TOKUYAMA SHIELD FORCE PLUS

Technical Report
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TOKUYAMA SHIELD FORCE PLUS

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1. Introduction

Dentinal hypersensitivity\(^{1-4}\) is a long known pain and is frequently encountered disease in clinical practice. Some reports indicate it occurs in 15% to 20% of all dental patients. A specific definition of dentinal hypersensitivity is given below, after which the mechanism of hypersensitivity is described in terms of hydrodynamic theory.

**Definition of dentinal hypersensitivity:**
A temporary pain (lasting no longer than 10 seconds) induced by thermal (generally, cold below 20 degrees Celsius), dry, tactile, osmotic, or chemical stimulation of exposed dentin and not attributable to any other dental pathology or defect.

**Mechanism of dentinal hypersensitivity:**
Stimulation applied to the dentin induces fluid flow in the dentinal tubules, stimulating free nerve endings and causing pain.

Various methods and materials, described later, have been used to suppress hypersensitivity, all sharing the general approach of sealing the open dentinal tubules.

In the meanwhile, we have developed and introduced TOKUYAMA BOND FORCE, one component Self-etching Light-cured dental adhesive based on SR technology.\(^5,6\)

TOKUYAMA BOND FORCE is characterized by an SR monomer component that penetrates into the tooth substrate, multi-point interactions with apatite calcium, and three-dimensional cross-linking reactions. It forms a thin, even, hard coating on the tooth surface for superior bonding strength to the tooth substance. Given these characteristics, we considered applying SR technology to develop a superior sealant for tooth surface. In November 2009, we introduced the TOKUYAMA SHIELD FORCE, protective sealant for treatment of hypersensitive dentin in Japan. The development concept is described below.

**Development concept**

- **Quick pain relief and long-term coating/sealing**
  A thin, even durable coating and resin tags block external stimulation.

- **Ease of handling**
  Handled just like a bonding material (no rinsing nor rubbing)

In 2010, TOKUYAMA SHIELD FORCE has been improved mainly in terms of treatment time and re-launched as TOKUYAMA SHIELD FORCE PLUS.
2. Cause of clinical hypersensitivity

i) Cases caused by periodontal treatment
Hypersensitivity often occurs after excessive scaling and root planing (SC/RP), particularly SC/RP performed to remove subgingival calculus or to smoothen root surfaces. It also occurs after various types of periodontal surgery that expose dentin on root surfaces previously protected by periodontal tissue and leave the dentinal tubules open.

ii) Cases caused by home care
Inappropriate brushing (over-brushing or inappropriate brushing methods) or inappropriate selection of toothbrushes (hard toothbrushes or electric toothbrushes) can lead to problems such as gingival recession and wedge-shaped defects.

iii) Cases caused by occlusion
Occlusal trauma, excessive bruxism, and clenching can cause problems. Wedge-shaped defects known as abfractions and caused by occlusion are also problematic. (It is generally believed that excessive lateral forces on a tooth cause alternating tension and compression in the cervical region, ultimately damaging the chemical bonds between enamel prisms and generating numerous minute cracks that ultimately form wedge-shaped defects.)

iv) Cases caused by restorative procedures
Hypersensitivity often occurs after cavity preparations for a vital tooth or abutment tooth preparations. For example, preparing a molar typically exposes one to two million dentinal tubules, eventually inducing hypersensitivity. If the hypersensitivity persists after the final restoration is in place, micro-leakage at the marginal areas of the restoration is the likely cause.

v) Cases caused by orthodontic treatment
If force is applied to the tooth outward toward the lips (or the cheeks) as part of an orthodontic procedure, gingival recession may occur in association with absorption of the alveolar bone.

vi) Cases caused by whitening
When 35% hydrogen peroxide solution is applied to a vital tooth (as part of a clinical bleaching procedure), hypersensitivity will sometimes occur during treatment or within several hours after treatment. Some data shows that a third of patients who undergo such whitening treatment experience hypersensitivity to some degree. When hydrogen peroxide solution is applied to the enamel surface, free radicals form and diffuse in the enamel through the surface of the dentin, causing hypersensitivity.
3. Treatment of hypersensitivity

A. Noninvasive treatment

i) Toothpaste for hypersensitivity (e.g., Sensodyne, GlaxoSmithKline)

Principle: Potassium nitrate contained in the toothpaste generates potassium ions, which form ionic barriers around the pulp nerve. The barrier blocks neural transmission in the pulp and quiets pain or discomfort associated with hypersensitivity.

ii) Fluoride-containing gel (e.g., Diadent Dental Gel 5%, Showa Yakuhin Kako Co., Ltd.)

iii) Protein precipitating agent (e.g., Gluma Desensitizer, Heraeus Kulzer)

Principle: Glutaraldehyde in the desensitizer coagulates and stops the motion of dentin fluid, thereby suppressing hypersensitivity.

iv) Iron oxalate, potassium oxalate, aluminum (e.g., Super Seal, Phoenix Dental, Inc.; MS Coat and MS Coat ONE, Sun Medical Co., Ltd.)

