TOKUYAMA UNIVERSAL BOND

Technical Report
Ver. 1.2

Tokuyama Dental Corp.
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1. Introduction

1.1 Development Background

Recently, "universal" type products have been widely used in the dental bonding material market because they are simple to handle. Although the definition of "universal" has not been standardized, THE DENTAL ADVISOR[1)] lists the following three requirements:

1. Compatibility with different etching techniques: total-, self-, or selective-etch.
2. Compatibility with dual- and self-cure materials without the use of a separate activator.
3. Can be used as a primer for silica-based and/or zirconia-based and metallic restorations.

Of the "universal" type products commercially available, there are no bonding materials that meet all the three requirements. In addition, the objects to be bonded (bondable materials) are limited according to the product, and the usage method may vary from object to object; therefore, caution is required in the use of these products.

Tokuyama Dental Corp. intended to commercialize a bonding material that meets all the three requirements mentioned above.

We have succeeded in covering all the etching modes for adhesion to tooth substance by improving our unique adhesive monomer (3D-SR monomer). This improved 3D-SR monomer also enhanced bond strength to zirconia or metals and it particularly enabled a strong bond to not only tooth substance but various materials including ceramics, CAD/CAM blocks or metal in combination with various functional monomers such as a new silane coupling agent or adhesive monomer to precious metals.

In addition, we have achieved a high bond strength that does not require photoirradiation by applying our own polymerization initiator (BoSE technology) characterized by excellence in polymerization under acid conditions and promotion of polymerization from the adhesive interface (Contact Cure). Conventionally, a process where no photoirradiation is applied to resin materials such as composite resin or resin cement used in combination, decreased bond strength has been generally observed[2)]. However, application of BoSE technology could solve this problem that arose when using any of the dual-curing and self-curing type resin materials, leading to development of "TOKUYAMA UNIVERSAL BOND" with performance worthy of being called "universal."

1.2 Product Description

TOKUYAMA UNIVERSAL BOND is a two-component self-cured dental adhesive system for both direct and indirect restorations that can be used with Self-etch, Selective-enamel-etch and Total-etch techniques. As a universal adhesive, TOKUYAMA UNIVERSAL BOND has been designed to be fully compatible with light-cured, self-cured and dual-cured composite materials. TOKUYAMA UNIVERSAL BOND improves the bond strength of polymerizable resin material (adhesive resin cement, acrylic resin and composite resin) to indirect restorative materials such as glass-ceramics (porcelain), oxide-ceramics (zirconia and alumina), metals (precious and non-precious) and resin materials including inorganic filler.

Features of TOKUYAMA UNIVERSAL BOND

[Universal Use]

- Compatibility with self-etch, total-etch and selective-etch techniques
- Applicability to direct and indirect restoration
- Compatibility with light-curing, dual-curing and self-curing composite materials without the use of a separate activator
- Can be used as a primer for silica-based, zirconia based and metallic restorations

[Simple Handing]

- No need to apply separately for tooth and restoratives
- No need to wait after bond application
- No need to light-cure

[Reliable]

- High bond strength

2. Composition and Instructions

2.1 Composition

The composition of TOKUYAMA UNIVERSAL BOND is presented in *Table 1.*

TOKUYAMA UNIVERSAL BOND contains the following: adhesive monomer new 3D-SR monomer (phosphoric acid monomer), 6-methacryloyloxyhexyl 2-thiouracil- 5-carboxylate (MTU-6), γ-methacryloxypropyl triethoxy silane (γ-MPTES), which are adhesive monomers for adhesion to tooth and various prosthetics; several monomers (HEMA, Bis-GMA, and TEGDMA) to form bonding layers; acetone, isopropyl alcohol, and water as solvents; and a borate catalyst and peroxide as polymerization initiators.

TOKUYAMA UNIVERSAL BOND has excellent storage stability as these functional ingredients are separated into two liquids, and bond strength is not inferior to that of light-curing type adhesive by using a highly active polymerization catalyst.

Table 1 Composition of TOKUYAMA UNIVERSAL BOND

Bond A

Basic components	**Function**
Phosphoric acid monomer （New 3D-SR monomer）	Adhesion for tooth Formation of bonding layer Adhesion for zirconia , alumina, and non-precious metal
MTU-6	Adhesion for precious metal
HEMA	Penetration into the tooth substance Formation of bonding layer
Bis-GMA	Formation of bonding layer
TEGDMA	Formation of bonding layer
Acetone	Solvent

Bond B

Basic components	**Function**
γ-MPTES	Adhesion for glass ceramics and resin composite
Borate	Polymerization catalyst
Peroxide	Polymerization catalyst
Acetone, Isopropyl alcohol	Solvent
Water	Solvent

2.2 Adhesion Mechanism

TOKUYAMA UNIVERSAL BOND enables reliable adhesion to the tooth due to the adoption of 3D-SR technology using a new 3D-SR monomer, and BoSE technology using a borate initiator.

TOKUYAMA UNIVERSAL BOND also contains the following adhesion monomers that are effective for a range of prosthetics: new 3D-SR monomer (for adhesion to non-precious metal, zirconia, and alumina), MTU-6 (for adhesion to precious metals), and γ-MPTES (for adhesion to glass ceramics and resin composite). New 3D-SR monomer has effect to adhesion on both tooth and some prosthetics (non-precious metal, zirconia and alumina).

The mechanism of adhesion to tooth and each prosthetic is described in detail below.

2.2.1 Mechanism of adhesion to tooth substance

Tokuyama Dental Corp. developed 3D-SR technology to improve adhesion of the bonding agent TOKUYAMA BOND FORCE to the tooth substance[3, 4]. This 3D-SR monomer (1st generation), having several functional groups that can interact with calcium and polymerizing groups per molecule *Fig. 1*, interacts with calcium in the tooth substance at multiple points to create strong adhesion to the tooth structure surface. Further, three-dimensional cross-linking occurs via calcium, and copolymerization between adhesive 3D-SR monomers and other monomers contributes to the formation of a very strong bonding layer. *Fig. 2*

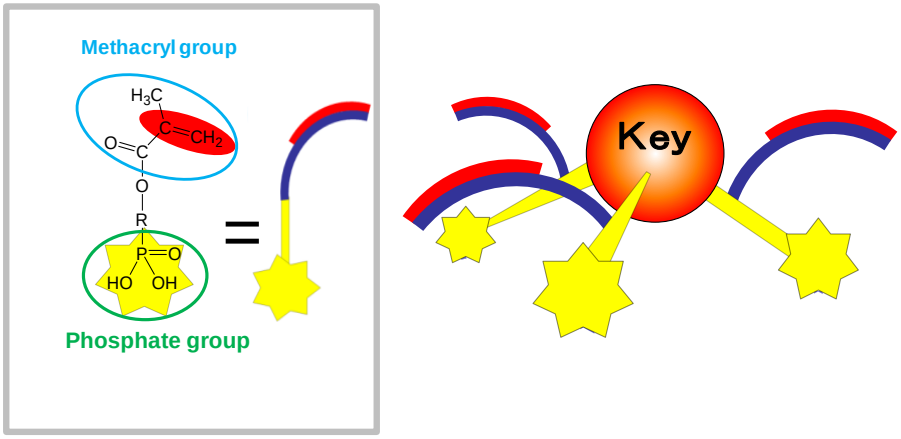


Fig.1 1st Generation 3D-SR Monomer

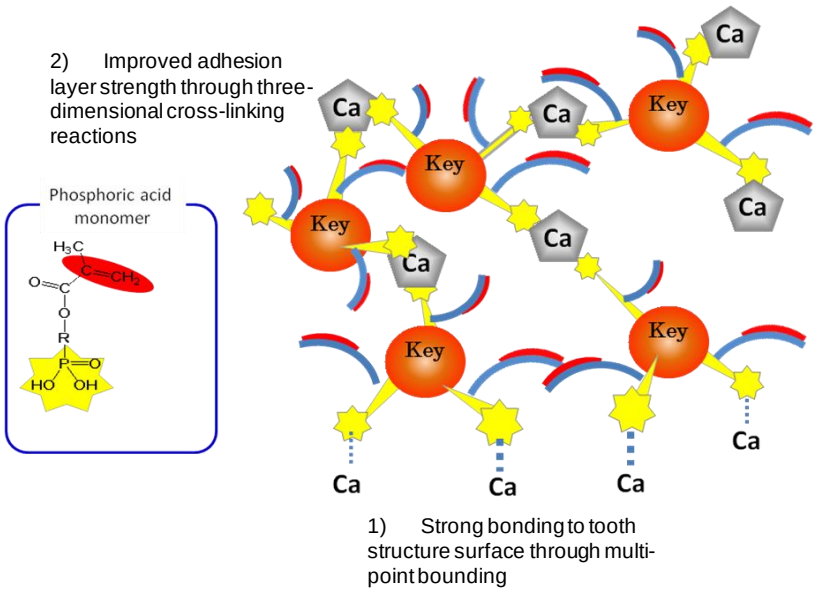


Fig.2 Three-dimensional cross-linking reactions of adhesive SR monomers and calcium ions

To achieve the enhanced properties, our patented 3D-SR monomers were improved and the chemistry was optimized (2nd generation, *Fig. 3*). 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. 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 and ESTELINK provide decreased application time from the previous 20 seconds down to 10 seconds.

Furthermore, the 2nd-generation 3D-SR monomer was adopted for TOKUYAMA UNIVERSAL PRIMER as a zirconia adhesive monomer.

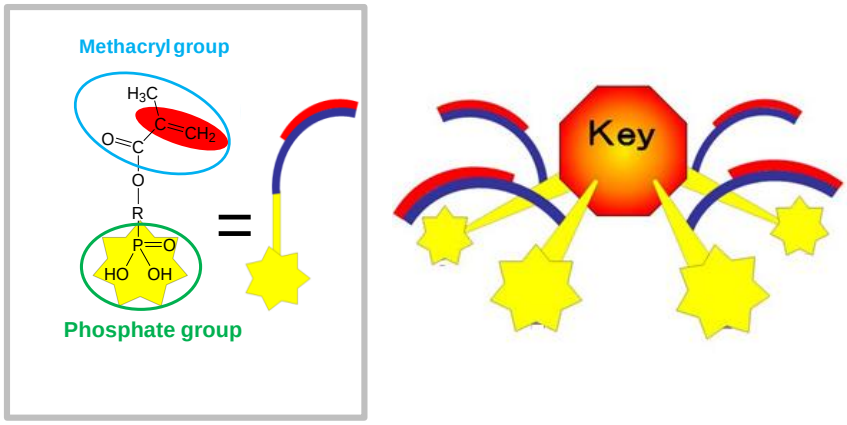


Fig.3 2nd Generation 3D-SR Monomer

TOKUYAMA UNIVERSAL BOND has an enhanced response to tooth calcium and durability by combining several phosphoric acid monomers having different chain lengths of the main chain, the alkylene group (the 3rd-generation 3D-SR monomer, *Fig. 4*). This could reduce the tooth application time from 10 to 0 seconds.

Furthermore, this 3rd-generation 3D-SR monomer contributes to adhesion to non-precious metal as well as tooth, zirconia.

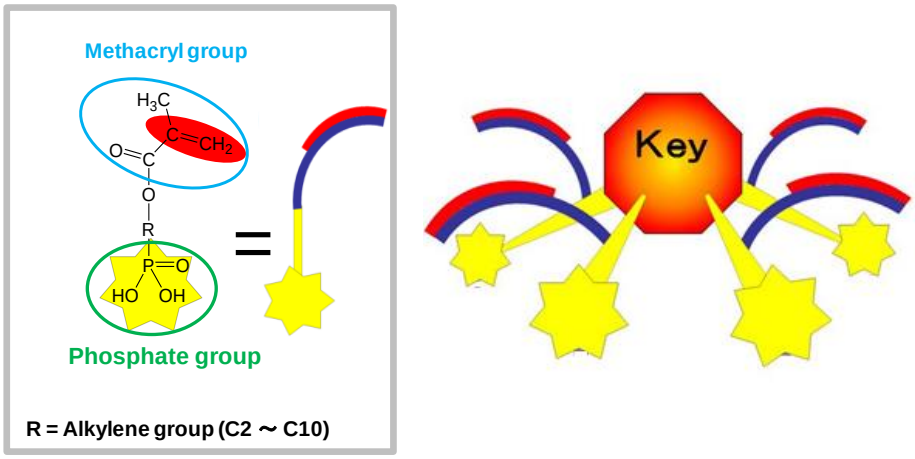


Fig.4 3rd Generation 3D-SR Monomer

2.2.2 Mechanism of adhesion to precious metal

The adhesive monomer for precious metal is MTU-6. As shown in *Fig. 5*, the sulfur atom in the thiouracil group of MTU-6 interacts with precious metal (covalent bond) and additionally, the methacryl group co-polymerizes with monomers in dental-curable materials (resin cements, bonding agents, resin composites, etc.) for adhesion.

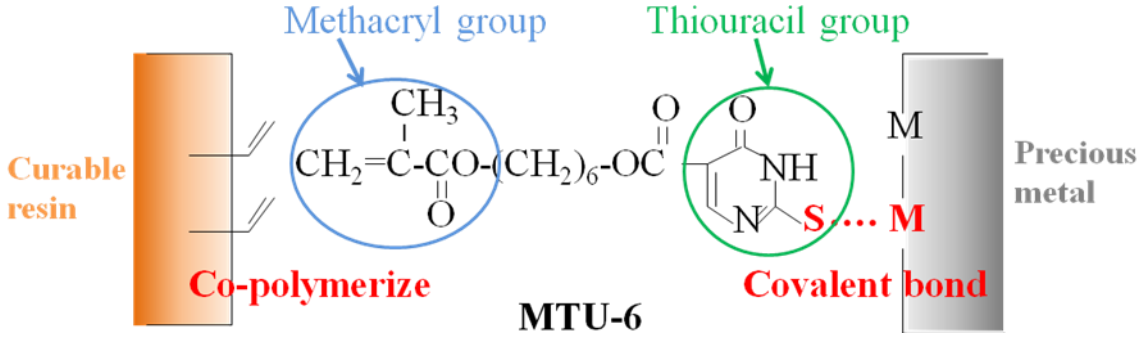


Fig.5 Mechanism of adhesion to precious metal

2.2.3 Mechanism of adhesion to non-precious metal

The adhesive monomer for non-precious metal is new 3D-SR monomer. As shown in *Fig. 6*, the phosphate group of new 3D-SR monomer interacts with the oxygen atom of the passive layer of a nonprecious metal surface (hydrogen bond) and additionally, the methacryl group co-polymerizes with monomers in dental curable materials(resin cements, bonding agents, resin composites, etc.) for adhesion.

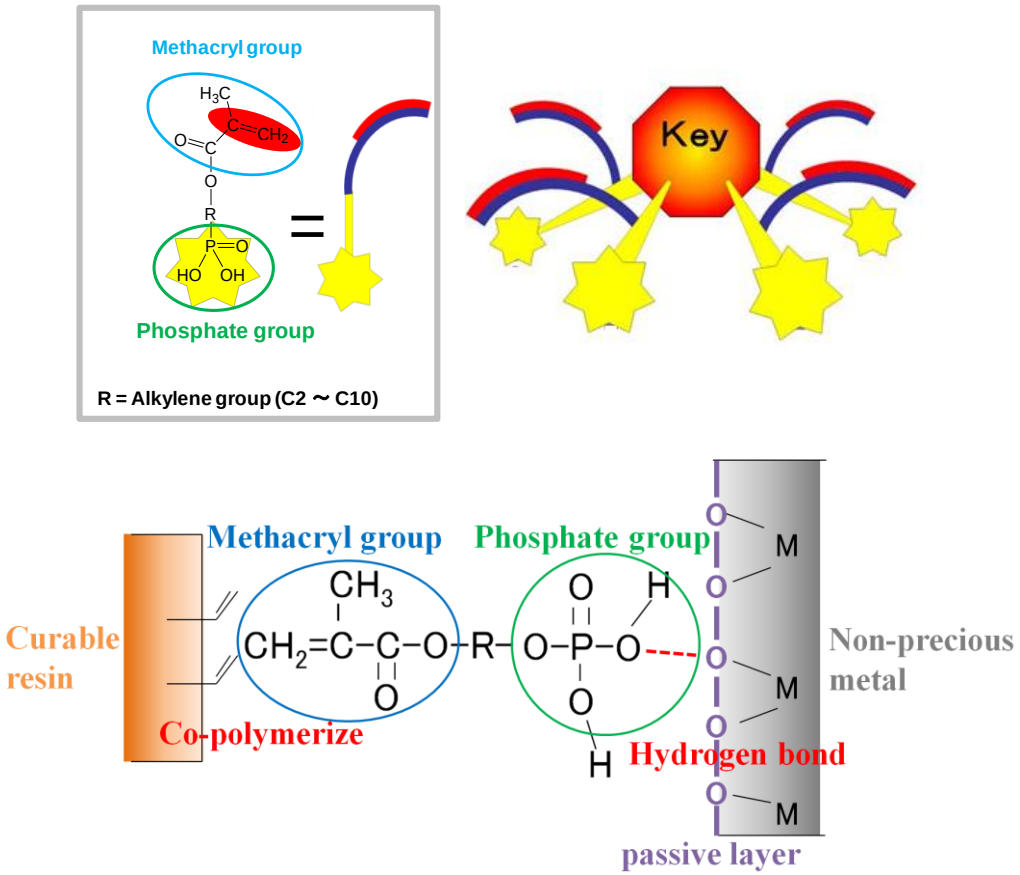


Fig.6 Mechanism of adhesion to non-precious metal

2.2.4 Mechanism of adhesion to glass-ceramics/resin

The adhesive monomer for glass-ceramics, porcelain and resin materials including inorganic filler is the new silane coupling agent, γ-MPTES. First, the alkoxy group in γ-MPTES reacts with water to form a silanol group *Fig. 7* and next, a siloxane bond is formed by a dehydration and condensation reaction with the silanol group on the ceramic surface. Additionally, the methacryl group co-polymerizes with monomers in dental curable materials (resin cements, bonding agents, resin composites, etc.) for adhesion. *Fig. 8*

TOKUYAMA UNIVERSAL BOND is a two bottle-type product, and stabilization of the silane coupling agent in bottles is excellent, and the risk of deterioration of

the silane coupling agent that may occur in a one bottle-type product[5] is reduced. In addition, since the new silane coupling agent, γ-MPTES is more stable in the bottle than the conventional one (γ-MPS), the adhesion effect lasts for a long time.

