YAMAKIN

From Production Skills to Scientific Knowledge,
and Onward to Fusion with

Controlled Medical Device – Dental glazing material
(Coloring material for polymer-based crown, Bonding Material for Dental Resin)

Nu:le Coat

Product Report

**The performance and key points for the use of "Nu:le Coat"
in response to clinical practice**

Edited by YAMAKIN Ph.D. Group

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What is the YAMAKIN Ph.D. Group?

This is a group of experts in various specialized fields who bring together their knowledge, experience and technical expertise to act as a prime motivator in the continuous generation of innovation.

1. Introduction

In the finishing touch of dental restoration, it is required that the material surface is abundant in terms of aesthetical purposes and the inhibition of plaque adhesion.

While traditional methods involving mirror finishing with grinding tools and abrading agents have been indispensable to glaze the material surface, there is a growing trend toward the use of surface glazing material which can easily glaze the surface in a short time.

Performance requirements for the dental glazing material are as follows: to be able to be applied thinly and evenly on the material surface, to form a hard coating, and to be stable in the oral cavity. Technical challenges in designing a dental glazing material include cure shrinkage strain of resin, discoloration after curing, and the restraint of a decrease in luster caused by abrasion.

“Nu:le Coat”, released by YAMAKIN in September 2021, is a dental glazing material that has a lower strain hardening, less discoloration after curing, and better gloss retention. In this technical report, the concept of “Nu:le Coat”, usage instructions and physical properties are introduced from a perspective based on material science and engineering. We hope that this product will be of interest to you.

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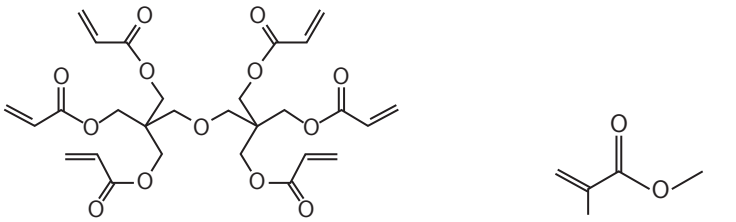
2. What is dental glazing material?

The application of dental glazing material can create a smooth surface. The surface should be smooth and scratch-resistant without any unpolymerization after curing and have less discoloration after curing. To accomplish these functions, it is important to consider the selection of monomers or photoinitiators. The optimal monomers and photoinitiators for dental glazing material are explained below.

2.1. Monomer

Dental glazing material requires monomers characterized by high reactivity and reduced susceptibility to oxygen, which can impede polymerization inhibition of the material surfaces. The unpolymerized layer after curing will hinder the development of a gloss finish. It is often used Methacrylate Monomers such as Urethane Dimethacrylate (UDMA) for light-cured indirect composite resin for the crown, however, it can reduce oxygen inhibition by using Acrylate Monomers that are more reactive or monomers with polyfunctional groups in the monomer and higher cross-linked density, such as Dipentaerythritol Hexaacrylate (DPHA), which has six functional groups, for glazing material (Figure 2-1). In addition, DPHA alone is impractical for direct application due to its high viscosity, therefore, monomers with monofunctional groups such as Methyl methacrylate (MMA) are often used together to adjust viscosity. MMA is also used in autopolymerizing resin for denture bases but it has a volatile and specific odor, so dental glazing material basically has an odor derived from MMA before curing.



Dipentaerythritol Hexaacrylate (DPHA)

Methyl methacrylate (MMA)

Figure 2-1 Structures of monomers

2.2. Photoinitiator and the absorption wavelength

As mentioned above, since it cannot prevent polymerization inhibition by oxygen completely even using monomers with polyfunction groups, methods that reduce the relative impact of oxygen inhibition are used, including adding the amount of photoinitiator (hereinafter referred to as initiator) and increasing the radical generation per unit time by combining a high-power light source. Dental glazing material uses radical polymerization as same as light-cured indirect composite resin for the crown, but the initiator is different. A light-cured indirect composite resin such as TWiNY (YAMAKIN CO., LTD.) mainly uses a camphorquinone-based initiator (example; camphorquinone; CQ, Figure 2-2), and causes radical by blue

light with a wavelength around 470 nm (Figure 2-3) [1, 2].