Principle: Crystals (e.g., calcium oxalate) seal dentinal tubules.

v) Resins (e.g., TOKUYAMA SHIELD FORCE PLUS, Tokuyama Dental Corp.; Hybrid Coat, Sun Medical Co., Ltd.; Clinpro XT Varnish, 3M ESPE; Seal & Protect, Dentsply Caulk)

Principle: Resins or resin-modified glass ionomer cements seal dentinal tubules.

B. Invasive treatment

vi) Glass ionomer cement; vii) resin filling; viii) periodontal surgery; ix) pulp removal and root canal filling (RCF); x) tooth extraction

As indicated above, treatment strategies fall into two general approaches: a) sealing the orifices of dentinal tubules; b) blocking the transmission of stimulation to pulp nerves. The treatment of last resort is pulp removal and tooth extraction. This report provides as an attachment a list of desensitizing materials commercially available in Japan.
4. Indications, composition, handling, and mechanism of hypersensitive dentin treatment of TOKUYAMA SHIELD FORCE PLUS

4.1 Indications
TOKUYAMA SHIELD FORCE PLUS is indicated for:
- Treatment of hypersensitive dentin,
- Reduction of abrasion and erosion of exposed cervical dentin,
- Alleviation and/or prevention of tooth sensitivity after tooth preparation for direct and indirect restorations.

4.2 Composition
Table 1 shows the composition of TOKUYAMA SHIELD FORCE PLUS. The product contains a phosphoric acid monomer, necessary for decalcifying the tooth substance and forming the matrix of the SR monomer; various monomers used to build the coating; an alcoholic solvent; water; and camphorquinone as the photopolymerization catalyst.

Table 1: Composition of TOKUYAMA SHIELD FORCE PLUS

<table>
<thead>
<tr>
<th>Basic components</th>
<th>Function</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phosphoric acid monomer</td>
<td>Decalcification of tooth substance, SR monomer matrix, coating material</td>
</tr>
<tr>
<td>Bis-GMA</td>
<td>Coating material</td>
</tr>
<tr>
<td>3G (TEGDMA)</td>
<td>Coating material</td>
</tr>
<tr>
<td>HEMA</td>
<td>Monomers that penetrate into the tooth substance, coating material</td>
</tr>
<tr>
<td>Alcohol</td>
<td>Solvent</td>
</tr>
<tr>
<td>Water</td>
<td>Decalcification of tooth substance</td>
</tr>
<tr>
<td>Camphorquinone</td>
<td>Photopolymerization catalyst</td>
</tr>
</tbody>
</table>

4.3 Handling
Figure 1 shows how TOKUYAMA SHIELD FORCE PLUS is used. It does not require rubbing during application or rinsing after application, unlike products from other manufacturers, and can be handled in the same way as bonding materials.
**Sealing of prepared tooth to alleviate / prevent tooth sensitivity (Direct Restorations)**

1. Dispense
2. Apply TOKUYAMA SHIELD FORCE PLUS, then leave for 10 sec
3. Apply weak air (5 sec) → Strong air (5 sec)
4. Light cure for 10 sec

*Adhesive treatment ➔ Composite resin filling*

*If using a total etch adhesive system, sealant must be applied "AFTER" TOKUYAMA SHIELD FORCE PLUS procedure.*

**Sealing of prepared tooth to alleviate / prevent tooth sensitivity (Indirect Restorations)**

1. Dispense
2. Apply TOKUYAMA SHIELD FORCE PLUS, then leave for 10 sec
3. Apply weak air (5 sec) → Strong air (5 sec)
4. Light cure for 10 sec
5. Wipe unpolymerized layer with alcohol

*Impression ➔ Temporary filling / cementing application ➔ Permanent Restoration*

**Before taking impression, it is essential to wipe dentin surface with alcohol, removing unpolymerized layer of TOKUYAMA SHIELD FORCE PLUS that could prevent an accurate impression.