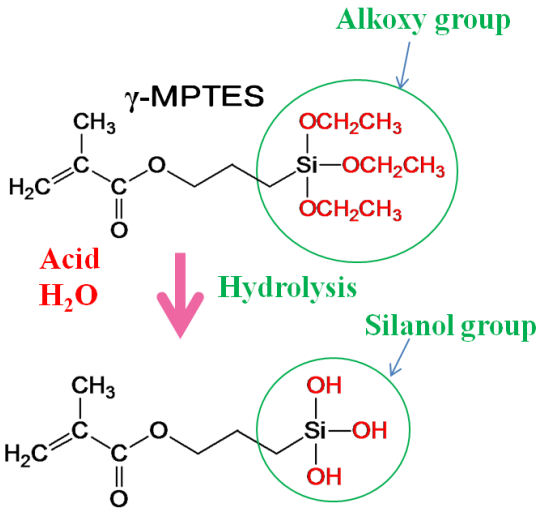


Fig.7 Hydrolysis of γ-MPTES

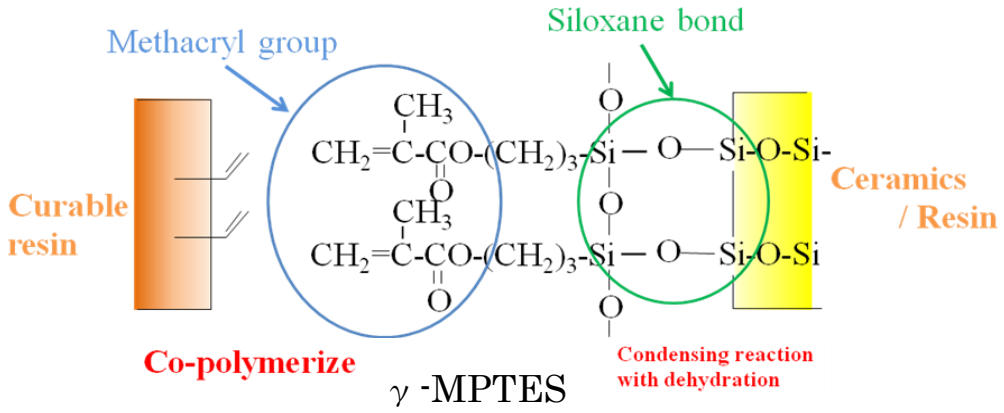
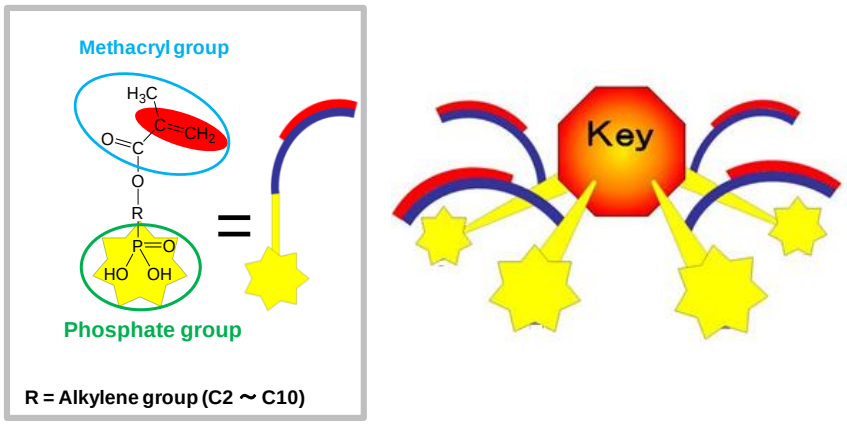


Fig.8 Mechanism of adhesion to glass-ceramics/resin

2.2.5 Mechanism of adhesion to zirconia/alumina

The adhesive monomer for zirconia/alumina is the new 3D-SR monomer (phosphoric acid monomer). It is believed that the phosphate group of new 3D-SR monomer forms chemical bonds with the zirconia/alumina surface for adhesion. *Fig. 9*.



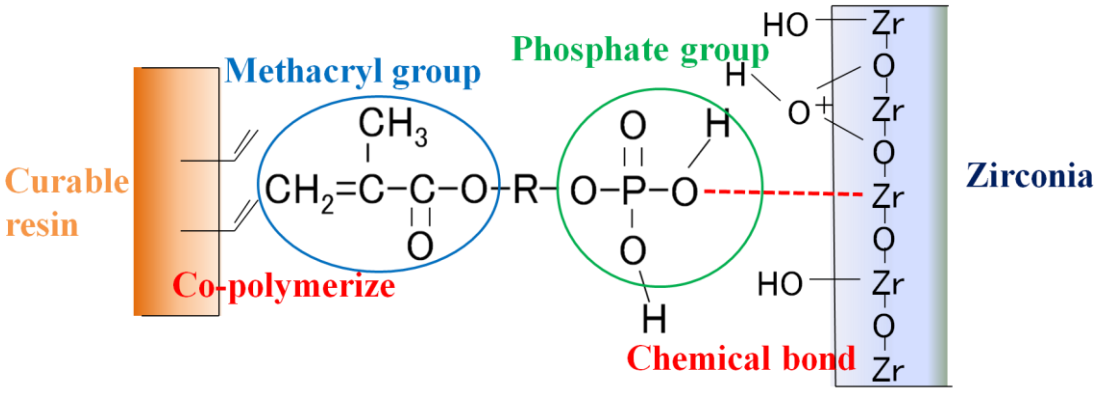


Fig.9 Mechanism of adhesion to zirconia

2.2.6 Mechanism of polymerization initiator "Contact Cure"

TOKUYAMA UNIVERSAL BOND employs our original BoSE technology with a borate initiator. The borate initiator is decomposed by acid (phosphoric acid monomer) and transformed into a borane compound which produces free radicals. In addition, TOKUYAMA UNIVERSAL BOND contains a peroxide that accelerates degradation of the borane compound and serves as a highly active chemical polymerization initiator *Fig. 10*. BoSE technology is superior to the conventional chemical polymerization initiator, a benzoyl peroxide/amine system, because it exhibits high catalytic activity under strongly acidic conditions.

A thin bonding layer formed after air blow becomes hard because of rapid progression of polymerization and curing on its adhesive interface (Contact Cure), when it comes into contact with resin materials such as composite resin.

Excellent polymerization under acidic conditions made it possible to cover self-curing as well as light-curing and dual-curing type resin materials.

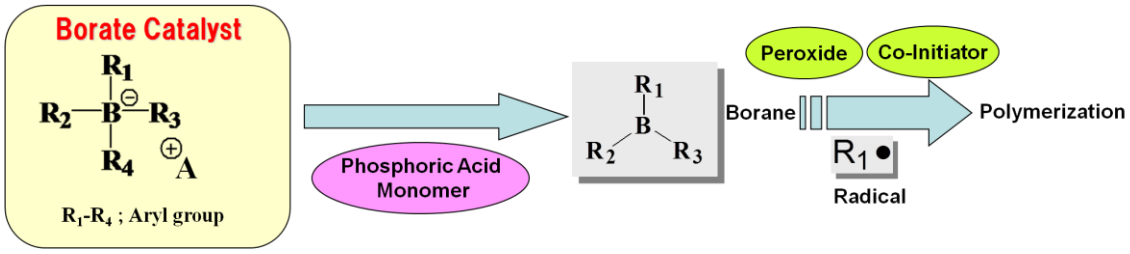


Fig.10 Mechanism of polymerization initiator

2.3 Indications

-Direct anterior and posterior restorations with light-curing, dual-curing, and self-curing composite materials

- Intraoral repair of composite restorations, porcelain fused to metal, metal, and all-ceramic restorations without an additional primer
- Cementation of indirect restorations and veneers when combined with light-cure, dual-cure, and self-curing resin cements
- Bonding of core build-ups made of core build-up materials
- Bonding of denture resin to metal base, clasp or attachment
- Repair of denture with metal base, clasp or attachment
- Bonding of opaque resin to a metal base in the fabrication of resin-faced stainless steel crowns

3. Features - Universal Use

Although the definition of "universal bond" has not been standardized, the following three requirements are listed by THE DENTAL ADVISOR[1]. *Fig. 11*. However, no "universal" type products meeting all these three requirements are commercially available and products meeting only one or two of these requirements are also referred to as "universal." The applicability of commercially available "universal" type bonding materials is shown in *Table 2*. TOKUYAMA UNIVERSAL BOND is the only product satisfying all the three application requirements.

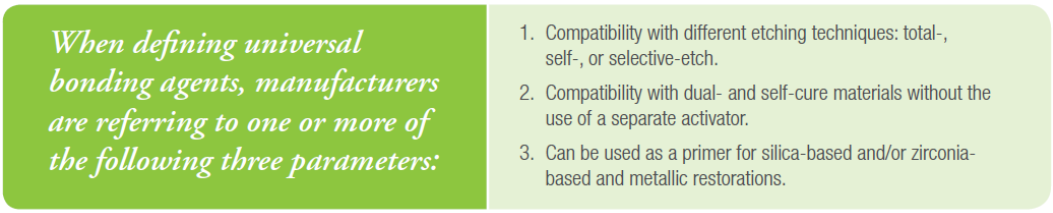


Fig.11 Definition of universal bonding agents by THE DENTAL ADVISOR

Table 2 Applicability of "universal" type bonding materials

Manufacture	Tokuyama Dental	3M ESPE	GC	Voco	Bisco	Kuraray Noritake Dental	Dentsply		Ivoclar Vivadent	Heraeus Kulzer
Product	TOKUYAMA UNIVERSAL BOND	Scotchbond Universal Adhesive	G-Premio BOND	Futurabond U	All-Bond Universal	Clearfil Universal Bond	Prime & Bond Elect	Xeno Select	Adhese Universal	iBond Universal
1. Total-etch, Self-etch, Selective-etch	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
2. Compatible with all light-curing, dual-curing or self-curing composites	✓	△ *1	− *2	✓	✓	△ *6	△ *3	−	✓	✓
2-1. Direct restorations	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
2-2. Indirect restorations	✓	△ *1	△ *3	✓	✓	△ *6	△ *3	−	✓	✓
2-3. Intraoral Repair	✓	✓	△ *4	✓	△ *4	✓ *7	△ *4	−	△ *8	△ *4
3. Primer for prosthesis	✓	△ *1	−	−	✓ *5	△ *6	−	−	−	✓ *5

*1 Requires Dual Cure Activator (DCA) unless it is used with Rely X Ultimate
*2 Bonding of dual-cured core build up composites to tooth structure as long as these materials are light-cured
*3 Requires DCA
*4 Requires Primer
*5 Requires light-curing. Ceramic materials requires ceramics primer
*6 Requires DCA and light-curing unless it is used with CLEARFIL DC CORE PLUS and PANAVIA SA CEMENT
*7 Primer recommended
*8 Only composite repair

3.1 Compatibility with all etching protocols

TOKUYAMA UNIVERSAL BOND can be used with self-etch, total-etch and selective-etch techniques for adhesion to dentin and enamel. *Table 3*.

Table 3 Compatibility with etching protocols

	TOKUYAMA UNIVERSAL BOND	TOKUYAMA BOND FORCE Ⅱ	TOKUYAMA EE BOND
Self-etch	✓	✓	⊖
Total-etch	✓	⊖	⊖
Selective-etch	✓	optionally	✓

3.2 Compatibility with light-curing, dual-curing and self-curing composite materials

TOKUYAMA UNIVERSAL BOND can be used for both direct and indirect restorations and it can also be used with light-curing, dual-curing and self-curing composite materials without the use of a separate activator.

3.3 Can be used as a primer for silica-based, zirconia based and metallic restorations

TOKUYAMA UNIVERSAL BOND can be used as a primer for silica-based, zirconia-based and metallic restorations without the use of separate primers. It can reduce the risk of adhesion failure caused by incorrect material selection and wrong handling and it enables the management of inventory, as bonding materials/primers specific to various objects to be bonded are not required.

4. Features - Simple Handing

As TOKUYAMA UNIVERSAL BOND is applicable to the tooth substance and various prosthetics; for example, it is not necessary to identify the material substance of an object to be bonded or apply it in a different manner according to material substance for intraoral repair. Therefore, it can reduce the risk of adhesion failure caused by incorrect material selection and wrong handling. Furthermore, the product can shorten chair time, as waiting time after application and photoirradiation are not necessary. *Fig. 12.*

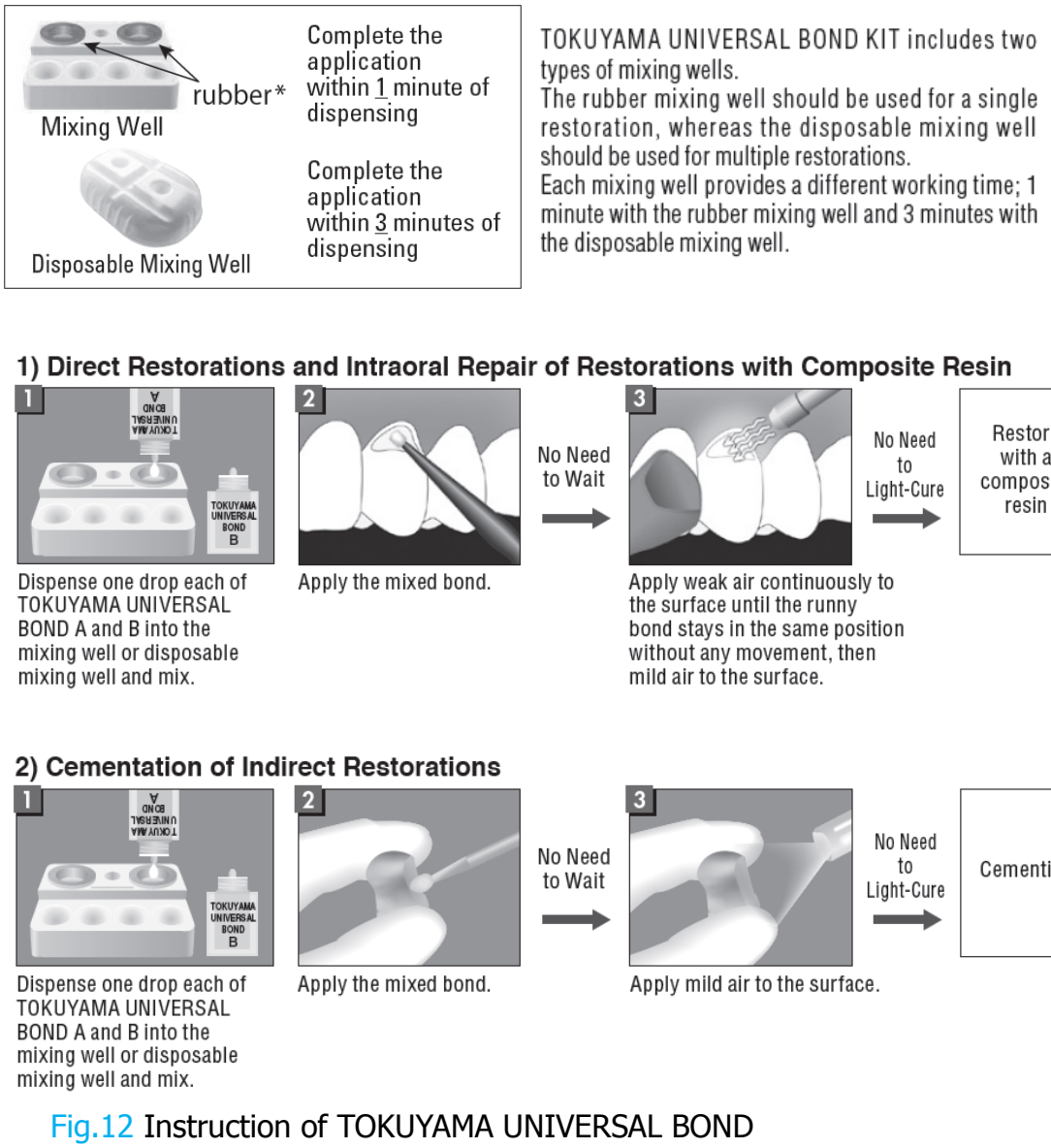


Fig.12 Instruction of TOKUYAMA UNIVERSAL BOND

4.1 Chair Time (Direct Restoration)

TOKUYAMA UNIVERSAL BOND is a two bottle-type bonding material and it takes time to collect and mix the two liquids. However, as time after application and photoirradiation are not necessary, it shortens total chair time compared with competitors' products. In particular, the material can reduce risk of adhesion failure induced by saliva and blood contamination as well as patient burden because of the shorter intraoral chair time.

