-SR monomers and is designed to inhibit phase separation; the simpler process of mild air blowing for five seconds is therefore recommended for Tokuyama Bond Force II. The adhesion interface of dentin was observed with a laser microscope (Laser Scanning Microscope VK-9700 (Keyence Corp.), mirror surface polished by diamond paste (final: 0.25 µm), × 140)[7] *Figure 5*. Tokuyama Bond Force II did not exhibit any gaps at the interface with the dentin, demonstrating its excellent properties of adhesion to dentin, and formed a thin, uniform bonding layer. These results are due to the polymerization of reaction products of the new adhesive 3D-SR monomers (developed as an essential element of Tokuyama Bond Force II) and the calcium forming a strong bonding layer on the surface of the tooth, and because—due to optimized composition—no phase separation occurs even after air-blowing. This chemistry and technique provides excellent dentinal sealing, and decreased post-operative sensitivity. In Figure 11, the adhesion interface to the dentin produced by G-aenial Bond, which is a phase-separating bonding agent, is shown for comparison. Voids by phase separation were observed at the adhesive interface.

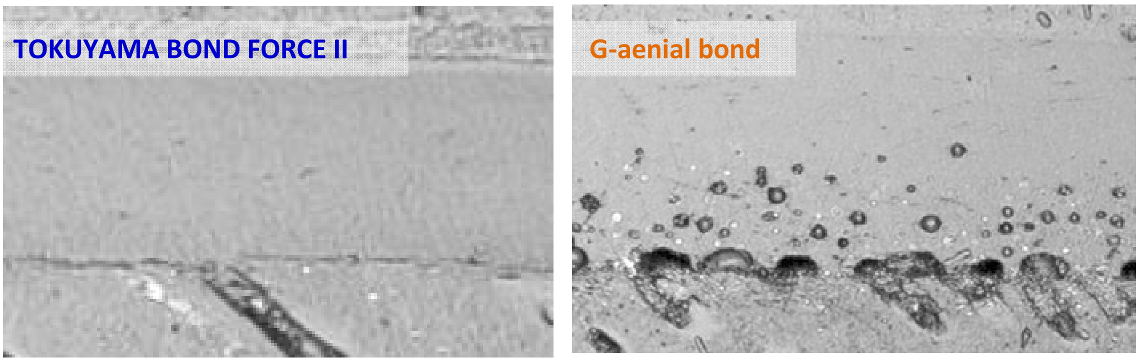


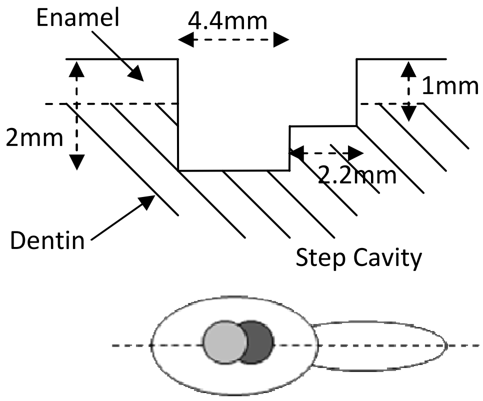
Figure 5 *Laser microscopic images of bonding interfaces with dentin*

3.3 CAVITY ADAPTATION

Cavity adaptation of Tokuyama Bond Force II was compared with that of Scotchbond Universal (3M Espe), as shown in *Figures 6-7*. Gap free bonding layer and uniform thickness were observed on the cavity floors, corners, and edges. This demonstrates excellent cavity adaptation of Tokuyama Bond Force II.