***Do NOT use temporary resin-based material, it may adhere to the cured TOKUYAMA SHIELD FORCE PLUS. The use of water-based cement would be recommended for temporary cementing, and temporary stopping would be recommended for temporary filling.***

Figure 1: Procedure for TOKUYAMA SHIELD FORCE PLUS

**Procedure for products from other manufacturers**

1. Gluma Desensitizer
   - Apply the smallest possible amount (15 seconds) ➔ Allow to stand (30 to 60 seconds) ➔ Air dry ➔ Rinse with water.
2. Super Seal
   - Apply and rub (at least 30 seconds) ➔ Air dry (30 seconds).
3. MS Coat
Mix Liquid A and Liquid B -> Apply and rub (30 to 120 seconds) -> Air dry (10 seconds) -> Rinse with water.

4. MS Coat ONE
Apply and rub (30 seconds) -> Air dry (10 seconds) -> Rinse with water.

5. Clinpro XT Varnish
Dispense paste -> Mix (10 to 15 seconds) -> Apply -> Light-cure (20 seconds) -> Remove unpolymerized layer.

6. Hybrid Coat
Apply to tooth surface (for 10 to 20 seconds with special sponge) -> Weak air blow (5 to 10 seconds) -> Light-cure (5 to 10 seconds).

7. Hybrid Coat II
Apply to tooth surface (for 10 to 20 seconds with special sponge) -> Weak air blow (5 to 10 seconds) -> Light-cure (3 to 10 seconds).

4.4 Mechanism of hypersensitive dentin treatment
The mechanism of hypersensitive dentin treatment of TOKUYAMA SHIELD FORCE PLUS is believed to be based on the double-block effect, illustrated in Figure 2 below.

Mechanism of hypersensitive dentin treatment of TOKUYAMA SHIELD FORCE PLUS

1. When TOKUYAMA SHIELD FORCE PLUS is applied to the affected area, the adhesive monomer (3D-SR monomer) and calcium in the tooth substance react, and the reaction product accumulate in the dentinal tubules and on the coated surface.

2. When the solvent component and water are removed with a stream of air, a thin film forms on the surface affected by hypersensitivity. At this stage, the dentinal tubules are sealed, and the treatment effect (pain relief) appears.

3. Exposure to light cures the reaction product in the dentinal tubules and the thin film on the coated surface, forming a strong coating.

4. As described above, hypersensitivity is suppressed when the dentinal tubules are sealed by the double-block effect: that is, blocking by the reaction product of the adhesive monomer and the calcium of the tooth substance and blocking by the formation of a durable coating on the dentin surface by curing.
Hypersensitivity model

Preparation method:
① Polish bovine tooth dentin.
② Clean with toothpaste and Robinson brush (3 minutes).
③ Ultrasonic cleaning (1 hour).

Light irradiation

Block 1
The reaction product of adhesive monomer and calcium seals the dentinal tubules.

Block 2
Curing forms a durable coating on the dentin surface

Figure 2: Mechanism of hypersensitive dentin treatment of TOKUYAMA SHIELD FORCE PLUS

On the interface of the dentin of an extracted human tooth treated with TOKUYAMA SHIELD FORCE PLUS, we observed an even layer of approximately 10 μm thick (Figure 3). Resin tags of approximately 50 μm appeared on the entire surface treated, indicating that TOKUYAMA SHIELD FORCE PLUS has penetrated into the dentinal tubules and firmly cured (Figure 4). These observations clearly demonstrate that TOKUYAMA SHIELD FORCE PLUS firmly seals and occludes open dentinal tubules.

Test method
① An extracted human tooth was polished with #600 waterproof abrasive paper to prepare the dentin surface.
② With a Robinson brush attached to a dental turbine and toothpaste, the dentin surface was cleaned and polished for 3 minutes, then, subjected to ultrasonic cleaning for 1 hour (Hypersensitivity model).
③ The sample treated with TOKUYAMA SHIELD FORCE PLUS was cut perpendicular to the treated surface with a diamond cutter, and the cut plane of the sample was mirror-polished.
The above sample was dried and sputter-coated with platinum. The adhesion interface was then observed with SEM.

![SEM observation of interface of dentin treated with TOKUYAMA SHIELD FORCE PLUS](image1)

**Figure 3:** SEM observation of interface of dentin treated with TOKUYAMA SHIELD FORCE PLUS

**Test method:**
The sample was prepared in a manner similar to Step 3 above and soaked in a 6N hydrochloric acid solution for 30 seconds and in a 1% sodium hypochlorite solution for 10 minutes to dissolve the dentin and collagen. The adhesion interface was then observed by SEM.