Comparison of chair time between competitors' universal-type bonding materials is shown below. *Fig. 13*.

Fig.13 Chair time comparison of composite restoration

4.2 Chair Time (Intraoral Repair)

For intraoral repair including PFM fracture, the surface to be bonded is often composed of multiple kinds of materials. TOKUYAMA UNIVERSAL BOND is applicable to any kind of material and can shorten chair time substantially, as it is unnecessary to apply it in a different manner according to the material substance using a metal primer, ceramics primer or other designated primers. *Fig. 14*.

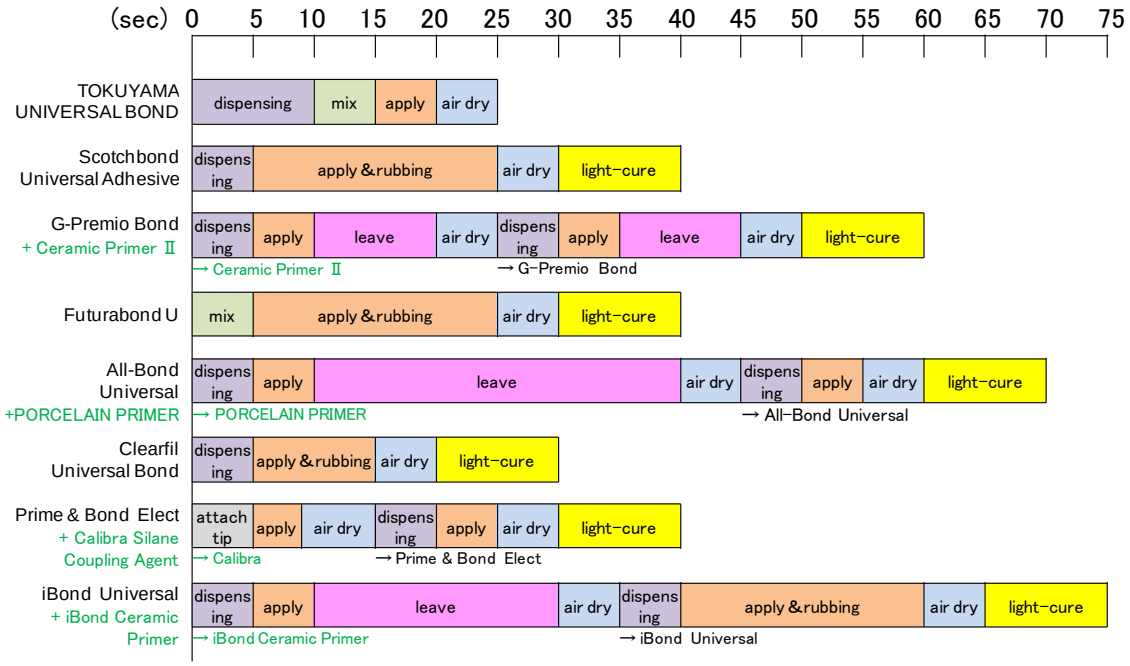


Fig.14 Chair time comparison of intraoral repair of ceramics

4.3 Influence of waiting time after application on bond strength

As mentioned above, TOKUYAMA UNIVERSAL BOND does not require a waiting time after it is applied to the surface to be bonded. *Fig. 15* and *16* show the effect of waiting time from bond application until air blow (0, 3, 5 and 10 seconds) on bond strength on tooth substance and prosthetics. TOKUYAMA UNIVERSAL BOND showed a bond strength on both tooth substance and prosthetics without waiting after bond application that was the same as the strength after a waiting time of 10 seconds.

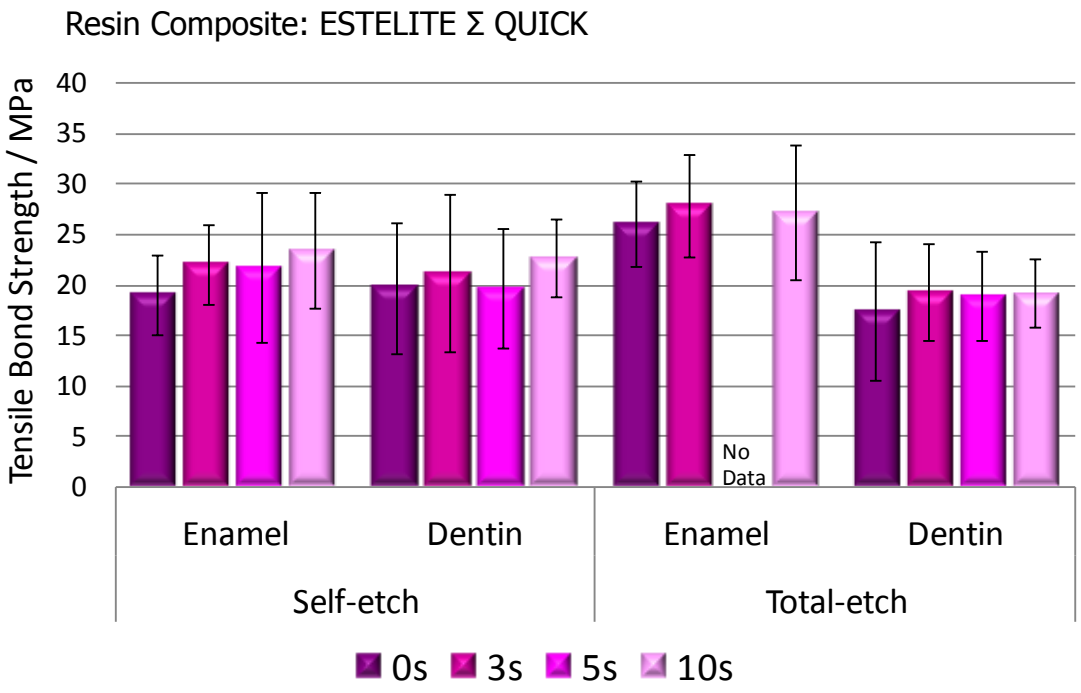


Fig.15 Tensile bond strength on enamel and dentin with varying waiting time when used with the self-etch and total-etch techniques

Table 4 Tested prosthetic materials and pre-treatment method

	Manufacturer	Product name	Composition	Pre-treatment
Precious metal	Tokuyama Dental	CASTMASTER12S	Au12/Pd20/Ag54 /Cu12/other2	1)Grind with #1500 SiC 2)Sandblast (50um of Al_2O_3)
Non-precious metal	Tokuyama Dental	ICROME	Co57.8/Cr31.6/Mo5.6 /other5	1)Grind with #1500 SiC 2)Sandblast (50um of Al_2O_3)
Ceramics (Silica-base ceramics)	Kuraray Noritake Dental	Super Porcelain AAA	—	Grind with #800 SiC
Indirect composite	Tokuyama Dental	PEARLESTE	—	1)Grind with #1500 SiC 2)Sandblast (50um of Al_2O_3)
Zirconia	TOSO	TZ-3Y-E	Yttria stabilized Zirconia (Yttria 3%)	1)Grind with #120 SiC 2)Sandblast (50um of Al_2O_3)

Resin Cement: ESTECEM Ⅱ (without light-cure)

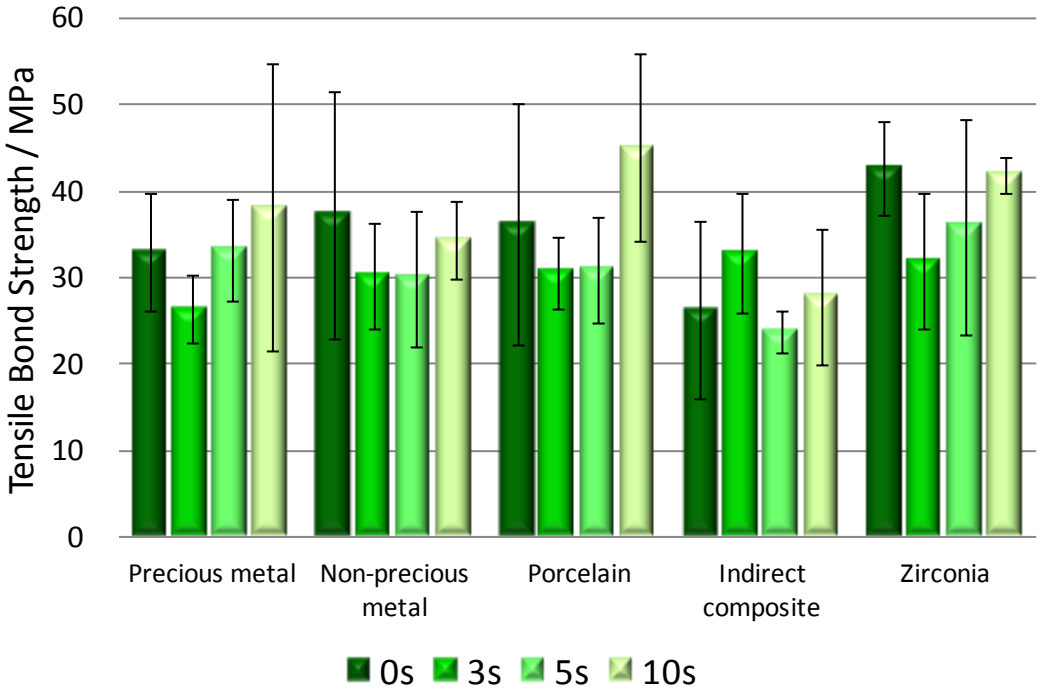


Fig.16 Tensile bond strength on prosthetic materials with varying waiting time

5. Features - Reliability

5.1 Compatibility with all etching protocols

5.1.1 Bond Strength

Tensile bond strength of TOKUYAMA UNIVERSAL BOND on dentin and enamel in the self-etch mode and total-etch mode was compared between competitive universal type products listed in *Table 5*. The result is shown in *Fig. 17–20*. In both self-etch mode and total-etch mode, TOKUYAMA UNIVERSAL BOND showed a bond strength that was equal or higher than the strength of competitors' products.

Table 5 Tested universal type bonding materials

Product Name	Manufacturer	Abbreviation
TOKUYAMA UNIVERSAL BOND	Tokuyama Dental	UB
TOKUYAMA BOND FORCE II	Tokuyama Dental	BF
Scotchbond Universal Adhesive	3M ESPE	SU
Adhese Universal	Ivoclar Vivadent	AU
Futura Bond U	Voco	FU
i Bond Universal	Heraeus Kulzer	IB
All-Bond Universal	Bisco	AB
Clearfil Universal Bond	Kuraray Noritake Dental	CU
G-Premio Bond	GC	GP

Resin composite: ESTELITE Σ QUICK

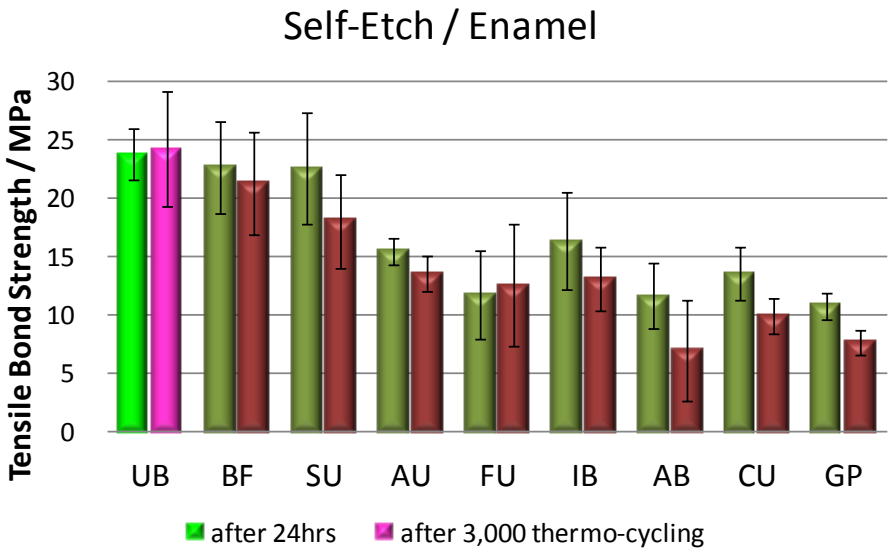


Fig.17 Tensile bond strength of universal adhesives in self-etch technique on enamel, before and after thermo-cycling.

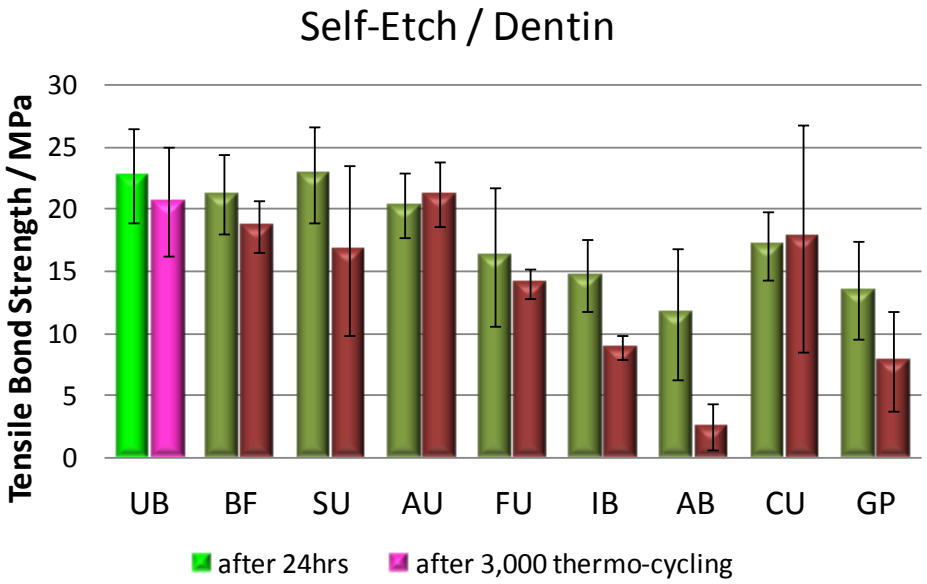


Fig.18 Tensile bond strength of universal adhesives in self-etch technique on dentin, before and after thermo-cycling.

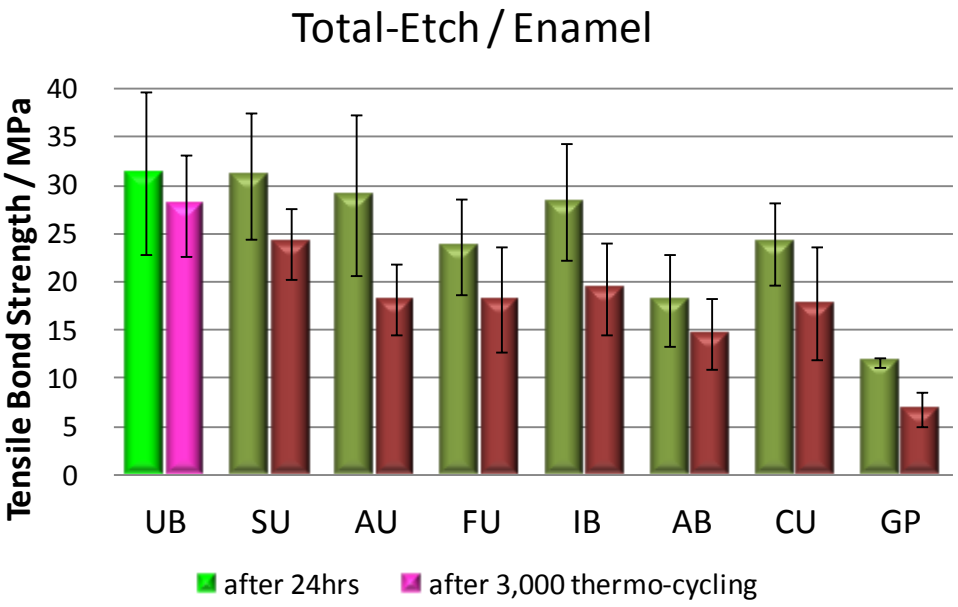


Fig.19 Tensile bond strength of universal adhesives in total-etch technique on enamel, before and after thermo-cycling.