Therefore, commercial light irradiators for dental use basically have a light source emitting blue light. Although CQ absorbs blue light efficiently, CQ itself has a strong yellow color. There is no problem using CQ in light-cured indirect composite resin for the crown since it uses little initiator and the yellow color in the dental crown does not stand out; however, for dental glazing material, the yellow color ruins the shade of the applied surface. In addition, CQ does not react sufficiently to acrylic monomers used in dental glazing material.

For this reason, generally, phosphine oxide-based initiators (e.g., 2, 4, 6-trimethylbenzoyl diphenylphosphine oxide; TMDPO, Figure 2-2) are used in hardening and dental glazing material applications. Since this initiator has an absorption wavelength of around 395 nm and reacts to violet light, polymerization equipment for dental laboratories or dental polymerization light irradiators corresponding to glazing material basically needs to have a light source emitting violet light (Figure 2-4).

One of the characteristics of TMDPO is that it does not affect the color of the monomer before curing because it becomes colorless and transparent when dissolved in the monomer [1, 2]. However, even phosphine oxide-based initiators in large amounts will turn reddish brown after curing, so it is necessary to adjust the amount and suppress the color change before and after curing as much as possible. Other initiators used in dental glazing material are, for example, 1-Phenyl-1,2-propanedione (PPD) and Bis (2,4,6-trimethoxyphenyl) phenylphosphine oxide (BTMPO).