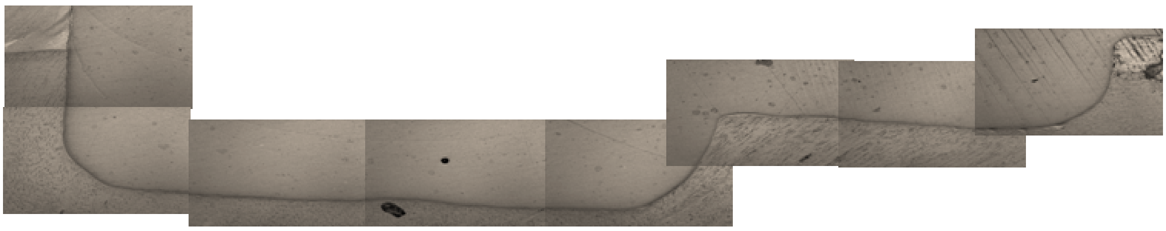
Test method:

1) Create simulated cavities in the form of steps on the labial aspect of an extracted bovine first anterior tooth.



2) Apply Tokuyama Bond Force II, wait for 10 secs, and air dry. Light cure for 10 secs. When applying Scotchbond Universal, wait 20 secs before air drying. Light cure for 10 secs
3) Fill with resin composite in layers (Estelite Flow Quick/Tokuyama Dental Corp. for Tokuyama Bond Force II, and Supreme Ultra Flow/3M Espe for Scotchbond Universal)
4) Cut perpendicular to the adhesion surface using a diamond cutter
5) Polish the cut surface using diamond paste (final: 0.25 µm)
6) Observe with laser microscope (VK9700, Keyence Corp.)