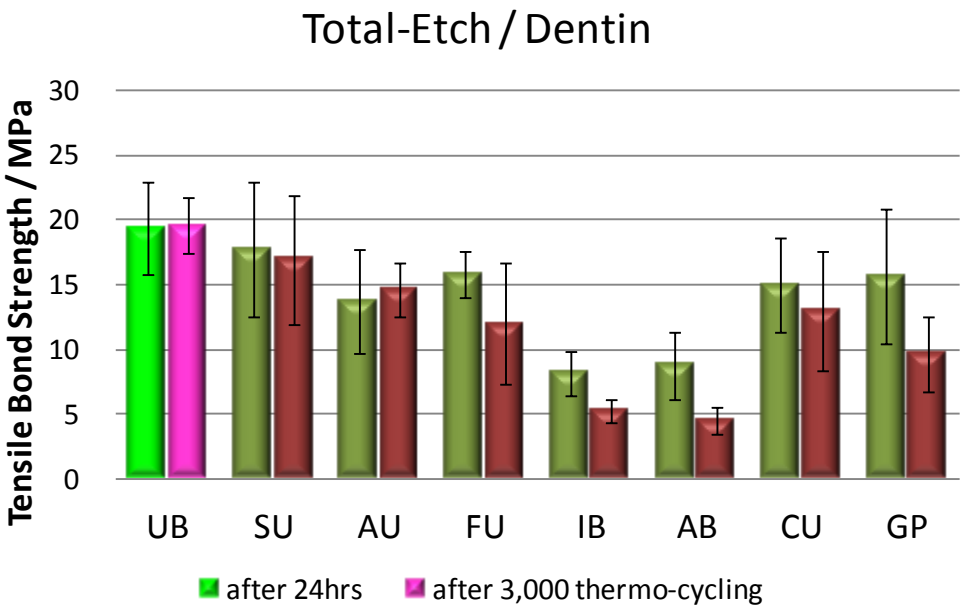


Fig.20 Tensile bond strength of universal adhesives in total-etch technique on dentin, before and after thermo-cycling.

Fig. 21, 22 show data[6)] generated by Dr. Miyazaki at Nihon University. The data show shear bond strength at 24hours. TOKUYAMA UNIVERSAL BOND was compared to competitive products in the self-etch mode and total-etch mode to dentin and enamel. The result of the shear test also showed that TOKUYAMA UNIVERSAL BOND had a good adhesion to tooth substance in the self-etch mode and total-etch mode.

Resin composite: CLEARFIL AP-X

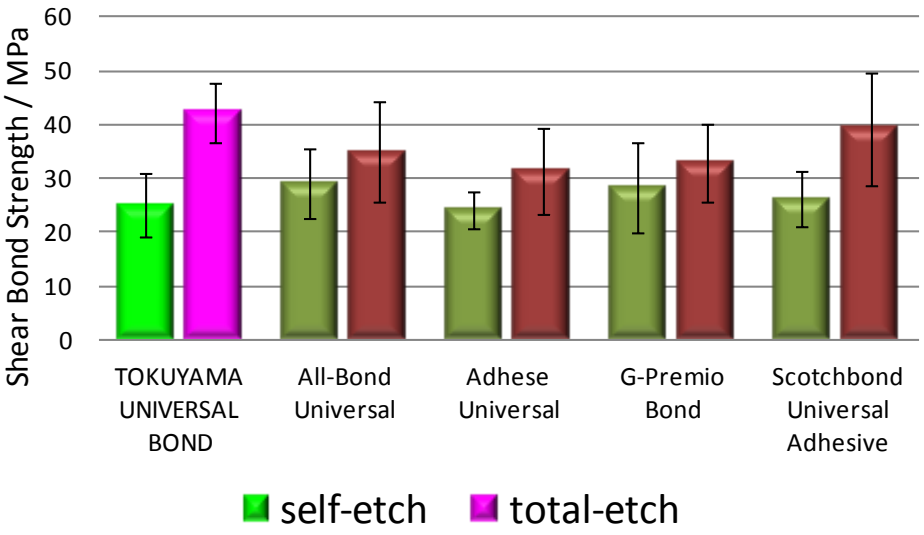


Fig.21 Shear bond strength of universal adhesives in self-etch and total-etch technique on enamel.

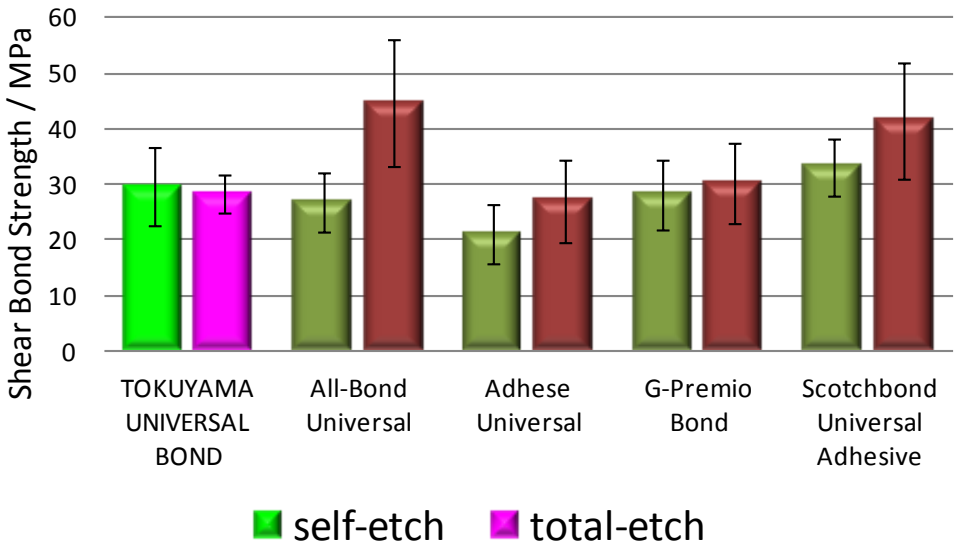


Fig.22 Shear bond strength of universal adhesives in self-etch and total-etch technique on dentin.

Fig.23 show data[7)] generated by Dr. Miyazaki at Nihon University. The data show shear bond strength at 24hours. TOKUYAMA UNIVERSAL BOND/ ESTECEM Ⅱ system was compared to Sctochbond universal Adhesive / RelyX Ultimate system in the self-etch mode and total-etch mode to dentin.

TOKUYAMA UNIVERSAL BOND/ESTECEM II system showed excellent adhesion with or without phosphate etching and photoirradiation to resin cement.

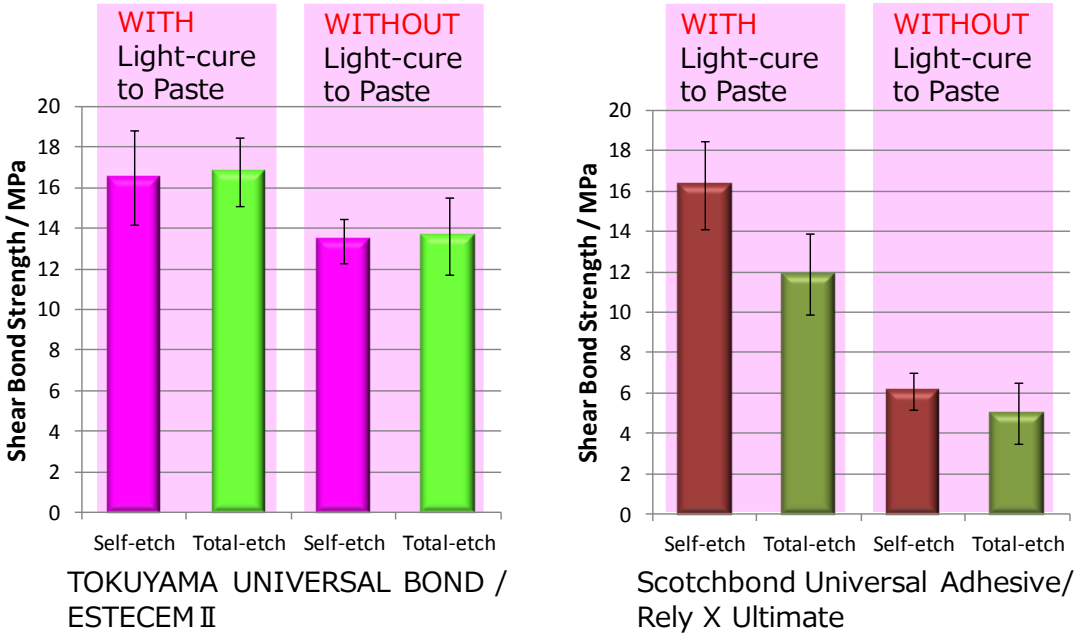


Fig.23 Shear bond strength to dentin in self-etch and total-etch technique w/wo light-curing.

5.1.2 Analysis of Adhesive Interface

[SEM images (Self-etch)]

In the self-etch mode, we performed SEM observation of adhesion interfaces of TOKUYAMA UNIVERSAL BOND to the enamel and dentin (FE-SEM XL30SFEG, PHILIPS). *Fig. 24, 25*. No gap between the enamel and dentin adhesive interfaces was seen and good adhesion to enamel and dentin as well as uniform and thin bonding layers were present. This result was based on formation of strong bonding layers on tooth substance surface to be bonded by polymerizing reaction products of the new 3D-SR monomer, an ingredient of TOKUYAMA UNIVERSAL BOND, and calcium in the tooth substance.

Resin composite: ESTELITE FLOW QUICK

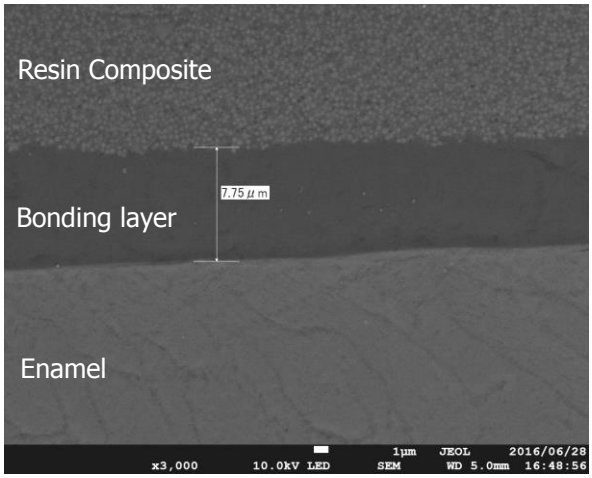


Fig. 24 Enamel (x 3,000)

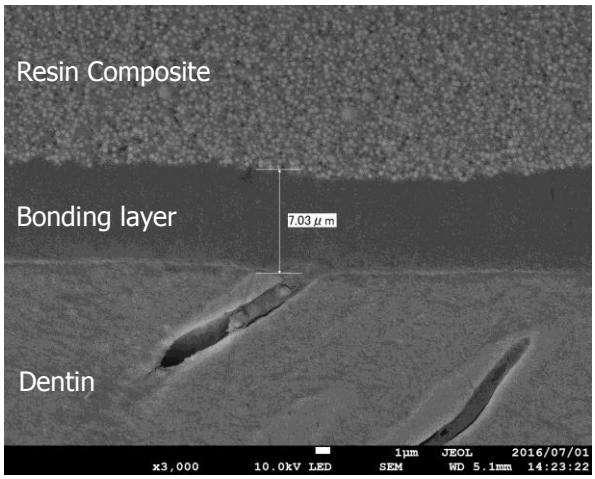


Fig. 25 Dentin (x 3,000)

[SEM images and micro-Raman spectroscopy analysis (Total-etch)]

We performed SEM observation and micro-Raman spectroscopy observation of tooth adhesive interface in the total-etch mode (FE-SEM XL30SFEG, PHILIPS). *Fig. 26*. There was no gap between the bonding layer and tooth, but a 1.5–3 μm layer that was considered to be a hybrid layer on dentin was observed.

Resin composite: ESTELITE FLOW QUICK

Etching condition：Etched 15 sec. with TOKUYAMA ETCHIN GEL HV and rinse 15 sec. with water, then dry with cotton pellets.

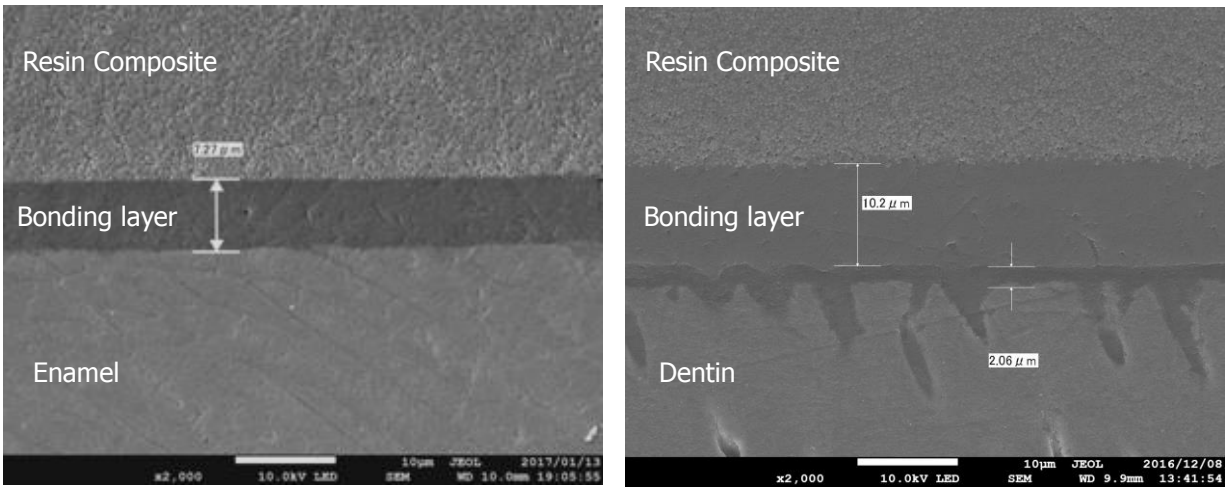


Fig. 26 Enamel and Dentin (x 2,000)

In the total-etch technique, there is a concern that durability may deteriorate because of tooth decalcification by phosphate etching and penetration gap between monomer ingredients of the bonding agent[8].

Using a micro-Raman spectroscopy, penetration of TOKUYAMA UNIVERSAL BOND into phosphate-etched dentin was measured. The measurement result is shown in *Fig. 27, 28*. The location where the peak of dentin Hydroxylapatite decreases is consistent with the one where the peak of bonding agent starts to be detected (line ①). The decrease in the peak of Hydroxylapatite and increase in the peak of monomer is transitional (line ② and ③). Therefore, it is estimated that the monomer penetrates into the phosphate-decalcified dentin part.