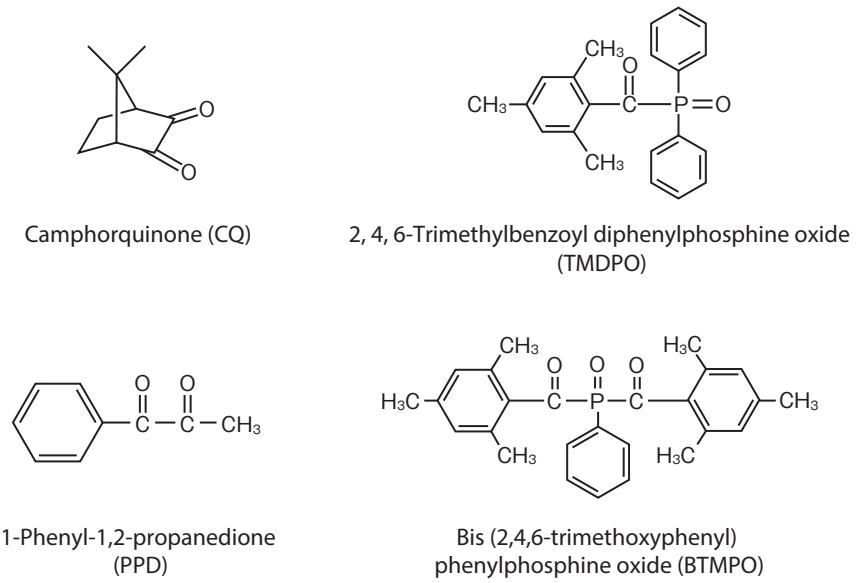


Figure 2-2 Chemical formulas of initiators

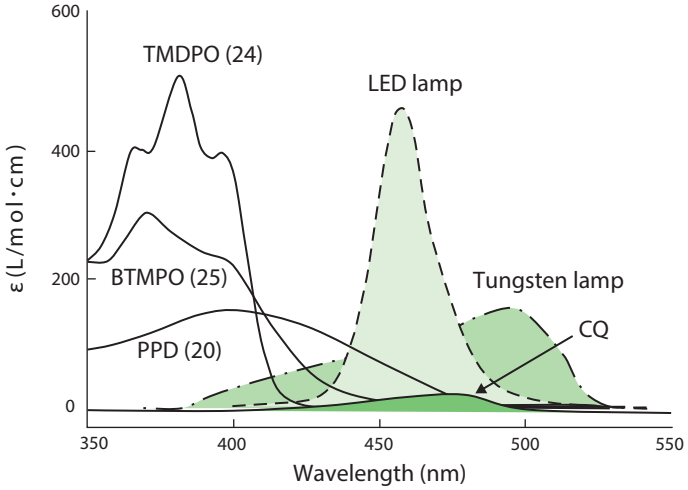


Figure 2-3 Absorption wavelength of initiators [1, 2)]

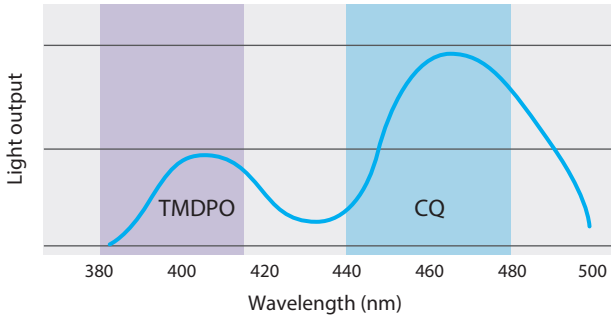


Figure 2-4 Wavelength of dental polymerization light irradiator and absorption wavelength of initiators

3. Features of “Nu:le Coat”

Also, as plus one application, it can be used for pre-treatment of PEEK before applying resin materials (Figure 3-1).

Figure 3-1 Features of Nu:le Coat

In order to confirm these concepts, the following tests were carried out using reference samples of “Nu:le Coat Liquid Clear” (hereinafter Liquid Clear) and “Nu:le Coat Gel” (hereinafter Gel), which consist of approximately twice as much initiator loading as “Nu:le Coat”.

3.1. Transparency

Since Liquid Clear has high transparency after curing (Figure 3-2), it has an insignificant effect on the original color tone of dental restorations even after layering, and is usable for whitening shades such as A0-GR of “KZR-CAD HR Block 4 E-VA (YAMAKIN CO., LTD.)” (hereinafter referred to as E-VA) (Figure 3-3).

• Test condition

(1) Pellet

We checked the appearance of a pellet which was made by pouring Liquid Clear into a jig with a diameter of 12 mm and a thickness of 1 mm, and light-cured for 60 seconds by “LED CURE Master (YAMAKIN CO., LTD.),” a curing light for indirect composite resin. As a reference, we also created pellets formed from the reference samples.

(2) Application on a whitening shade

We checked the appearance of samples, which were made by applying Liquid Clear and the reference samples to “E-VA” (A0-GR), light cured for 5 seconds by “LED CURE Master,” afterward, applied each sample once more, and light-cured for 60 seconds by “LED CURE Master”.

• Test result

As shown in Figure 3-2, 3-3, discoloration was visually confirmed on reference samples with a large amount of photoinitiators (Figure 3-2). Discoloration was also seen in the case when applied and cured on “E-VA” (A0-GR) (Figure 3-3). Instead, Liquid Clear remains almost colorless and transparent with a thickness of 1mm, and the appearance when applied on “E-VA” is almost unchanged as abrasive finishing.