![Observation of resin tags in dentin treated with TOKUYAMA SHIELD FORCE PLUS](image2)

**Figure 4:** Observation of resin tags in dentin treated with TOKUYAMA SHIELD FORCE PLUS

### 5. Features of TOKUYAMA SHIELD FORCE PLUS

#### 5.1 Dentinal tubule occlusion

We evaluated dentinal tubule occlusion and the durability of various desensitizing materials, including TOKUYAMA SHIELD FORCE PLUS. We did this by preparing hypersensitivity models, treating each sample with one of the desensitizing materials according to the
procedure specified in the corresponding manual, and observing dentinal tubule occlusion via SEM before and after subjecting the samples to a thermal shock test of 10,000 cycles. Figure 5 shows the results. As the figure clearly indicates, TOKUYAMA SHIELD FORCE PLUS resulted in highly successful dentinal tubule occlusion, a result that was also observed in repeated testing. Further, no dentinal tubules were observed after the thermal shock test, demonstrating excellent occlusion. These results are based on two mechanisms: the reaction product of the component adhesive monomer (3D-SR monomer) and calcium in the tooth substance firmly seals the dentinal tubules and the coated surface; and curing forms a durable coating on the dentin surface.

Studies conducted by Professor Yoshiyama et al. of Okayama University\textsuperscript{8}) have demonstrated TOKUYAMA SHIELD FORCE's superior dentinal tubule occlusion. Figure 6
Hypersensitivity model

Figure 6: Results of dentinal tubule occlusion test at Okayama University

Next, Figures 7 to 11 show the evaluation results for products from other manufacturers.

Figure 7: Gluma Desensitizer

With Gluma Desensitizer, the initial occlusion shows variations, probably indicating the influence of the coating methods. All samples show open dentinal tubules after the durability test.
Figure 8: Super Seal

With Super Seal, although slightly exposed dentinal tubules are observed, the initial occlusion is satisfactory. However, all samples showed open dentinal tubules after the durability test.

Figure 9: Seal & Protect

With Seal & Protect, although the initial occlusion is satisfactory, the film peels off after the durability test, and images with open dentinal tubules are obtained. (Sample ① has portions with the film peeled off and unpeeled.)
With Clinpro XT Varnish, although the initial occlusion is satisfactory, the images show that this material contains a considerable amount of filler larger than dentinal tubules. On the other hand, after the durability test, the large filler is observed to have detached. Samples also show portions with the film peeled off. The dentinal tubules under the peeled film are sealed.

5.2 Toothbrush abrasion

Next, we evaluated the toothbrush abrasion resistance of the cured film of TOKUYAMA SHIELD FORCE PLUS.

Test method:
① A bovine tooth was polished by #600 waterproof abrasive paper to expose the dentin.
② The tooth surface was treated with the desensitizing material, and half of the tooth surface was protected by ESTELITE LV High Flow.
③ The tooth was subjected to the toothbrush abrasion test with a 33% toothpaste solution (toothpaste: White & White, Lion Corporation) at a load of 400 g and 10,000 abrasion cycles (1 cycle corresponding to 12 toothbrush strokes). After the abrasion test, composite resin was layered on the entire treated surface of the sample.
④ The sample was cut perpendicular to the treated surface with a diamond cutter.
⑤ After embedding into a resin, the sample was mirror-polished and the cut surface observed with a laser microscope (VK9700, KEYENCE, Japan).
⑥ The extent of abrasion was gauged with respect to the top of the coating layer protected by ESTELITTE LV High Flow.
Figure 12: Schematic diagram of toothbrush abrasion test

Figure 13 shows the test results. As shown in the figure, the amount of abrasion for TOKUYAMA SHIELD FORCE PLUS was 1.5 μm (14% of the initial film). TOKUYAMA SHIELD FORCE PLUS showed better toothbrush abrasion resistance than the desensitizing/coating materials from other manufacturers (Figure 14) under the same conditions. Here, since Gluma Desensitizer and Super Seal do not form films, the images show wear on the tooth surface from toothbrush abrasion. The image of Clinpro XT Varnish also shows the worn tooth surface, indicating the relative weakness of the cured film (resin-modified glass ionomer cement). On the other hand, the cured films created by Seal & Protect which includes resin remained; however, they demonstrated greater abrasion than TOKUYAMA SHIELD FORCE PLUS.
5.3 Strength of cured resin

As shown in Section 5.2, the cured film of TOKUYAMA SHIELD FORCE PLUS has been proved to have high resistance against toothbrush abrasion. We measured the strength of the cured resin of TOKUYAMA SHIELD FORCE PLUS, using Hybrid Coat as the control. Table 2 gives the results.