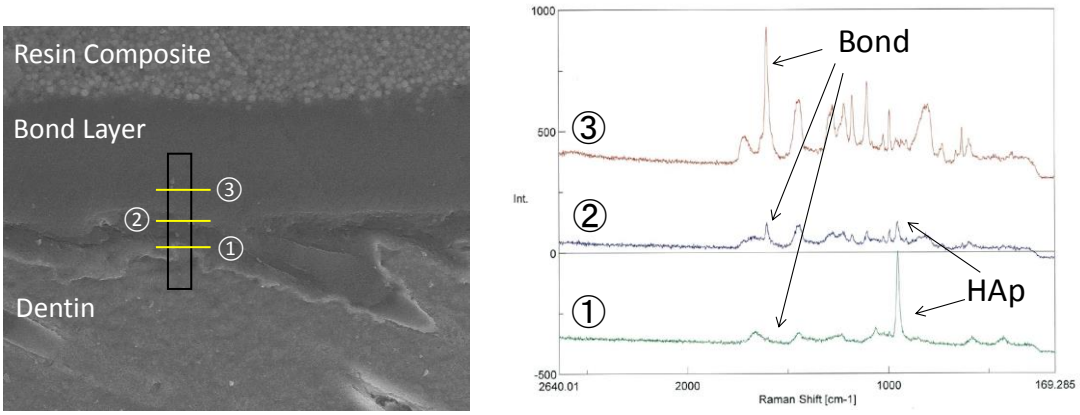


Fig. 27 Raman spectrum of TOKUYAMA UNIVERSAL BOND

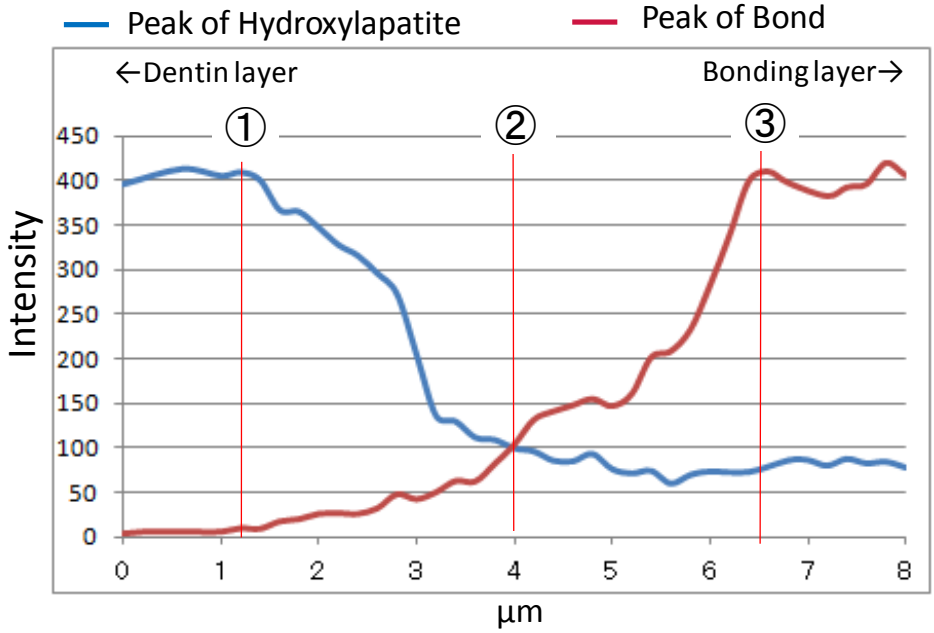


Fig. 28 Raman analysis of TOKUYAMA UNIVERSAL BOND

5.2 Applicability to direct and indirect restoration

-Compatibility with light-cured, self-cured and dual-cured composite materials-

TOKUYAMA UNIVERSAL BOND has been designed to be fully compatible with light-cured, self-cured and dual-cured composite materials. We evaluated tensile bond strength of TOKUYAMA UNIVERSAL BOND combined with resin materials that are commercially available.

[Light-curing resin composite]

We evaluated the bond strength when combined with each company's light-curing resin composite (universal, flowable). The result in *Fig. 29* and *30* showed an excellent bond strength for any resin composite.

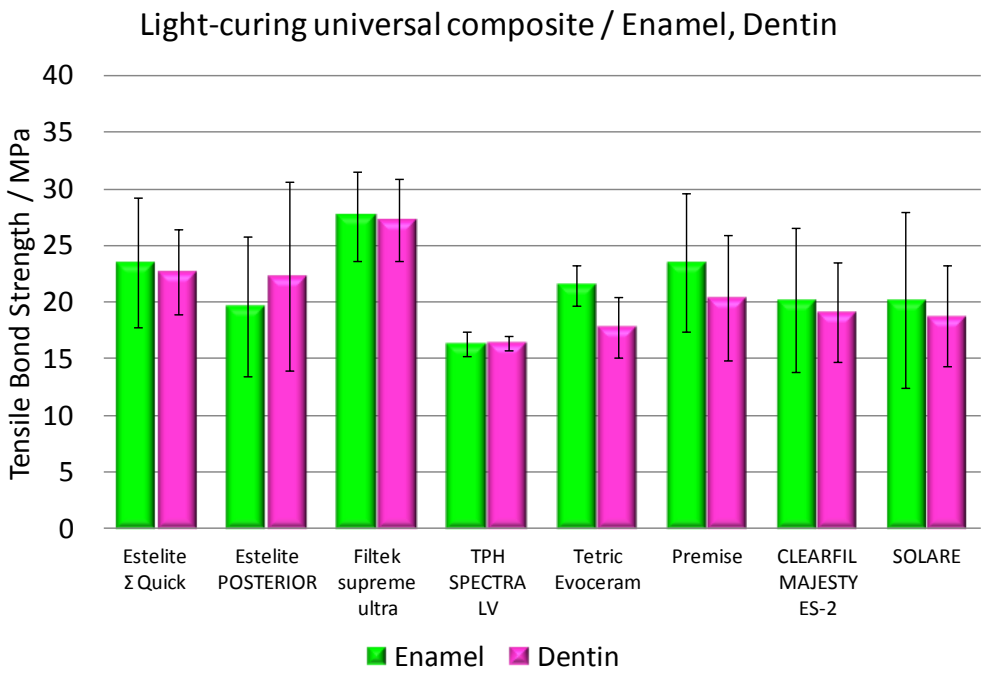


Fig.29 Tensile bond strength of TOKUYAMA UNIVRSAL BOND with various light-curing resin composite to enamel and dentin

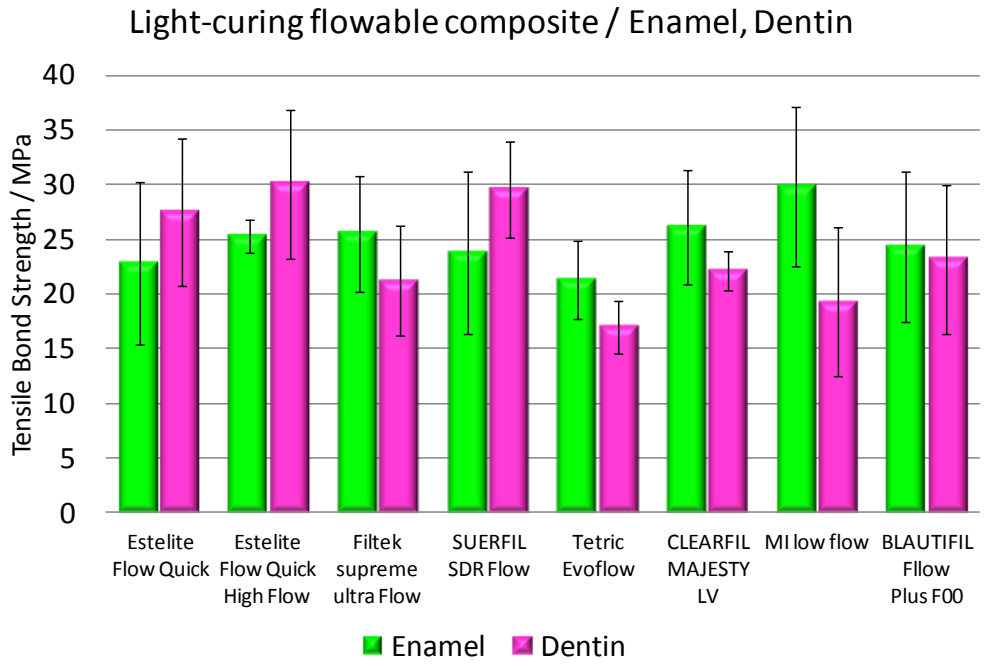


Fig.30 Tensile bond strength of TOKUYAMA UNIVRSAL BOND with various light-curing flowable resin composite to enamel and dentin

[Dual-cure resin composite (resin core material and resin cement)]

Using a commercially available dual-curing resin core or resin cement, bond strength was evaluated with or without photoirradiation. The result is shown in *Fig. 31*.

Bond strength was excellent for any resin core/resin cement. Furthermore, even without photoirradiation, it was equal to dual-curing bond strength with photoirradiation. Therefore, bond strength was excellent regardless of the curing conditions of materials.

Curing Mode:

Dual-cure; with light cure

Self-cure; without light cure

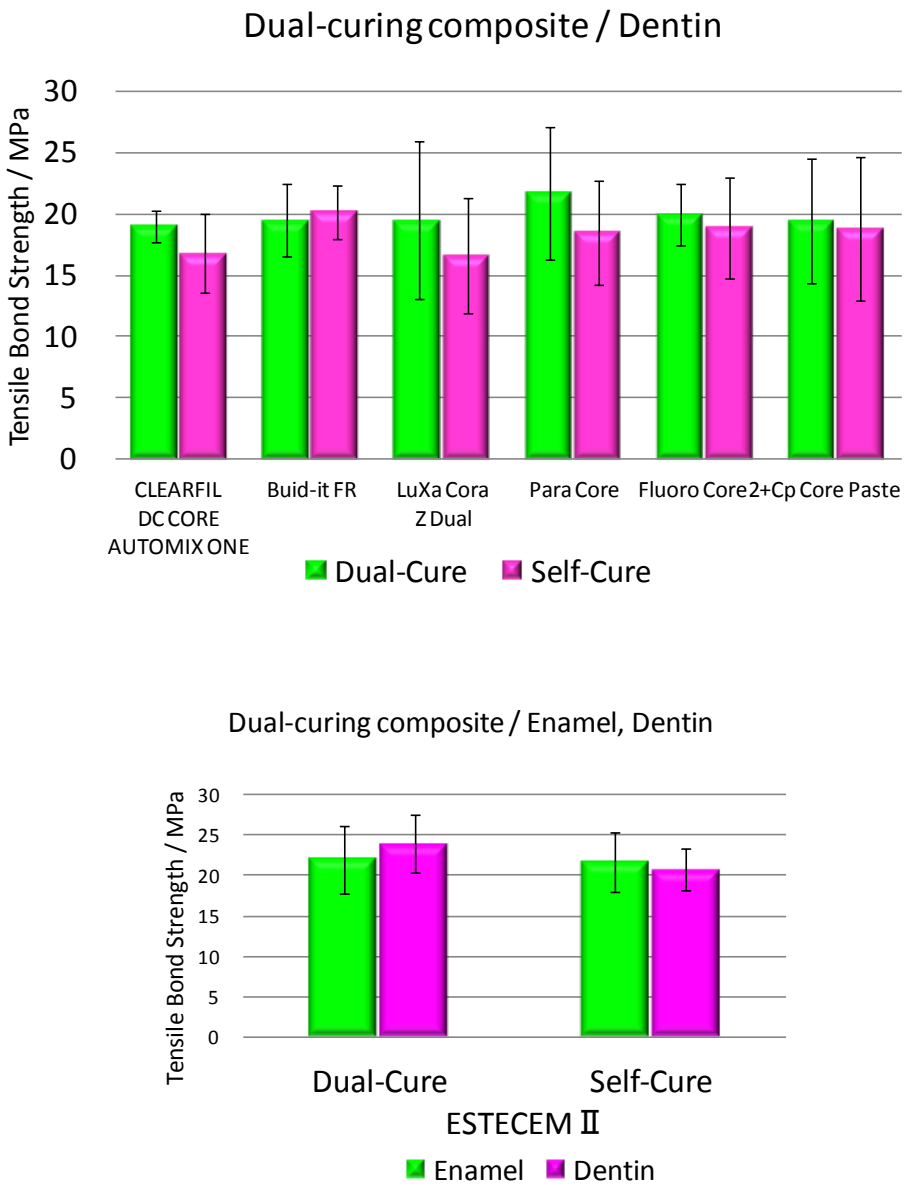


Fig.31 Tensile bond strength of TOKUYAMA UNIVRSAL BOND with various dual-curing resin composite to dentin

Fig.32 show data[9)] generated by Dr. Sano at Hokkaido University. The data show micro-tensile bond strength at 24hours. TOKUYAMA UNIVERSAL BOND was compared to Scotchbond Universal Adhesive with/without right irradiation mode to dentin.

TOKUYAMA UNIVERSAL BOND showed high bond strength in combination with light-curing resin composite. Furthermore, adhesion to tooth substance was excellent even without photoirradiation to resin cement, when using a dual-curing type one.

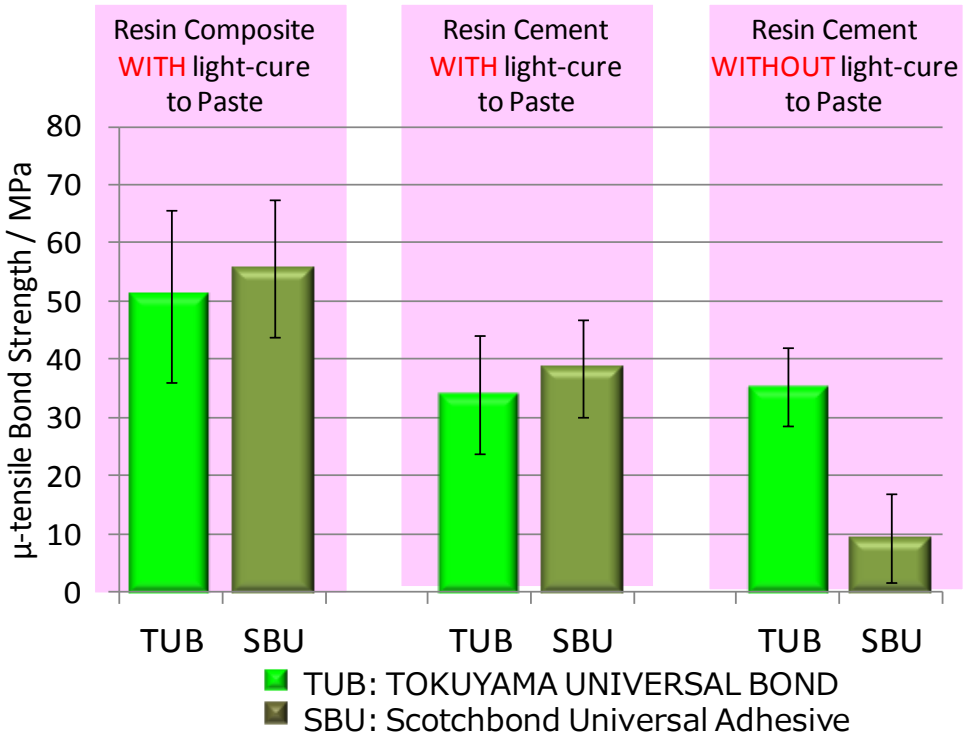


Fig.32 Micro-tensile bond strength on dentin with/without light-curing

5.2.1 Direct restoration

[Bond Strength]

The micro-tensile bond strength of TOKUYAMA UNIVERSAL BOND on dentin was measured. The result is shown in *Fig. 33*.

According to the micro-tensile method, bond strength of TOKUYAMA UNIVERSAL BOND was good.

・Resin composite : ESTELITE POSTERIOR
・Sample :Beam-shape 1mm square

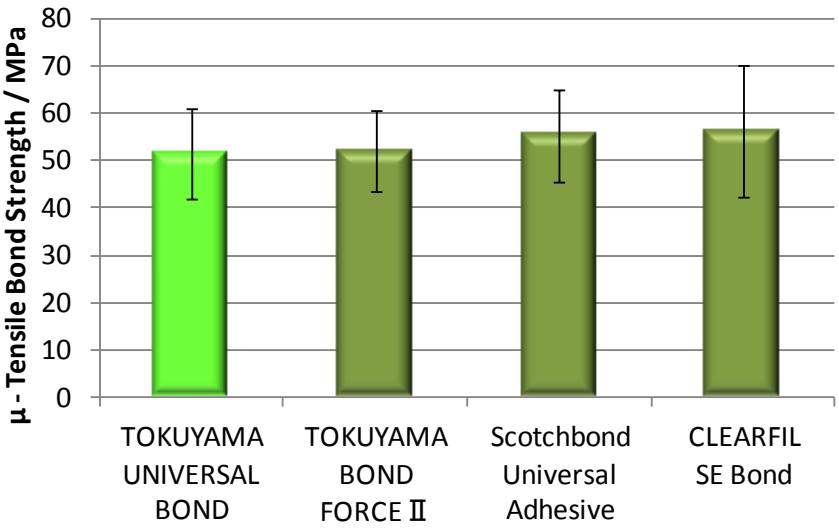


Fig.33 μ -tensile bond strength of TOKUYAMA UNIVERSAL BOND and commercial products on dentin

Using a light-curing resin composite, a shear bonding test was performed to examine the bond strength of TOKUYAMA UNIVERSAL BOND on tooth substance immediately after light-curing. The result is shown in *Fig. 34*. The result of the shear bonding test performed one minute after curing resin composite showed that the bond strength was comparable with that measured 24 hours later. A feature of TOKUYAMA UNIVERSAL BOND is that curing of bonding layers progresses once chemical polymerization is initiated rapidly after it comes into contact with resin materials after air blow with borate catalyst, a highly active polymerization catalyst. Therefore, a high bond strength can be obtained immediately after curing, even without photoirradiation of the bond.