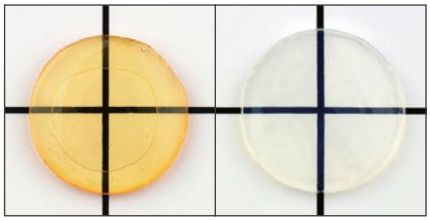


Figure 3-2 (left) Liquid Clear (right) reference sample
(immediately after light-curing) thickness 1.0 mm

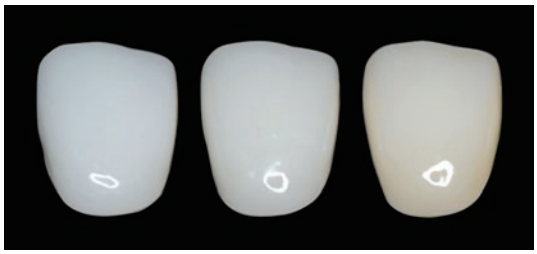


Figure 3-3 “E-VA” (A0-GR)
(left) only polishing (middle) Liquid Clear two-layer coating
(right) reference sample two-layer coating

3.2. Curing strain and crack suppression

The combination of flexible polyfunctional monomers and tough cross-linkable monomers reduced curing strain. Therefore, cracks and/or floated surfaces are less likely to occur even if the applied layer becomes thicker because of overglaze. As below, the existence of curing strain or cracks was examined in the case that “Nu:le Coat” or the reference sample is applied on a pellet or the pit and fissure.

• Test condition

(1) Evaluation of curing strain

Material is set in the model with a diameter of 12 mm and a thickness of 0.5 mm, inserted between transparent PET films, and light-cured for 60 seconds.

By measuring the entire thickness of a light-cured pellet four times in increments of 45° by using a caliper “ABS digimatic caliper” (Mitsutoyo Corporation), the strain amount is defined as the amount calculated by subtracting the thickness before light-curing (0.5 mm) from the thickness after curing (it becomes thicker compared to before curing by the curl due to strain) (n=3). Three types of pellets were made from

Liquid Clear, Gel and the reference sample.

(2) Evaluation of cracks

After light-curing Liquid Clear and a reference sample dripped directly into the pit and fissure of crowns, the presence of cracks and the floated surface is assessed by a microscope.

• Test result

(1) Result of curing strain evaluation

Compared to the thickness of a pellet before light-curing (0.5 mm), the curing strain of the relative sample is approximately 0.7 mm, and the strain can also be visually observed. On the other hand, the curing strain of “Nu:le Coat” is under 0.1 mm, which marks a substantial reduction (Figure 3-4).

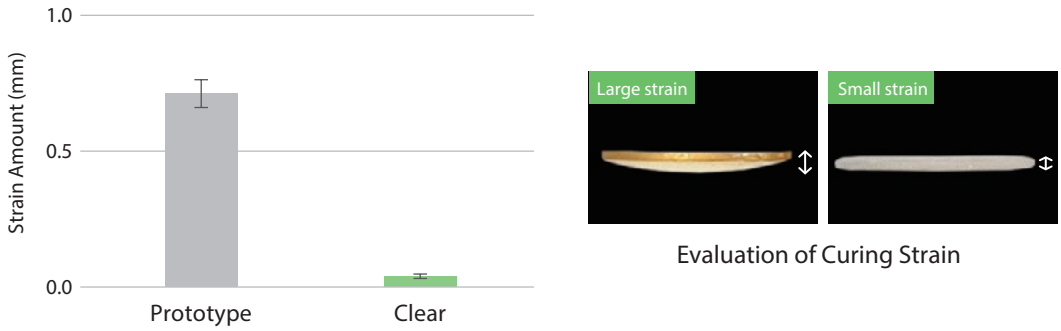


Figure 3-4 Evaluation of curing strain

(2) Result of crack evaluation

When evaluated under the assumption that a lot of liquid has been applied onto the pit and fissure of crowns or the junction, the emergence of cracks is seen in the reference sample, whereas there is no appearance of cracks on Liquid Clear (Figure 3-5). These results indicate that “Nu:le Coat” is expected to inhibit cracking and floated surface.