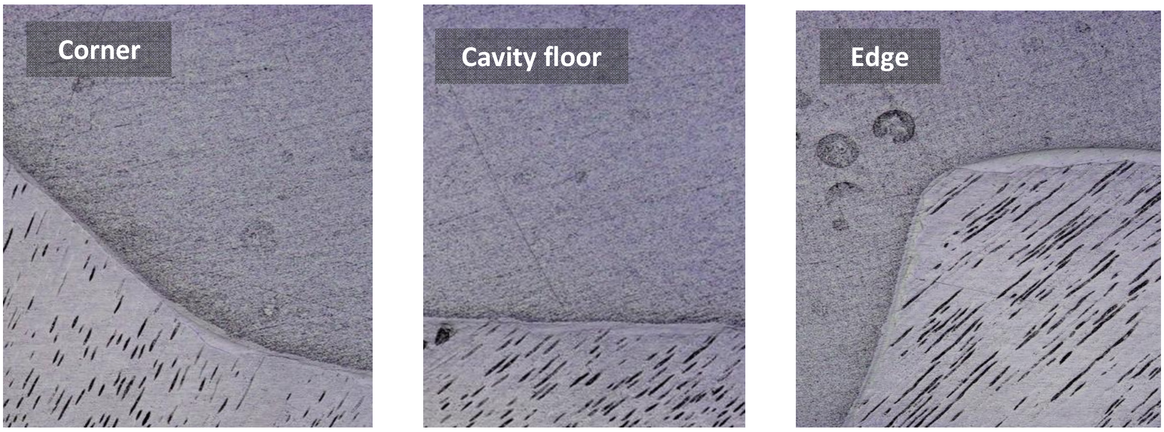
TOKUYAMA BOND FORCE II



Scotchbond Universal

Figure 6 *Cavity adaptation*

TOKUYAMA BOND FORCE II



Scotchbond Universal

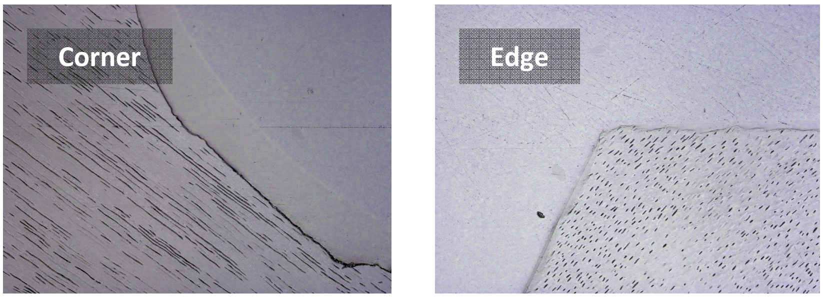


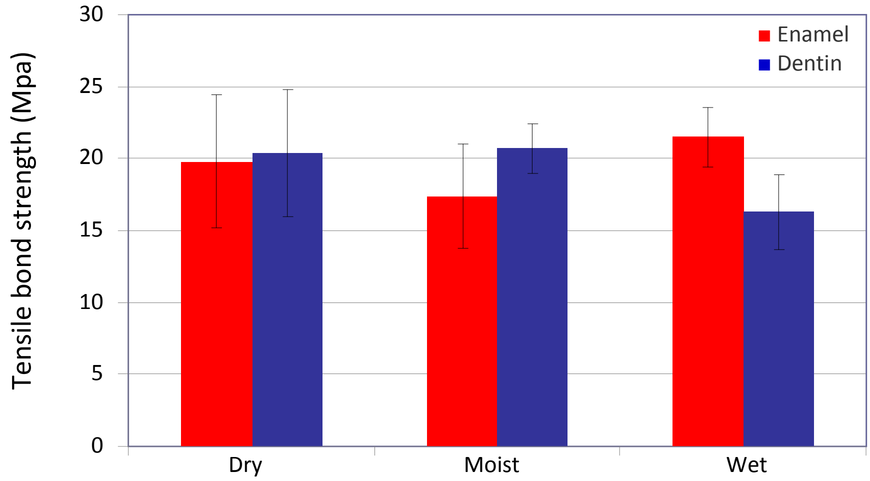
Figure 7 *Cavity adaptation (x280)*

3.4 EFFECT OF TOOTH SURFACE CONDITION

Although it is recommended to dry the bonding surface prior to the bonding procedure, there are a number of cases where moisture cannot be removed during clinical use. The bonding strength of Tokuyama Bond Force II was therefore evaluated under different moisture conditions (wet, moist, and dry), as shown in *Graphic 7*. The results show that adhesive properties are largely unaffected by moisture on the adhesion surface.

Test method:

1) Using #600 Si-C paper, grind an adhesive flat surface on the labial surface of an extracted first anterior bovine tooth. Determine the adhesion area using double-sided tape with 3-mm-diameter holes. Attach a wax sheet with a size of 8 mm in diameter and 0.5 mm in thickness hole to create a simulated cavity
   - DRY dry using a dental air syringe
   - MOIST after rinsing the adhesion surface with water, wipe gently with a Kimwipe
   - WET blow water mist onto the bonding surface
2) Apply Tokuyama Bond Force II, wait 10 secs, and then air dry. Light cure for 10 secs
3) Fill with resin composite (Estelite Posterior, Tokuyama Dental Corp.)
4) After immersing the specimens in water at a temperature of 37°C for 24 hours, carry out a tensile test (n=4) at a crosshead speed of 2 mm/min using an autograph (AG-5000D, Shimadzu Corp.)