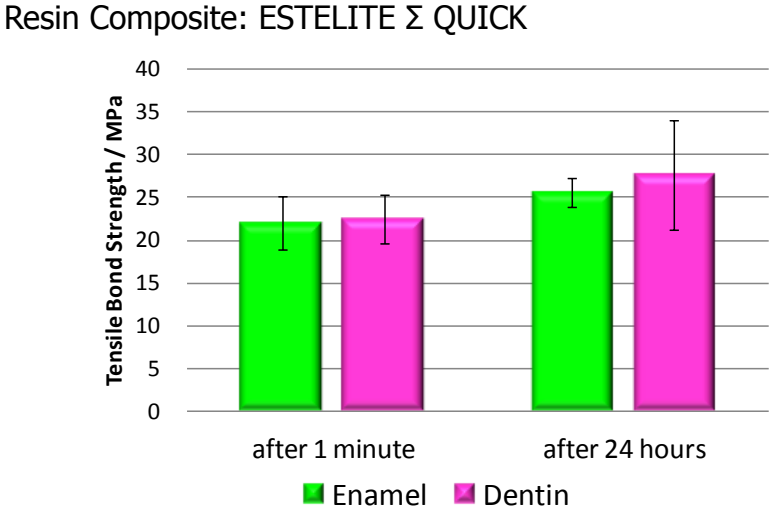


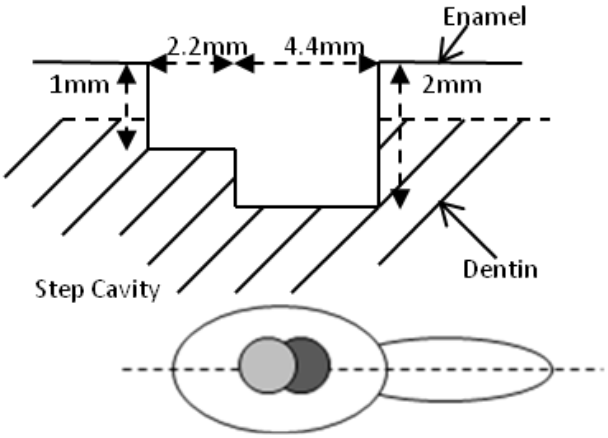
Fig.34 Shear bond strength immediately after curing

[Cavity Adaptation]

Cavity adaptation of TOKUYAMA UNIVERSAL BOND was compared with that of Scotchbond Universal Adhesive (3M ESPE), as shown in *Fig.35*. Gap free bonding layer and uniform thickness were observed on the cavity floors, corners, and edges. This demonstrates excellent cavity adaptation of TOKUYAMA UNIVERSAL BOND.

Test method:

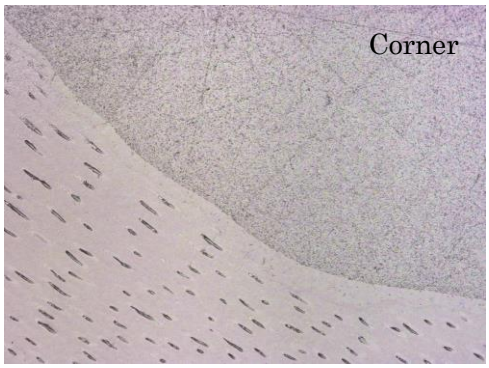
1) Create simulated cavities in the form of steps on the labial aspect of an extracted bovine first anterior tooth.

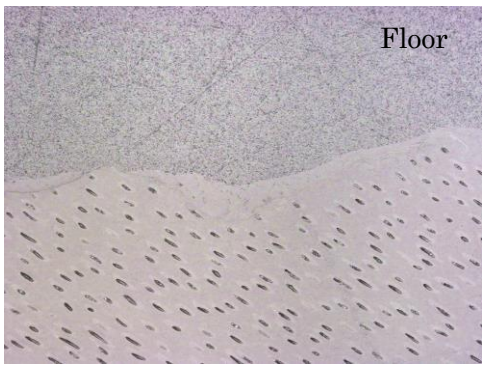


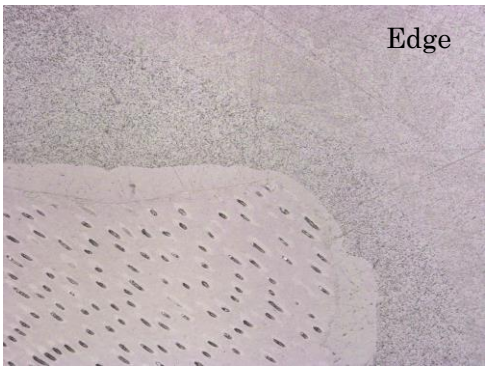
2) Apply TOKUYAMA UNIVERSAL BOND and air dry. When applying Scotchbond Universal Adhesive, reb 20 secs before air drying. Light cure for 10 secs.
3) Fill with resin composite in layers (Estelite Flow Quick HF (EFQH)+ EsteliteΣQuick (ESQ) / Tokuyama Dental Corp)
4) Cut perpendicular to the adhesion surface using a diamond cutter
5) Polish the cut surface using diamond paste (final: 0.25 μm)
6) Observe with laser microscope (VK9700, Keyence Corp.)

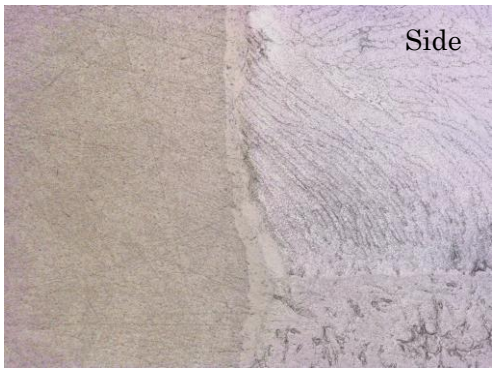
TOKUYAMA UNIVERSAL BOND (x 200)

TOKUYAMA UNIVERSAL BOND (x 1,000)



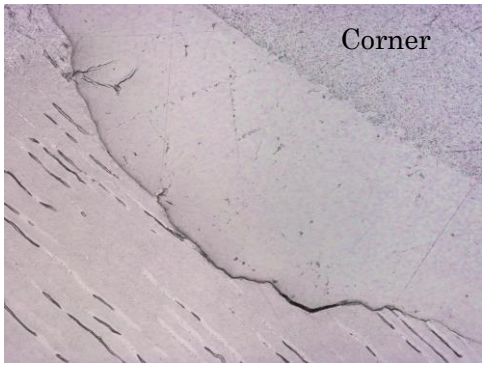


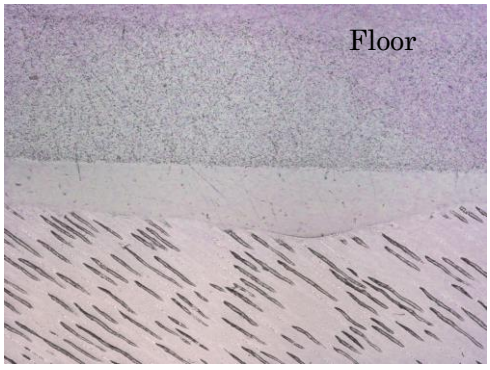




Scotchbond Universal Adhesive (x 200)

Scotchbond Universal Adhesive (x 1,000)





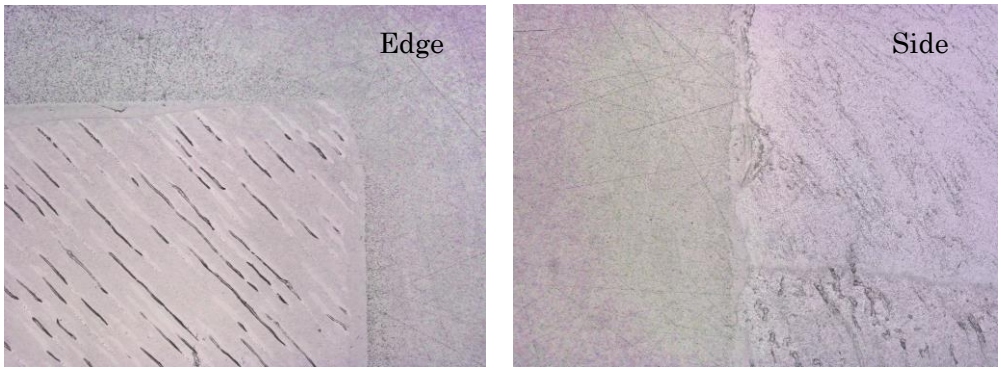


Fig. 35 Cavity adaptation

[Marginal Leakage]

The marginal leakage of TOKUYAMA UNIVERSAL BOND was evaluated *Table 6*. Pigment penetration was not observed, and TOKUYAMA UNIVERSAL BOND 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 UNIVERSAL BOND and air dry. When testing Scotchbond Universal Adhesive, apply and wait for 20 seconds, and air dry. Then light cure for 10 seconds.
3) Fill with resin composite (Estelite Flow Quick HF + Estelite Σ Quick, 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 6 Marginal leakage

Bonding Agent	Marginal leakage (n=10)									
TOKUYAMA UNIVERSAL BOND	—	—	—	—	—	—	—	—	—	—
Scotchbond Universal Adhesive	—	—	—	—	—	—	+	—	+	—

– : no pigment penetration + : penetration into enamel

++ : penetration into dentin +++ : penetration into cavity floor

5.2.2 Intraoral repair

TOKUYAMA UNIVERSAL BOND can be used as a bond for intraoral repair, as it is applicable not only to tooth substance but silica-based, zirconia-based and metallic restorations. We performed a tensile bonding test and shear bonding test to examine the bond strength of TOKUYAMA UNIVERSAL BOND when bonded to various prosthetic materials and to compare the strength with that of competitors' products.

[Bond Strength]

Bond strength was evaluated by a tensile bonding test. *Table 7* and *8* show materials and pre-treatment methods used for the test and competitors' universal bond products used for comparison, while *Fig. 36–40* show the test result.

TOKUYAMA UNIVERSAL BOND showed stable and high initial bond strength and durability when bonded to various materials. In particular, a comparison with competitors' universal bond products showed that the bond to precious metal and ceramics has excellent durability.

Resin Composite: ESTELITE Σ QUICK

Table 7 Tested prosthetic materials and pre-treatment method

	Manufacturer	Product name	Composition	Pre-treatment
Precious metal	Tokuyama Dental	CASTMASTER12S	Au12/Pd20/Ag54 /Cu12/other2	1)Grind with #1500 SiC 2)Sandblast (50um of Al_2O_3)
Non-precious metal	Tokuyama Dental	ICROME	Co57.8/Cr31.6/Mo5.6 /other5	1)Grind with #1500 SiC 2)Sandblast (50um of Al_2O_3)
Ceramics (Silica-base ceramics)	Kuraray Noritake Dental	Super Porcelain AAA	—	Grind with #800 SiC
Indirect composite	Tokuyama Dental	PEARLESTE	—	1)Grind with #1500 SiC 2)Sandblast (50um of Al_2O_3)
Zirconia	TOSO	TZ-3Y-E	Yttria stabilized Zirconia (Yttria 3%)	1)Grind with #120 SiC 2)Sandblast (50um of Al_2O_3)

Table 8 Tested universal type bonding materials

Product Name	Manufacturer	Abbreviation
TOKUYAMA UNIVERSAL BOND	Tokuyama Dental	UB
Scotchbond Universal Adhesive	3M ESPE	SU
Futura Bond U	Voco	FU
i Bond Universal	Heraeus Kulzer	IB
All-Bond Universal	Bisco	AB
Clearfil Universal Bond	Kuraray Noritake Dental	CU
G-Premio Bond	GC	GP

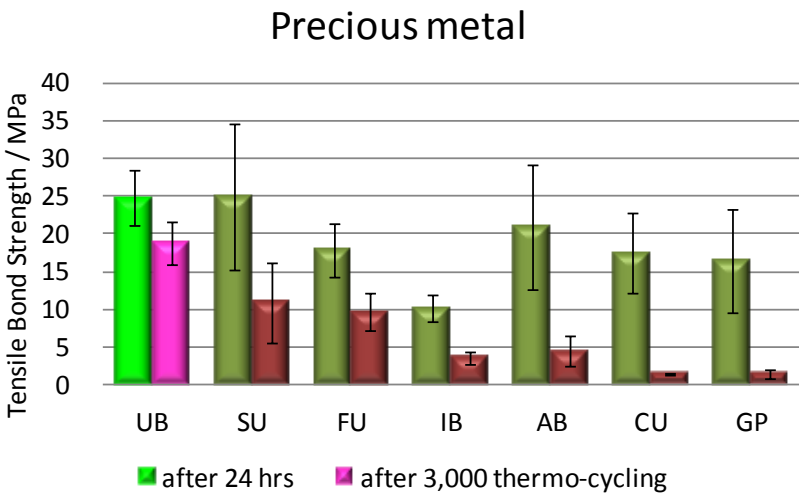


Fig.36 Tensile bond strength of universal adhesives on precious metal, before and after thermo-cycling.

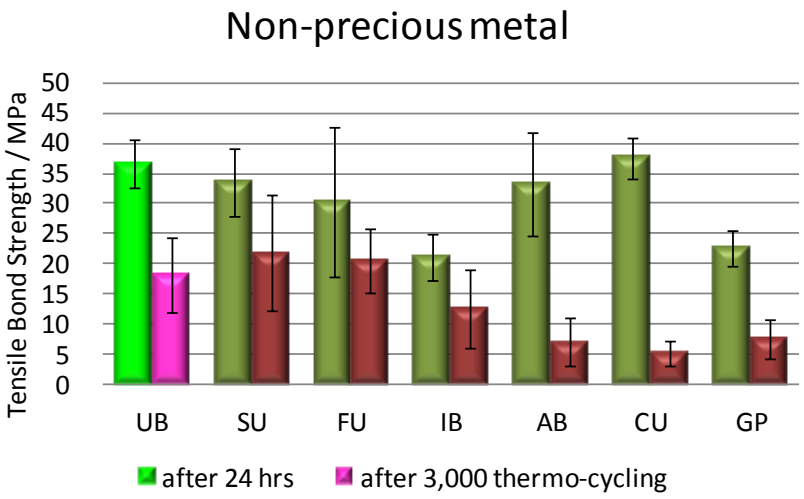


Fig.37 Tensile bond strength of universal adhesives on non-precious metal, before and after thermo-cycling.

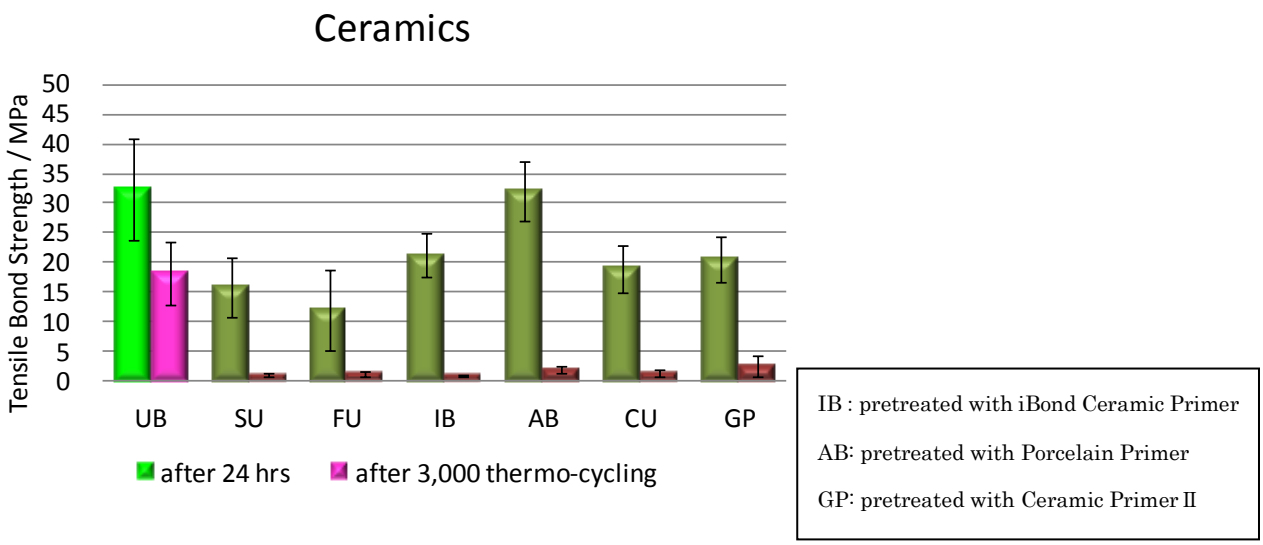


Fig.38 Tensile bond strength of universal adhesives on ceramics, before and after thermo-cycling.