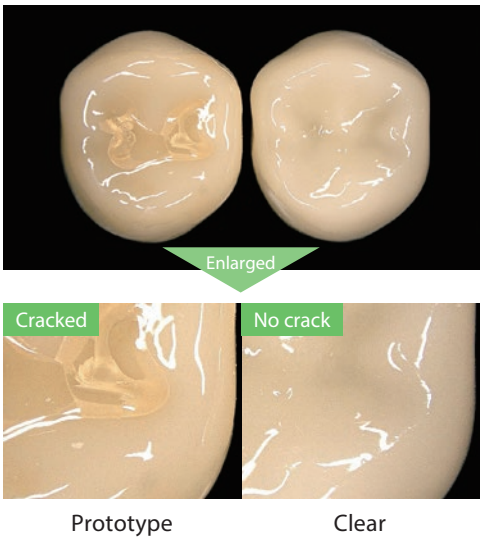


Figure 3-5 Evaluation of crack on the pit and fissure of crowns

3.3. Pencil hardness

The hardness of surface coating material is evaluated by various tests, including the pencil hardness test, the steel wool abrasion test, the cross-cut test, and other tests. Particularly, in recent years, the pencil hardness test has been used as an index for familiar coating materials such as the screen protector of smartphone screens. The hardness of "Nu:le Coat" is evaluated by the pencil hardness test.

• Test condition

Resin pellets are prepared by filling E3 of "Luna-Wing" (YAMAKIN CO., LTD.), a composite resin for crowns, into a mold with a diameter of 15 mm and a thickness of 1mm. The mold is then sandwiched between two transparent PET films and light-cured for 90 seconds each from both faces. Both Liquid Clear and Gel are applied on each resin pellet and light-cured for 60 seconds by "LED CURE Master", a curing light for indirect composite resin.

Afterward, the pencil hardness of the surface after light-cured is measured in accordance with JIS K 5600-5.4 (Scratch hardness (Pencil method)) (n=3).

• Test result

The test result shows that the 7H pencil for Gel and the 9H pencil for Liquid Clear did not leave the pencil scratch (Figure 3-6).

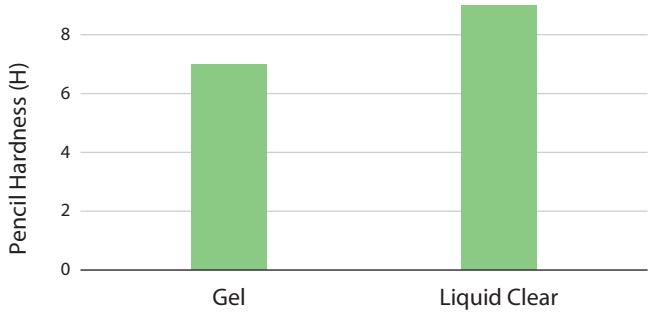


Figure 3-6 Pencil Hardness

Additionally, Figure 3-7 illustrates the examples of the pencil hardness and Mohs hardness for materials familiar to us. The pencil hardness of 9H that Liquid Clear results in is equivalent to nearly 5 of Mohs hardness, which corresponds to the hardness between platinum and the blade of a knife.

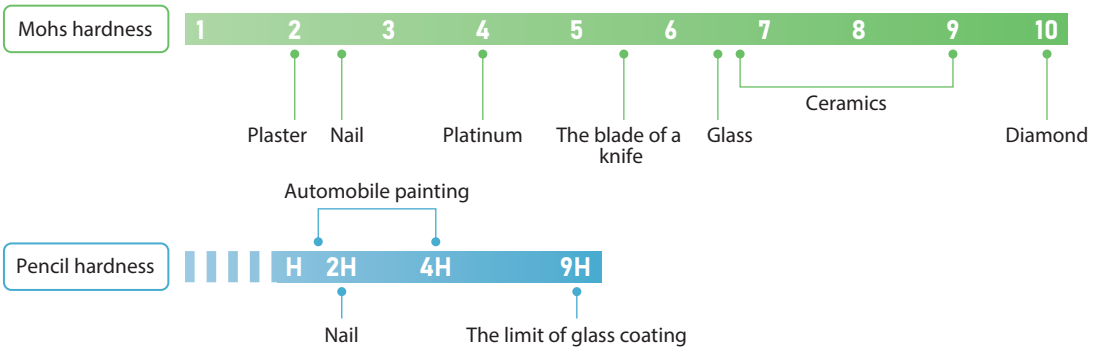


Figure 3-7 Pencil hardness and Mohs hardness [3)]