As shown in the table, the strength (bending strength and Knoop hardness) of the cured TOKUYAMA SHIELD FORCE PLUS resin is significantly greater than that of Hybrid Coat. The high strength of the cured resin is probably a major factoring in producing high toothbrush abrasion resistance.

Table 2: Strength of cured TOKUYAMA SHIELD FORCE PLUS resin

<table>
<thead>
<tr>
<th></th>
<th>Flexural Strength (MPa)</th>
<th>Knoop hardness (Kgf/mm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TOKUYAMA SHIELD FORCE PLUS</td>
<td>99.6 (9.7)</td>
<td>25.2 (0.3)</td>
</tr>
<tr>
<td>Hybrid Coat</td>
<td>19.0 (4.1)</td>
<td>15.7 (1.4)</td>
</tr>
</tbody>
</table>

6. Evaluation as coating material

The resin coating technique has grown increasingly common in recent years. The resin coating technique\(^9\)-\(^12\) is used in indirect restorations. A coating material is applied to bond
and cover the prepared dentin, after which impressions of the resin-coated surface are taken for restoration. This method offers benefits with respect to pulp protection (suppression of post operative sensitivity) for vital teeth and improves the tooth bonding strength of resin cements, sealing in marginal areas of restorations, and cavity compatibility. This method was developed by Professors Tagami and Nikaido of Tokyo Medical and Dental University, who evaluated commercially available bonding materials, including TOKUYAMA BOND FORCE and Hybrid Coat (a dental sealing and coating material), as coating materials.

Total-etching bonding materials have now entered widespread use in Europe and North America, resulting in occasional complaints among patients of post-operative sensitivity. Cases have been reported in which treatment incorporates desensitizing materials such as Gluma Desensitizer.

However, as described above, dentinal tubule occlusion tests and toothbrush abrasion tests indicate TOKUYAMA SHIELD FORCE PLUS forms a thin, even, hard coating. Based on the consideration that this coating might block external stimulation, we evaluated the potential of TOKUYAMA SHIELD FORCE PLUS as a coating material.

6.1 Evaluation as coating material for direct restorations
We evaluated the potential of desensitizing/coating materials including TOKUYAMA SHIELD FORCE PLUS as a tooth-coating material after cavity preparation in direct restorations. That is, we evaluated the bonding strength of each bonding material to tooth substances treated with each desensitizing material (Table 3). The results indicate that all bonding materials tested, domestic and international self-etching or total-etching materials, show equivalent or greater bond strength with tooth substances treated with TOKUYAMA SHIELD FORCE PLUS, compared to the control bond strength to untreated tooth substances. This confirms that TOKUYAMA SHIELD FORCE PLUS does not affect the bonding strength of bonding materials. The test also showed that bonding materials with low bonding strength to dentin, such as Single Bond Plus, have higher bonding strength when combined with TOKUYAMA SHIELD FORCE PLUS. TOKUYAMA SHIELD FORCE PLUS is suitable for use as a tooth coating material after cavity preparation in direct restorations.

Super Seal, a desensitizing material provided by another manufacturer, reduces the bonding strength of the bonding materials, likely due to the calcium oxalate generated when the component oxalate ions react with the calcium in the tooth substance. The adverse effect of Super Seal on the bonding strength of the bonding materials was also reported in reference.
As with TOKUYAMA SHIELD FORCE PLUS, Gluma Desensitizer does not affect the bonding strength of the bonding materials, while the effects of Seal & Protect (due to its relatively low bonding strength to tooth structure) depend on the bonding material used.