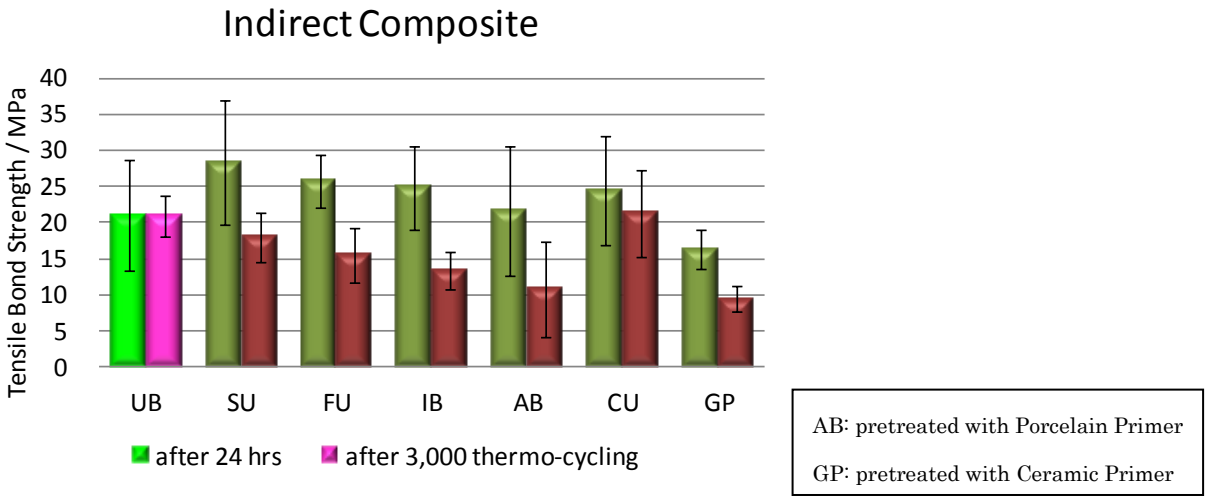


Fig.39 Tensile bond strength of universal adhesives on indirect composite, before and after thermo-cycling.

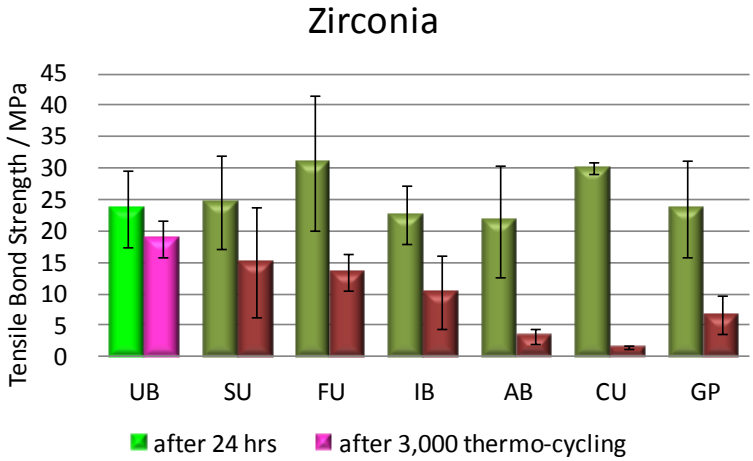


Fig. 40 Tensile bond strength of universal adhesives on zirconia, before and after thermo-cycling.

Table 9 and *Fig41 - 44* show data[10] generated by Dr. Miyazaki at Nihon University. The data show shear bond strength at 24hours and after thermo-cycle. TOKUYAMA UNIVERSAL BOND was compared to competitive products to various prosthetic materials. The result of the shear bonding test also showed that TOKUYAMA UNIVERSAL BOND has an excellent bond strength as well as initial bond strength and durability when bonded to various prosthetic materials.

Table 9 Tested prosthetic materials

	Product Name	Manufacturer
Lithium Disilicate	IPS e-max press	Ivoclar Vivadent
Zirconia	-	Japan Fine Ceramics
Alumina	-	Japan Fine Ceramics
Precious metal	SuperCrystal KP5	Yamakin

Sandblasting method: Silica sandblasting (Cojet) / Alumina sandblasting

Resin Composite：CLEARFIL AP-X

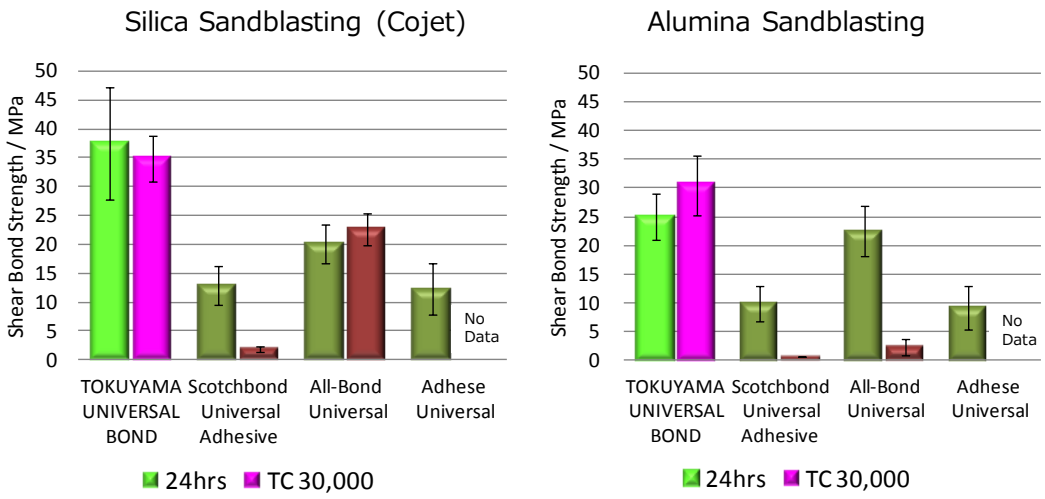


Fig.41 Shear bond strength of universal adhesives on Lithium disilicate (IPS e-max press), before and after thermo-cycling.

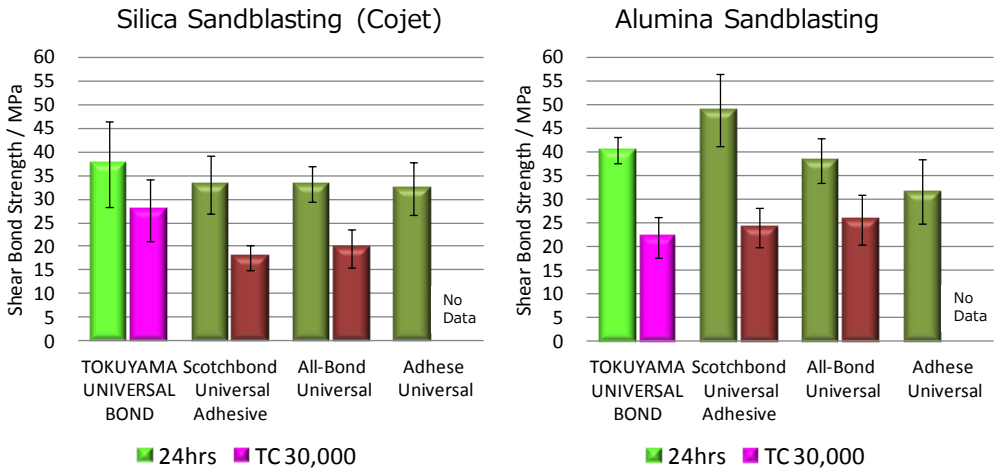


Fig. 42 Shear bond strength of universal adhesives on Zirconia, before and after thermo-cycling.

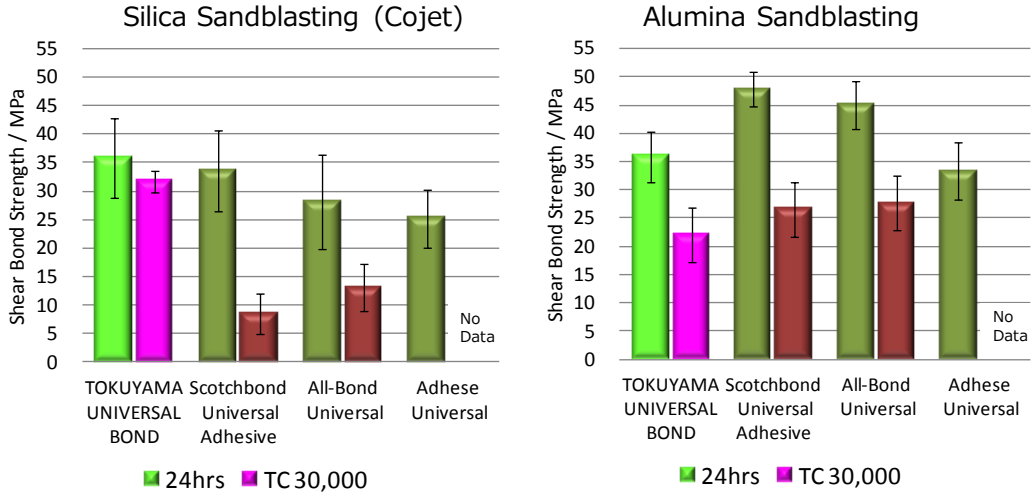


Fig. 43 Shear bond strength of universal adhesives on Alumina, before and after thermo-cycling.

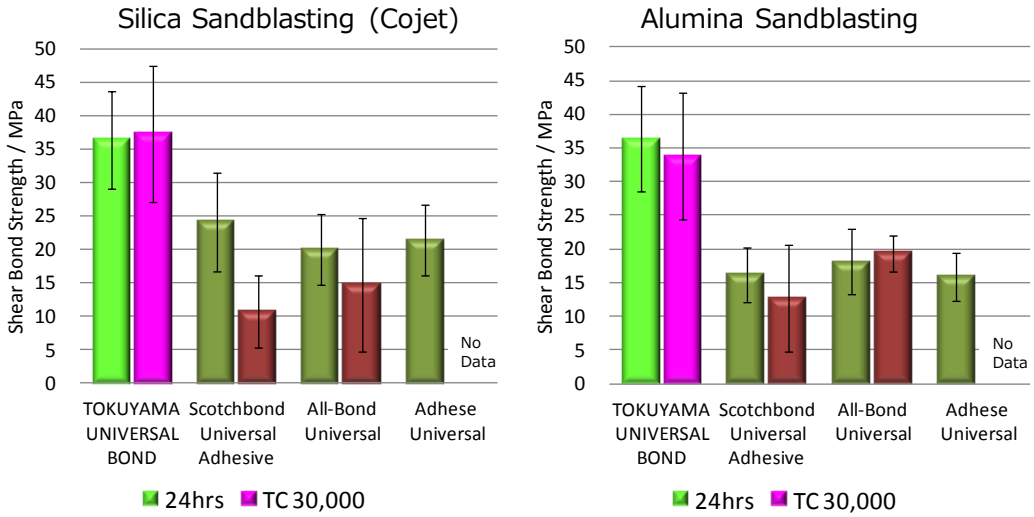


Fig. 44 Shear bond strength of universal adhesives on precious metal, before and after thermo-cycling.

5.2.3 Indirect restoration

TOKUYAMA UNIVERSAL BOND can be used for indirect restoration, as it can be combined with dual-curing or self-curing composite materials.

[Bond Strength]

The bond strength of ESTECEM II, a resin cement using TOKUYAMA UNIVERSAL BOND, on tooth substance was compared with that of competitors' resin cement system by a tensile bonding test. Competitive products used for comparison are listed in *Table 10*, while the evaluation result is shown in *Fig. 45* and *46*. Both initial bond strength and durability (3,000 times) was equal to or higher than those of competitors' products.

Curing condition: Self-cure (without light irradiation)

Table 10 Tested resin cement system

Adhesion for tooth	Resin Cement	Manufacturer	Abbreviation
TOKUYAMA UNIVERSAL BOND	ESTECEM Ⅱ	Tokuyama Dental	UB
ESTELINK	ESTECEM	Tokuyama Dental	EL
Scotchbond Universal Adhesive	Rely X Ultimate	3M ESPE	SU
Opti Bond XTR	NX3	Kerr	OX
All-Bond Universal	Duo-Link	Bisco	AB
ED Primer	Panavia F 2.0	Kuraray Noritake	EP
Multilink Primer	Multilink Automix	Ivoclar Vivadent	MP

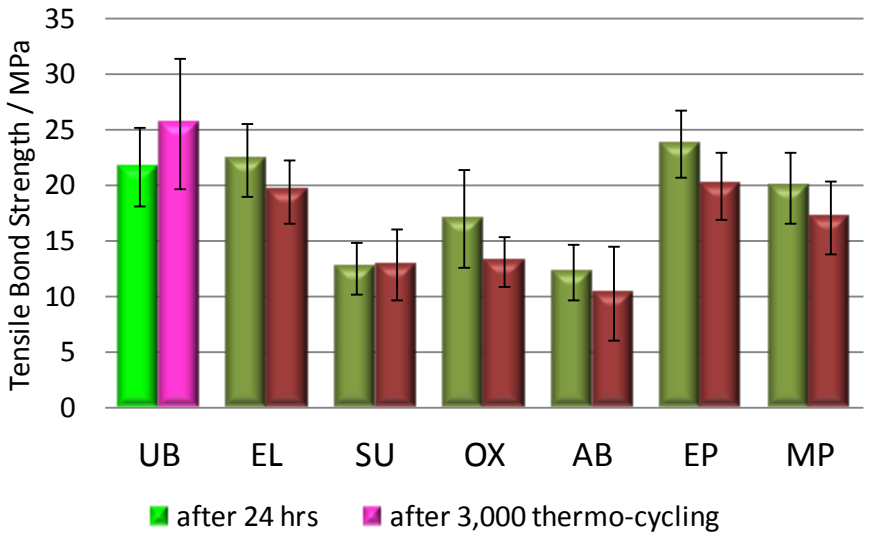


Fig. 45 Tensile bond strength of resin cement systems on enamel, before and after thermo-cycling.

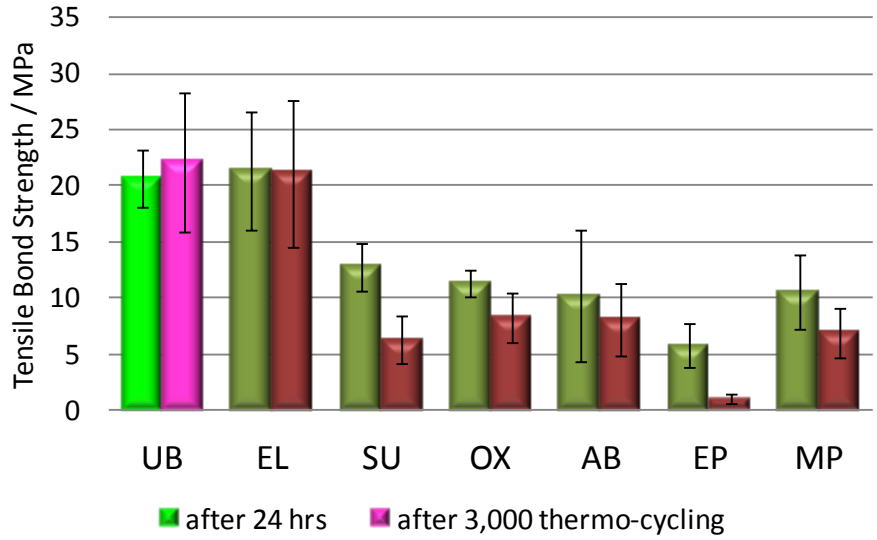


Fig. 46 Tensile bond strength of resin cement systems on dentin, before and after thermo-cycling.

5.3 Adhesion to Various Indirect Substrates

-Used as a primer for silica-based, zirconia based and metallic restorations-

TOKUYAMA UNIVERSAL BOND can be used as a primer for silica-based, zirconia-based and metallic restorations without the use of separate primers. Therefore, treatment of tooth substance and prosthetics can be completed with TOKUYAMA UNIVERSAL BOND for cementation and no other bonds or primers are required.

[Bond strength]

The tensile bond strength of TOKUYAMA UNIVERSAL BOND on various prosthetics for indirect restoration was evaluated. *Fig. 47* and *48* show the bond strength on various prosthetics listed in *Table 11*.