3.4 Thickness of coated layer

Dental glazing material needs to be applied thinly and evenly to give smoothness because problems such as affecting the adaption of dental prosthesis can occur when the coated layer is thick. The thickness of the coated layer is evaluated for Liquid Clear and Gel.

• Test condition

Pellets with a thickness of 1 mm are made by using "Luna-Wing," and after polishing the surface, Liquid Clear or Gel is applied onto pellets and light cured. The thickness of the coating layer is measured by comparing the thickness before and after application with a micrometer.

• Test result

The thickness of one coating layer, as shown in Figure 3-8, is about 5 μm for Liquid Clear and about 20 μm for Gel (Figure 3-8). When using Gel, Liquid Clear needs to be used as the base layer. Hence, the thickness of the Gel is 25 μm. The contact zone between adjacent teeth of crown restorations should range from 50 μm to 110 μm, and it is appropriate the condition where a contact gauge of 50 μm can be inserted but that of 110 μm cannot be inserted. Also, it is said that the interdental space of 150 μm can cause food impaction, food stuck between the gaps, with a high probability[4]. The Gel can be used to adjust the contact in the case when the gap between adjacent teeth is large after setting in the oral cavity.

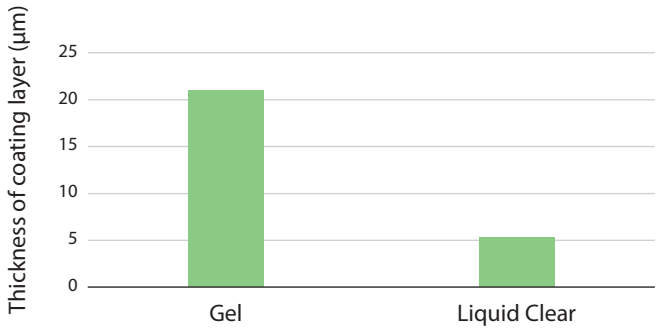


Figure 3-8 Thickness of coating layer

3.5. Applicable time

Since dental glazing material generally contains various components, they should be used as soon as possible after collection. Similarly, "Nu:le Coat" contains volatile components, which will volatilize and lead to increased viscosity if they are left unattended. However, there may be cases in which some time after collection is required depending on work. In this study, we collected Liquid Clear on an ordinary mixing pad or a dedicated plate attached to the product and confirmed whether or not it is possible to apply after a certain amount of time has elapsed.

• Test condition

Liquid Clear is collected on an ordinary mixing pad or a dedicated plate, and evaluated to see if it could actually be applied with a brush after 10 seconds, 30 seconds, 1 minute, 2 minutes, 4 minutes and 5 minutes.

• Test result

Table 3-1 Applicable time of Liquid Clear depending on collecting methods

Mixing pad	Dedicated plate
Usable up to 1 minute after collection	Usable up to 4 minutes after collection

The volatile components of Liquid Clear volatilized within one minute after being collected on a mixing pad, the increased viscosity made it impossible to apply it evenly with a brush.

On the other hand, the dedicated plate allowed uniform application with a brush even after 4 minutes. Therefore, we recommend the use of the dedicated plate when time is required after collection, such as when applying to multiple teeth.

Another technique, which will be explained in a later chapter, is to collect the liquid color type on a mixing pad and leave it for more than one minute to volatilize the MMA, thereby increasing the viscosity and making it easier to apply color to certain parts of the teeth.