Table 3: Evaluation as coating material in direct restorations

<table>
<thead>
<tr>
<th>Bonding Agent</th>
<th>Desensitizing/coating material</th>
<th>Bonding strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Dentin</td>
</tr>
<tr>
<td>TOKUYAMA BOND FORCE</td>
<td>TOKUYAMA SHIELD FORCE PLUS</td>
<td>24.7(4.9)</td>
</tr>
<tr>
<td></td>
<td>Gluma Desensitizer</td>
<td>23.8(7.4)</td>
</tr>
<tr>
<td></td>
<td>Seal &amp; Protect</td>
<td>12.7(2.8)</td>
</tr>
<tr>
<td></td>
<td>Super Seal</td>
<td>4.4(2.4)</td>
</tr>
<tr>
<td></td>
<td>—</td>
<td>23.3(2.5)</td>
</tr>
<tr>
<td>Optibond All In One</td>
<td>TOKUYAMA SHIELD FORCE PLUS</td>
<td>24.9(5.0)</td>
</tr>
<tr>
<td></td>
<td>—</td>
<td>10.3(1.8)</td>
</tr>
<tr>
<td>SE Bond</td>
<td>TOKUYAMA SHIELD FORCE PLUS</td>
<td>25.0(5.7)</td>
</tr>
<tr>
<td></td>
<td>Gluma Desensitizer</td>
<td>26.2(3.4)</td>
</tr>
<tr>
<td></td>
<td>Seal &amp; Protect</td>
<td>11.6(2.9)</td>
</tr>
<tr>
<td></td>
<td>Super Seal</td>
<td>9.3(2.8)</td>
</tr>
<tr>
<td></td>
<td>—</td>
<td>20.9(2.7)</td>
</tr>
<tr>
<td>Scotch Bond Multi Purpose</td>
<td>TOKUYAMA SHIELD FORCE PLUS</td>
<td>20.6(5.6)</td>
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<tr>
<td></td>
<td>—</td>
<td>13.1(2.6)</td>
</tr>
<tr>
<td>Single Bond Plus</td>
<td>TOKUYAMA SHIELD FORCE PLUS</td>
<td>25.8(7.5)</td>
</tr>
<tr>
<td></td>
<td>Gluma Desensitizer</td>
<td>11.6(3.8)</td>
</tr>
<tr>
<td></td>
<td>Seal &amp; Protect</td>
<td>10.4(1.9)</td>
</tr>
<tr>
<td></td>
<td>Super Seal</td>
<td>6.8(2.1)</td>
</tr>
<tr>
<td></td>
<td>—</td>
<td>10.6(2.3)</td>
</tr>
<tr>
<td>Prime&amp;Bond NT</td>
<td>TOKUYAMA SHIELD FORCE PLUS</td>
<td>24.7(5.9)</td>
</tr>
<tr>
<td></td>
<td>—</td>
<td>11.3(2.8)</td>
</tr>
<tr>
<td>Optibond solo Plus</td>
<td>TOKUYAMA SHIELD FORCE PLUS</td>
<td>25.3(6.1)</td>
</tr>
<tr>
<td></td>
<td>—</td>
<td>18.7(2.1)</td>
</tr>
</tbody>
</table>

Test method:
① A set of four freshly extracted bovine teeth were prepared. The teeth were polished parallel to the labial surface in running water with #120, then #600 waterproof abrasive paper, to prepare the enamel and dentin surface.
② A piece of 180-μm thick double-faced adhesive tape with an opening measuring 3 mm in diameter was attached to the polished surface to define the bonding area. Each desensitizing/coating material was applied with a microbrush (as specified in the product manual). In the case of TOKUYAMA SHIELD FORCE PLUS, unpolymerized resin remaining on the surface after 10 seconds of light irradiation was not removed.
③ The tooth substance treated with each material was treated with various bonding materials and light-cured.
A piece of 0.5-mm thick paraffin wax with a hole of a diameter of 8 mm was attached over the 3-mm hole so that the centers of the openings matched. Composite resin (ESTELITE POSTERIOR) was applied to fill the 8-mm opening. It was then compressed by a polypropylene film from above and irradiated by light for 10 seconds. Then, a metal attachment was bonded to the composite resin surface with resin cement (BISTITE II DC), and the sample was soaked overnight in water at 37 degrees Celsius.

Using an Autograph (AG5000D, Shimadzu), we measured the tensile bonding strength at a cross head speed of 2 mm/min. Using the same procedure, as a control, we separately measured the bonding strength of each bonding material to the tooth substance.

6.2 Evaluation as coating material for indirect restorations

We assessed the potential of various desensitizing/coating materials, including TOKUYAMA SHIELD FORCE PLUS, as tooth coating materials after cavity preparation and abutment tooth preparations in indirect restorations. For the tooth substance treated with each desensitizing/coating material, we took impressions, applied temporary filling materials, removed the temporary filling materials, then evaluated the bonding strength of each cement.

6.2.1 Effects on impression accuracy

When Hybrid Coat is used as a coating material on the abutment tooth surface, reports indicate the unpolymerized resin layer of the coating material prevents the curing of various silicone impression materials.\(^{14,15}\) We evaluated the impression accuracy of silicone impression materials when using TOKUYAMA SHIELD FORCE PLUS as the tooth coating material.