TOKUYAMA UNIVERSAL BOND showed excellent bond strength on any material. These results are based on reaction of adhesive monomer (MTU-6, New 3D-SR monomer, γ-MPTES), an ingredient of TOKUYAMA UNIVERSAL BOND, and prosth

Resin Cement: ESTECEM Ⅱ

Curing condition: Self-cure (without light irradiation)

Table 11 Tested prosthetic materials and pre-treatment method

	Manufacturer	Product name (abbr.)	Composition	Pre-treatment
Precious metal	Tokuyama Dental	CASTMASTER12S (12S)	Au12/Pd20/Ag54/Cu12/other2	1)Grind with #1500 SiC 2)Sandblast (50um of Al_2O_3)
	GC	Casting Gold M.C. TYPEⅣ (CG4)	Au70/Pd3/Ag8/Cu16/Pt2/other1	
		Casting Gold M.C. TYPEⅢ (CG3)	Au75/Pd3/Ag5/Cu16/other1	
		Casting Gold M.C. TYPEⅡ (CG2)	Au76/Pd2/Ag7/Cu14/other1	
	DENTSPLY SANKIN	MIRO BRIGHT (MB)	Ag72/Zn13/Sn9/In6	
		SUNSILVER CB (SS)	Ag77/Zn10/In7/Cu5	
Non-precious metal	Tokuyama Dental	ICROME (IC)	Co57.8/Cr31.6/Mo5.6/other5	1)Grind with #1500 SiC 2)Sandblast (50um of Al_2O_3)
		TITADENT (TI)	Ti 99.5%以上	
		TITADENTⅡ (TI2)	Ti90/Al16/V4	
Ceramics (Silica-base ceramics)	Kuraray Noritake Dental	Super Porcelain AAA (SP)	—	1)Grind with #800 SiC
	Ivoclar vivadent	IPS Empress (IE)	—	
	Ivoclar vivadent	IPS e.max CAD (EC)	—	
Indirect composite	SHOHU	CERAMAGE (CE)	—	1)Grind with #1500 SiC 2)Sandblast (50um of Al_2O_3)
	Kuraray Noritake Dental	ESTENIA C&B (ES)	—	
CAD/CAM BLOCK	VITA	Enamic (EM)	—	1)Grind with #1500 SiC 2)Sandblast (50um of Al_2O_3)
	DENTSPLY SANKIN	KZR-CAD HR2 (KH)	—	
Zirconia	TOSO	TZ-3Y-E (TZ)	Yttria stabilized Zirconia (Yttria 3%)	1)Grind with #120 SiC 2)Sandblast (50um of Al_2O_3)
	3M ESPE	LAVA Zirconia(LZ)	—	
	Kuraray Noritake Dental	KATANA Zirconia(KZ)	—	

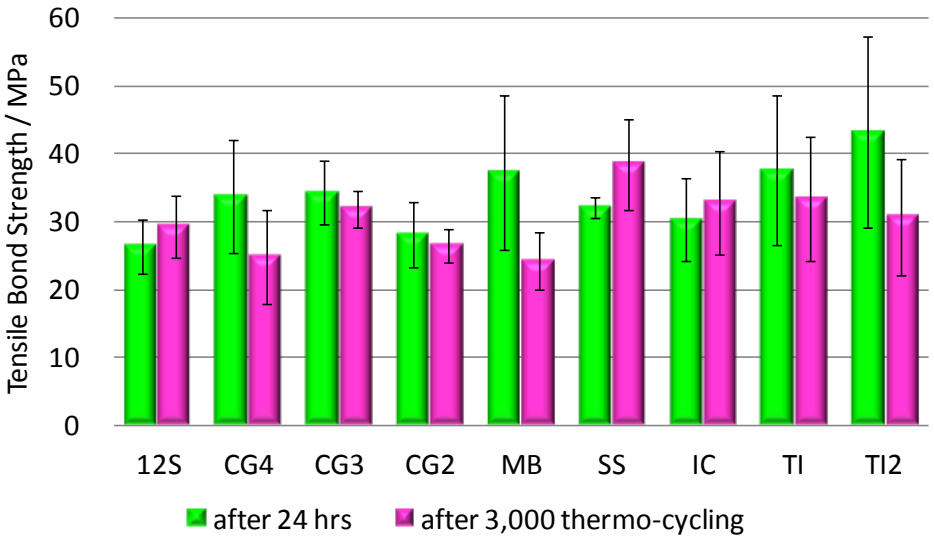


Fig.47 Tensile bond strength of TOKUYAMA UNIVERSAL BOND on metal

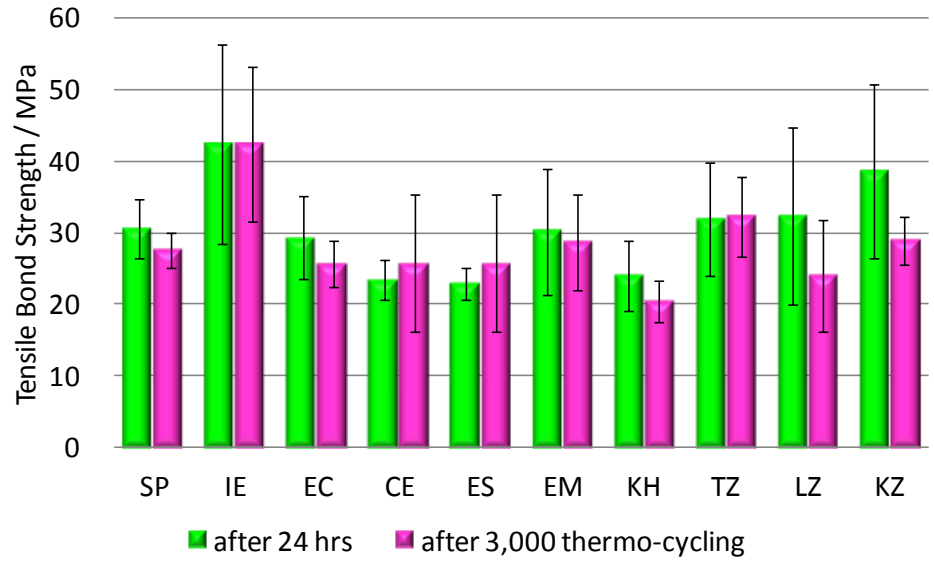


Fig.48 Tensile bond strength of TOKUYAMA UNIVERSAL BOND on ceramics, indirect resin, CAD/CAM block and zirconia

The bond strength of TOKUYAMA UNIVERSAL BOND was compared with that of competitors' prosthetic primers. Prosthetic primers used for comparison are listed in *Table 12 and 13*, while the evaluation result is shown in *Fig. 49–53*.

TOKUYAMA UNIVERSAL BOND showed stable and high initial bond strength and durability when bonded to various materials and they were also excellent when compared with competitors' products.

Table 12 Tested prosthetic materials and pre-treatment method

	Manufacturer	Product name	Composition	Pre-treatment
Precious metal	Tokuyama Dental	CASTMASTER12S	Au12/Pd20/Ag54/Cu12/other2	1)Grind with #1500 SiC 2)Sandblast (50um of Al_2O_3)
Non-precious metal	Tokuyama Dental	ICROME	Co57.8/Cr31.6/Mo5.6/other5	1)Grind with #1500 SiC 2)Sandblast (50um of Al_2O_3)
Ceramics (Silica-base ceramics)	Kuraray Noritake Dental	Super Porcelain AAA	—	1)Grind with #800 SiC
Indirect composite	Tokuyama Dental	PEARLESTE	—	1)Grind with #1500 SiC 2)Sandblast (50um of Al_2O_3)
Zirconia	TOSO	TZ-3Y-E	Yttria stabilized Zirconia (Yttria 3%)	1)Grind with #120 SiC 2)Sandblast (50um of Al_2O_3)

Table 13 Tested primers for prosthetic materials

Manufacturer		**Tokuyama Dental**		**3M ESPE**	**Ivoclar vivadent**	**Kuraray Noritake Dental**	**SHOFU**	**Bisco**
Resin Cement		ESTECEM Ⅱ	ESTECEM	RelyX Ultimate	Multilink Automix	Panavia V5	ResiCem	DUO-LINK
Prosthesis	Precious metal	TOKUYAMA UNIVERSAL BOND (UB)	TOKUYAMA UNIVERSAL PRIMER (UP)	Scotchbond Universal Adhesive (SU)	Monobond Plus (MP)	Clearfil Ceramic Primer Plus (CC)	Metallink (ML)	Z-Prime Plus (ZP)
	Non-precious metal							
	Ceramics						Porcelain Primer (PP)	Porcelain Primer (PP2)
	Indirect composite							Z-Prime Plus (ZP)
	Zirconia / Alumina						AZ Primer (AP)	

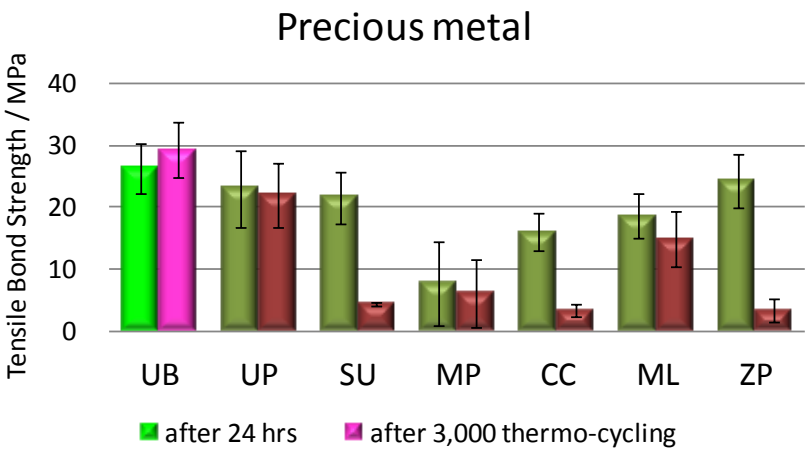


Fig.49 Tensile bond strength of TOKUYAMA UNIVERSAL BOND and primers on precious metal

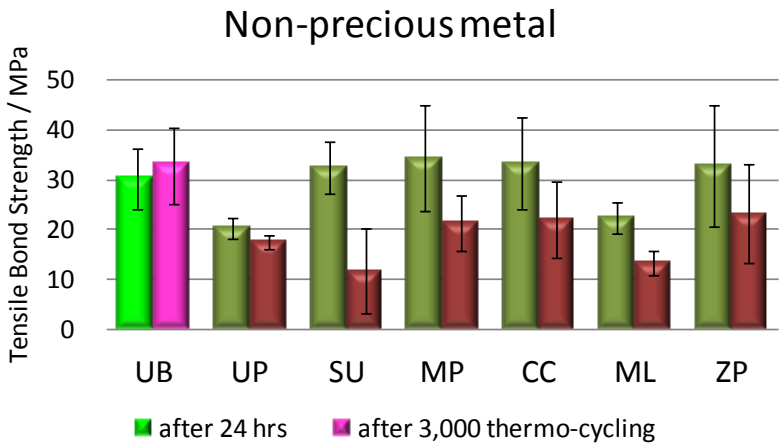


Fig.50 Tensile bond strength of TOKUYAMA UNIVERSAL BOND and primers on non-precious metal

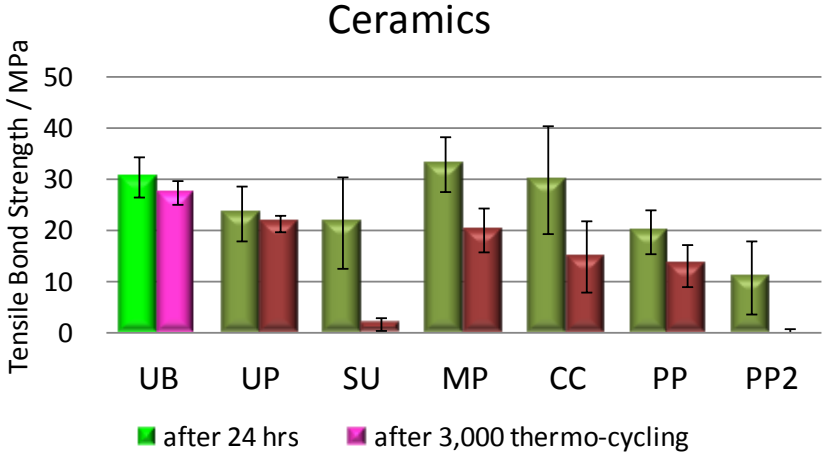


Fig.51 Tensile bond strength of TOKUYAMA UNIVERSAL BOND and primers on ceramics

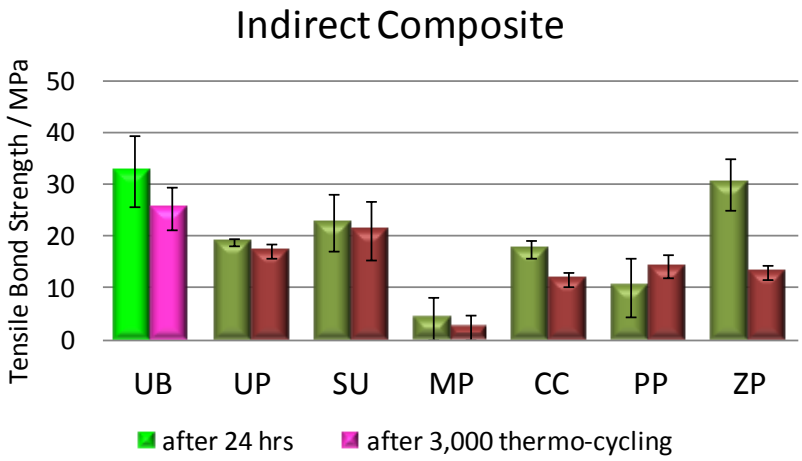


Fig.52 Tensile bond strength of TOKUYAMA UNIVERSAL BOND and primers on indirect composite

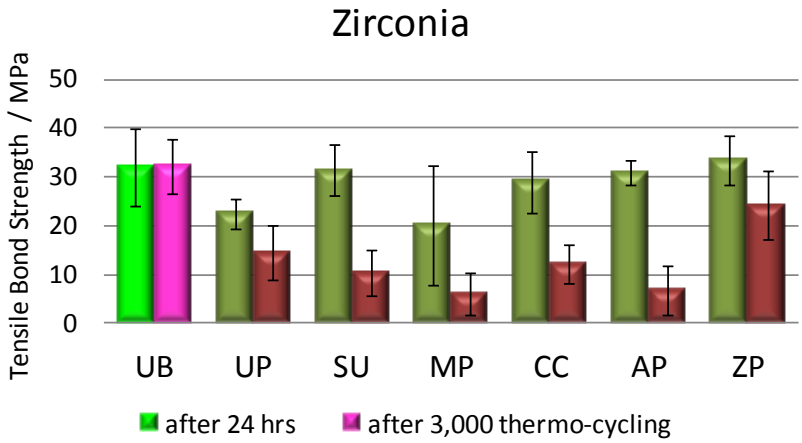


Fig.53 Tensile bond strength of TOKUYAMA UNIVERSAL BOND and primers on zirconia

Compatibility of TOKUYAMA UNIVERSAL BOND with competitors' resin cements was evaluated, when it was used as a pre-treatment agent for prosthetics.

Fig. 54 shows the evaluation result of bond strength on prosthetics using each competitor's resin cement. Resin cement was chemically cured.

Sufficient bond strength on various materials was obtained in combination with any of the cements.

Primer: TOKUYAMA UNIVERSAL BOND
Curing condition of Resin cement: Self-cure (without light irradiation)

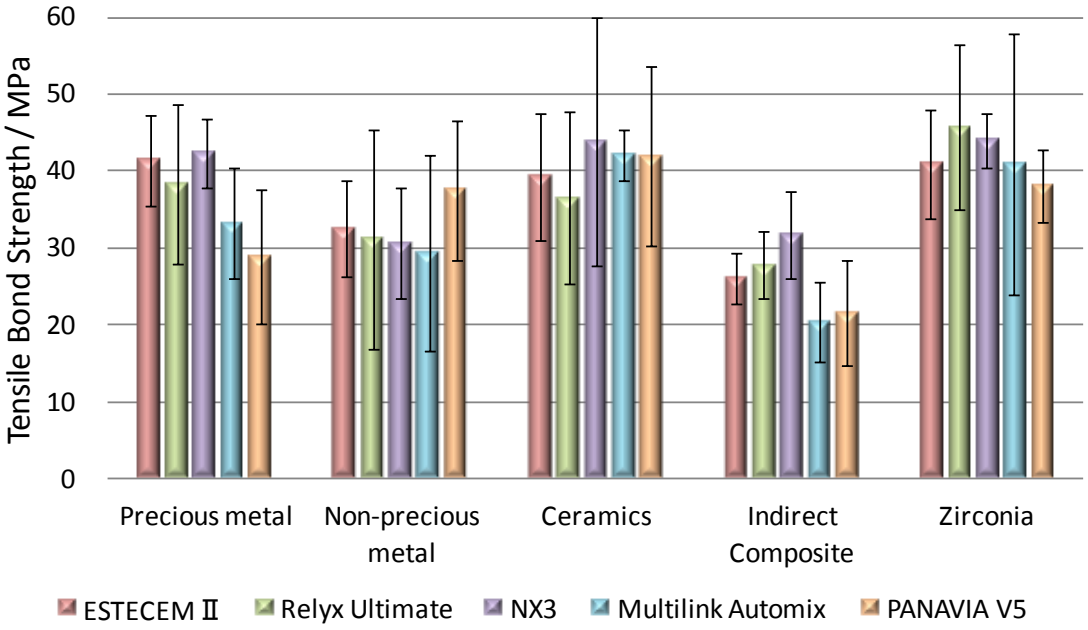


Fig.54 Bond strength on prosthetics using each competitor's resin cement.

6. Conclusion

TOKUYAMA UNIVERSAL BOND is a new adhesive system for both direct and indirect restorations and is a useful material for dental practice.

Features of TOKUYAMA UNIVERSAL BOND

[Universal Use]

- Compatibility with self-etch, total-etch and selective-etch techniques
- Applicability to direct and indirect restoration
- Compatibility with light-curing, dual-curing and self-curing composite materials without the use of a separate activator
- Use as a primer for silica-based, zirconia based and metallic restorations

[Simple Handing]

- No need to apply separately for tooth and restoratives
- No need to wait after bond application
- No need to light-cure

[Reliable]

- High bond strength

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