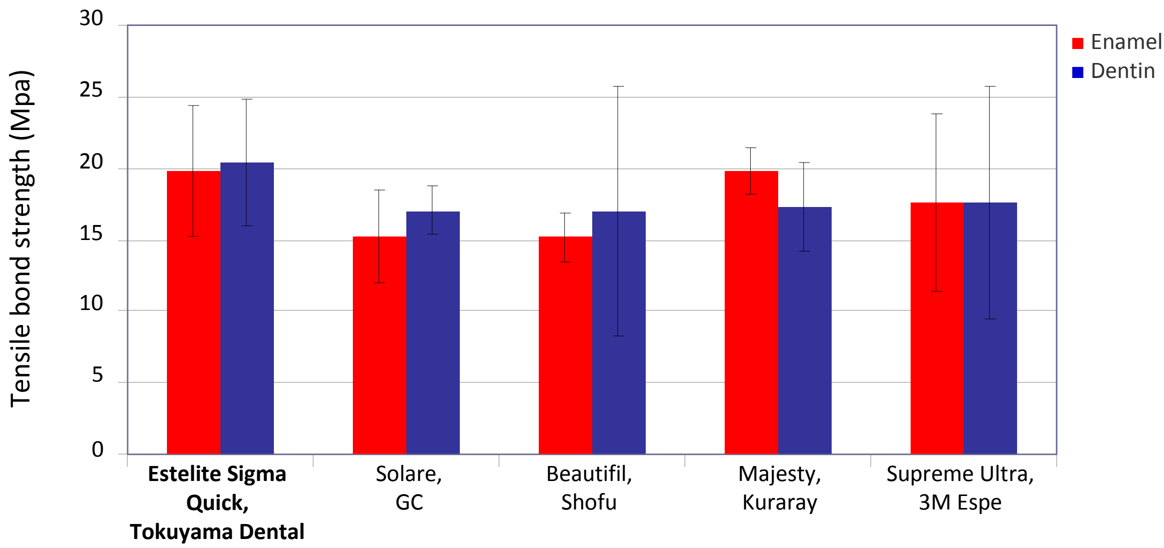


Graphic 7 *Evaluation of effect of moisture condition*

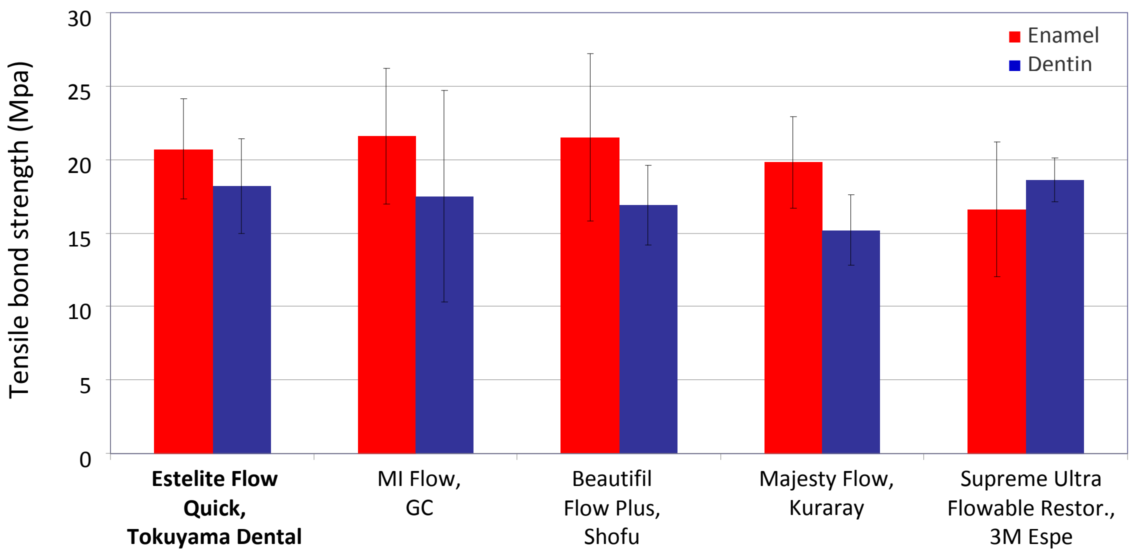
3.5 ADHESION TO RESIN COMPOSITES OF OTHER BRAND

The bond strength using various light-curing resin composites currently available on the market were evaluated. The results for universal resin composites are shown in *Graphic 8*, and those for flowable resin composites in *Graphic 9*. Tokuyama Bond Force II showed excellent adhesion to all of the resin composites produced by other brand; it can therefore be used with any light curing resin composite.

However, if a chemical curing-type resin composite is applied to a cured Tokuyama Bond Force II and the resin composite is then cured by chemical polymerization, no adhesion is exhibited. This is because the acidic monomer in the bonding agent inhibits the chemical polymerization in the chemical cure type resin composite. Hence, Tokuyama Bond Force II should not be used with chemical cure type resin composites.