Table 4: Effects of TOKUYAMA SHIELD FORCE PLUS on impression accuracy

<table>
<thead>
<tr>
<th>Silicone impression material</th>
<th>TOKUYAMA SHIELD FORCE PLUS</th>
<th>Hybrid Coat</th>
<th>Hybrid Coat II</th>
</tr>
</thead>
<tbody>
<tr>
<td>Exa Hiflex</td>
<td>○</td>
<td>○</td>
<td>○</td>
</tr>
<tr>
<td>Imprinsis</td>
<td>○</td>
<td>○</td>
<td>○</td>
</tr>
<tr>
<td>Aquasil Ultra Heavy</td>
<td>○</td>
<td>○</td>
<td>○</td>
</tr>
<tr>
<td>Aquasil Ultra LV</td>
<td>○</td>
<td>○</td>
<td>○</td>
</tr>
<tr>
<td>Imprint II Garant</td>
<td>○</td>
<td>○</td>
<td>○</td>
</tr>
</tbody>
</table>

Evaluation method:

Freshly extracted bovine teeth were polished parallel to the labial surface with #120 waterproof abrasive paper to prepare the dentin surface.
The coating material is applied to half of the prepared dentin surface.

The coated surface was classified into the three following groups:

(A) The unpolymerized resin layer was not removed.
(B) The unpolymerized resin layer was partially removed with alcohol-soaked cotton balls.
(C) The unpolymerized resin layer was completely removed with alcohol-soaked cotton balls.

The silicone impression material was poured into a tray (R-3) and the tooth pressed into the impression material with the coating surface oriented down until the impression material cured.

The bovine tooth was removed from the cured impression material, plaster poured into the negative impression obtained, and the impression accuracy (curing defects and surface roughness) assessed based on the surface condition of the plaster model.

*Evaluation standard:
○: The plaster surface is shiny (no effect).
△: The plaster surface is slightly cloudy (small effect).
×: The plaster surface shows corrugations (clear effect).

Table 4 shows the results, which confirm that TOKUYAMA SHIELD FORCE PLUS does not affect the curing of various silicone impression materials, whether or not an uncured resin layer is present.

On the other hand, the results confirm the effects of Hybrid Coat (results similar to those described in references[14] and[15]). Hybrid Coat II, an improved product based on Hybrid Coat, shows no effects.

6.2.2 Effects on resin cement bond strength

We evaluated the bonding strength of various resin cements and self-adhesive cements to tooth substances treated with TOKUYAMA SHIELD FORCE PLUS. As Table 5 shows, all samples tested show identical or better bonding strength compared to the dentin bonding strength of the adhesive resin cements themselves, regardless of the temporary filling material (accounting for bonding strength and fracture morphology). In short, coating with TOKUYAMA SHIELD FORCE PLUS does not reduce the bonding strength of the adhesive resin cement. With SA Luting, a self-adhesive cement, the interface between the cured resin of TOKUYAMA SHIELD FORCE PLUS and the cement fractured (marked as K’ in the table). This is probably due to the lower bonding strength between the SA Luting and the cured TOKUYAMA SHIELD FORCE PLUS resin caused by the silicone impression material and temporary filling material. Based on the above phenomenon and the bonding strength of the self-adhesive cement itself, we would recommend against using self-adhesive cements that do not require pretreatment agents.
Table 5: Effects of TOKUYAMA SHIELD FORCE PLUS on resin cement bond strength

<table>
<thead>
<tr>
<th>Coating material</th>
<th>Silicone impression material</th>
<th>Temporary filling material</th>
<th>Adhesive resin cement</th>
<th>Multilink Automix /Ivoclar</th>
<th>SA Luting /Kuraray</th>
</tr>
</thead>
<tbody>
<tr>
<td>TOKUYAMA SHIELD FORCE PLUS</td>
<td>Imprinsis</td>
<td>Caviton</td>
<td>10.3(4.9)</td>
<td>13.1(1.5)</td>
<td>20.8(3.8)</td>
</tr>
<tr>
<td></td>
<td>Imprinsis</td>
<td>HY-Bond Temp Cem</td>
<td>16.5(6.1)</td>
<td>14.0(2.8)</td>
<td>16.9(5.6)</td>
</tr>
<tr>
<td></td>
<td>Imprinsis</td>
<td>Stopping</td>
<td>19.0(5.5)</td>
<td>14.4(1.0)</td>
<td>14.0(2.5)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>14.9(3.6)</td>
<td>20.5(3.0)</td>
<td>22.9(2.9)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>13.1(3.1)</td>
<td>8.9(0.5)</td>
<td>12.8(1.1)</td>
</tr>
</tbody>
</table>

Evaluation method:

① A set of four freshly extracted bovine teeth were prepared. To prepare the dentin surface, the teeth were polished parallel to the labial surface in running water with #120, then with #600 waterproof abrasive paper.