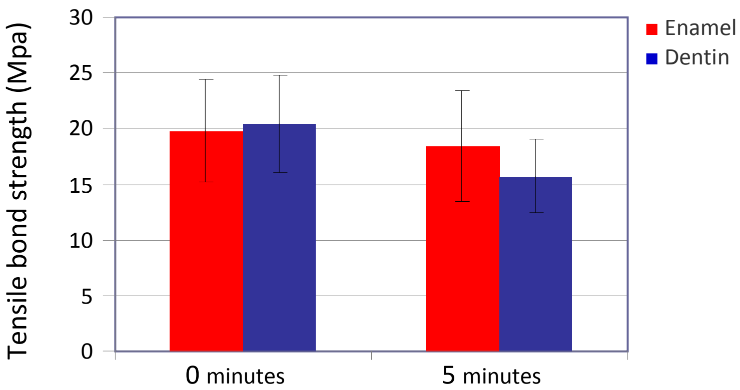
Graphic 8 *Adhesion to resin composites of other brand (universal)*



Graphic 9 *Adhesion to resin composites of other brand (flowable)*

3.6 WORKING TIME

In general, one-bottle one-step bonding agents contain volatile organic solvents. G-aenial bond contain highly volatile acetone, and once a certain amount of acetone has evaporated, the water in the bonding agent shows phase separation. For this reason, the working time is very short, it being necessary to use the product within one minute of dispensing the bonding agent in the dispensing well. If phase separation occurs, adhesion performance is dramatically reduced. Hence, careful use is required during use. In contrast, Tokuyama Bond Force II consists of homogenous bonding agent, with no phase separation of the bonding agent occurring even when all the added volatile organic solvents have vaporized. Alcohol, which is less volatile than acetone, is used as the solvent. For this reason, Tokuyama Bond Force II has the relatively long working time of five minutes from dispensing of one drop in the dispensing well, making clinical procedure less stressful. The effect on bonding strength of the working time - from dispensing one drop of bonding agent in the dispensing well- at room temperature without direct light is shown in *Graphic 10*. It demonstrates that even five minutes after dispensing, good adhesive properties are retained.



Graphic 10 *Effect of working time after dispensing on bonding strength*

3.7 MARGINAL LEAKAGE

The marginal leakage of Tokuyama Bond Force II was evaluated *Table 2*. Pigment penetration was not observed, and Tokuyama Bond Force II showed excellent resistance to marginal leakage compared to other brands.

Test methods:

1) Create a simulated cavity in the form of a cylinder of 4 mm in diameter × 4 mm on the labial surface of an extracted bovine first anterior tooth
2) Apply Tokuyama Bond Force II, wait for 10 seconds, and air dry. Light cure for 10 seconds. When testing Scotchbond Universal, apply and wait for 20 seconds, and air dry. Light cure for 10 seconds.
3) Fill with resin composite (Estelite Posterior, Tokuyama Dental Corp.) in layers
4) Immerse the specimens in water at 37°C for 24 hours, and then in 1% fuchsin solution at 37°C for 24 hours
5) Cut in a perpendicular direction to adhesion surface using a diamond cutter
6) Polish the cut surface using Si-C paper (final: #3000)
7) Observe pigment penetration with a laser microscope (Laser Scanning Microscope VK-9700, Keyence Corp.)

Table 2 *Composition of Tokuyama Bond Force II*

BONDING AGENTS	MARGINAL LEAKAGE (n=4)			
Tokuyama Bond Force II	–	–	–	–
Tokuyama Bond Force	–	–	–	–
Tokuyama EE-Bond	–	–	–	–
Scotchbond Universal	++	–	++	–

– : no pigment penetration
++ : penetration into dentin
+ : penetration into enamel
+++ : penetration into cavity floor

3.8 SELECTABLE CONTAINER

There are 3 types of dispensing systems (bottle, pen-type, and unit dose) for Tokuyama Bond Force II *Figure 8*. The bottle dispenses a greater amount, making it suitable for procedures on more than one tooth or larger cavities. The pen-type is suitable for use on smaller cavities, and the unit dose provides the best hygiene.