② A piece of 180-μm thick double-faced adhesive tape with an opening measuring 4 mm in diameter was attached to the polished surface to define the bonding area. The surface was treated with TOKUYAMA SHIELD FORCE PLUS or desensitizing/coating materials from other manufacturers by the procedures specified in the corresponding manuals. The unpolymerized resin layer on the surface was removed with alcohol-soaked cotton balls for TOKUYAMA SHIELD FORCE PLUS, Hybrid Coat, and Seal & Protect.

③ An impression of the sample surface was taken with each impression material.

④ A piece of 0.5-mm thick paraffin wax with an opening with a diameter measuring 8 mm was attached over the 4-mm opening, with the centers of the holes matching. Each temporary filling material was applied to the opening in the wax, and the sample was soaked overnight in water at 37 degrees Celsius.

⑤ After removing the temporary filling material, the sample surface was cleaned with alcohol-soaked cotton balls, and the coating surface was treated with the tooth pretreatment agents supplied with the corresponding resin cement. An attachment was then bonded to the coating surface with the resin cement, and the sample was soaked in water at 37 degrees Celsius for 24 hours.

*SA Luting, a self-adhesive cement, was bonded directly, without tooth pretreatment agents.

⑥ Using an Autograph (AG5000D, Shimadzu), we measured the tensile bonding strength at a cross head speed of 2 mm/min and observed the fracture morphology according to the description below.
Fracture morphology
K: Interface failure between dentin and cured TOKUYAMA SHIELD FORCE PLUS resin
K’: Interface failure between cement and cured TOKUYAMA SHIELD FORCE PLUS film
D: Failure in dentin
C: Cohesive failure in cement
A: Interface failure between attachment and cement

We evaluated the bonding strength of the adhesive resin cement to the tooth substance treated with desensitizing/coating materials from other manufacturers. As Table 6 shows, TOKUYAMA SHIELD FORCE PLUS offers superior performance.

Table 6: Effects of desensitizing/coating materials from other manufacturers on bonding strength of adhesive resin cements

<table>
<thead>
<tr>
<th>Impression material</th>
<th>Temporary cement</th>
<th>Cement</th>
<th>Desensitizing/coating material</th>
<th>Bond strength to dentin</th>
</tr>
</thead>
<tbody>
<tr>
<td>EXAHIFLEX /GC</td>
<td>Caviton</td>
<td>BISTITE II/ Tokuyama Dental</td>
<td>TOKUYAMA SHIELD FORCE PLUS</td>
<td>28.1(6.3)D</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Super Seal</td>
<td>8.2(5.3)K</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Gluma</td>
<td>8.1(5.1)K</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>MS Coat ONE</td>
<td>15.2(3.5)K</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Hybrid Coat II</td>
<td>13.5(4.3)D</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>—</td>
<td>10.4(5.2)K</td>
</tr>
<tr>
<td>Aquasil Ultra Heavy /Dentsply</td>
<td>Caviton</td>
<td>N.X.3 /Kerr</td>
<td>TOKUYAMA SHIELD FORCE PLUS</td>
<td>16.4(5.7)A</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Super Seal</td>
<td>9.2(1.1)K</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Gluma</td>
<td>12.0(4.5)K</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>MS Coat ONE</td>
<td>9.1(3.2)K</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Seal &amp; Pretect</td>
<td>10.4(4.8)K</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>—</td>
<td>10.6(7.5)K</td>
</tr>
<tr>
<td>Aquasil Ultra Heavy /Dentsply</td>
<td>Stopping</td>
<td>Multilink Automix /Ivoclar</td>
<td>TOKUYAMA SHIELD FORCE PLUS</td>
<td>18.6(7.6)D</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Super Seal</td>
<td>8.8(7.7)K</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Gluma</td>
<td>13.0(2.1)K</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>MS Coat ONE</td>
<td>16.1(3.3)K</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Hybrid Coat II</td>
<td>10.9(2.7)D,K</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>—</td>
<td>12.8(1.1)K,D</td>
</tr>
</tbody>
</table>

These results indicate TOKUYAMA SHIELD FORCE PLUS is suitable for use as a tooth coating material following cavity preparation or abutment tooth preparation in indirect restorations.
7. Conclusion
Introduced in 2010, TOKUYAMA SHIELD FORCE PLUS is resin based protective sealant with the features listed below. These features will provide superior effect of hypersensitive dentin treatment (quick & long term pain relief) and durable coating following cavity/tooth preparations in clinical settings.

Features of TOKUYAMA SHIELD FORCE PLUS
✓ Ease of handling and Quick pain relief
✓ Superior dentinal tubule occlusion and durability
✓ Superior toothbrush abrasion resistance
8. References

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