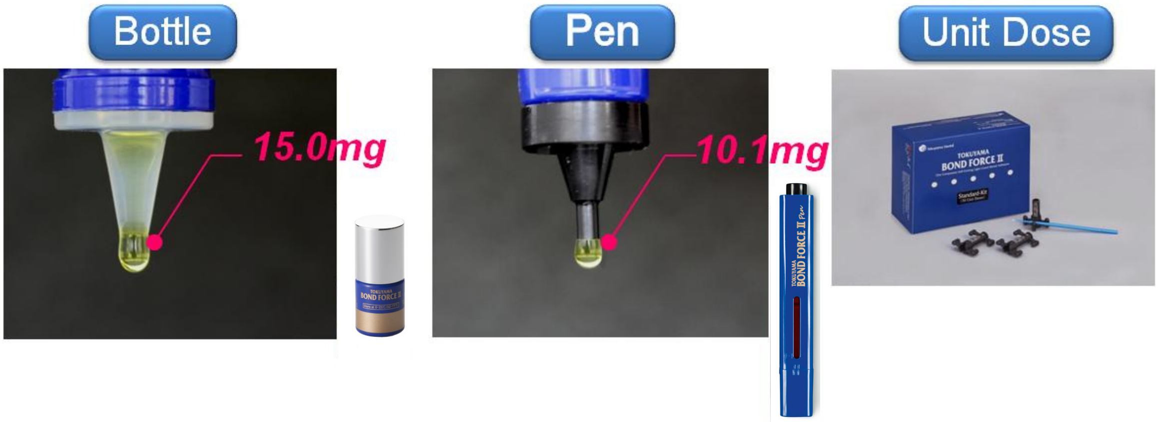


Figure 8 *Selectable container*

To aid in re-ordering, the pen-type has an improved visibility window with fluid level indicator. *Figure 9* shows the improved visibility window with fluid level indicator. By examining the indicator, the approximate amount of remaining material can easily be seen. Additionally, the nozzle tip has an improved design to aid in a cleaner dispensed drop.

Figure 9 *Visibility window with fluid level indicator*

4 Conclusion

Tokuyama Bond Force II, which was launched in Japan in January 2014, is an excellent one-bottle one-step bonding agent which, thanks to new adhesive 3D-SR monomers and optimized composition, demonstrates the features listed below. As a result of these features, it is anticipated that - because of the superior physical properties it provides - Tokuyama Bond Force II will be clinically useful as an excellent dental bonding agent to tooth substrate.

New features of Tokuyama Bond Force II:

1) Simple and quicker procedure with reliable adhesion
2) Ambient temperature storage (0-25°C)
3) Selectable container (Bottle and Pen)

5

5 References

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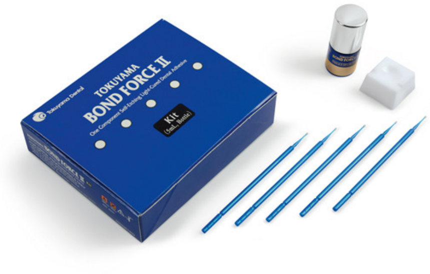
BOND FORCE II Packaging



14903 - BOND FORCE II KIT

Bond Force II Bottle, 5ml
1 Dispensing well
25 Disposable applicator

14968 - BOND FORCE II PEN KIT

Bond Force II Pen, 2ml
1 Dispensing well
25 Disposable applicator

notes:

Precision, adaptability, ease of use and conservation: the definitive solution to prevent the infiltration of enamel bevel!

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Tokuyama Dental