If MMA is volatilized too much, however, it resembles the composition of Gel, and adhesion to the surface of the material by itself is reduced. When using this technique, accordingly, it is necessary to apply one layer of Liquid Clear and light irradiation (10 seconds) before characterization.

Although there is no problem with the dedicated plate made of polypropylene, care should be taken when using a collection plate made of polystyrene because the MMA contained in “Nu:le Coat Liquid” may dissolve the components of the collection plate and affect the quality of the product.

3.6. Curing property

Indirect composite resin usually leaves an unpolymerized layer on the surface unless an air barrier material is used during polymerization. In ordinary circumstances, this unpolymerized layer is wiped off with alcohol after polymerization, or removed during morphological modification or polishing by using a diamond bar or the like. The use of dental glazing material with an unpolymerized layer remaining is contraindicated because it may cause insufficient polymerization and performance degradation. The surface smoothness and pencil hardness of Liquid Clear are measured when it’s applied to indirect composite resin with an unpolymerized layer.

• Test condition

A mold with a diameter of 15 mm and a thickness of 1 mm was filled with “Luna-Wing” and polymerized

with the surface exposed to air, and pellets with an unpolymerized resin layer remaining were prepared (unpolymerized side). Conversely, the pellets cured according to the standard method were used as comparison samples (polymerized side). Liquid Clear was applied to each pellet, and the smoothness of the surface was checked after light cure. The test was conducted to evaluate the smoothness by visual inspection and pencil hardness (Figure 3-9).

• Test result

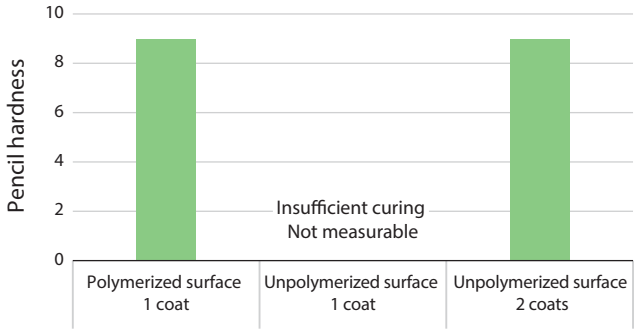


Figure 3-9 Pencil hardness when applied to polymerized/unpolymerized surfaces

When Liquid Clear was applied to an unpolymerized surface, the first coated mixed with the unpolymerized layer of composite resin and did not produce a smooth film due to the dental glazing material, and pencil hardness could not be evaluated. When Liquid Clear was reapplied (two coats) to the surface, the same smoothness and pencil hardness were confirmed as those of the film applied once to the polymerized surface.

3.7. Durability

A toothbrush abrasion test was conducted under clinical conditions to measure changes in gloss and surface roughness, by applying “Nu:le Coat” to the resin block for CAD/CAM “KZR-CAD HR Block 2 BG” (YAMAKIN CO., LTD.).

• Test condition

Test samples were rectangular pellets of 14.5 mm in length and width and 1mm in thickness made of “KZR-CAD HR Block 2 BG”, coated with Liquid Clear and photo-polymerized for 60 seconds (coated with “Nu:le Coat”). The samples polished to a mirror surface were used as comparison samples (polished finish). Toothbrush conditions were in accordance with ISO 14569-1, with a load of 2.0 N in a slurry to toothpaste: water =1:2 [5]. The back-and-forth toothbrush abrasion test was performed 50,000 times, and the gloss and surface roughness were measured every 10,000 cycles. The number of 50,000 toothbrush abrasion tests is equivalent to about 7 years, assuming that the toothbrushing is done twice a day and each tooth is brushed 10 times per toothbrushing.

• Test result

In the evaluation of the toothbrush abrasion test (50,000 times), both the gloss and surface roughness of the “Nu:le Coat” coating were equal to or more than those of the polished finish, confirming its excellent abrasion resistance. It is reported that plaque adhesion increases rapidly when the surface roughness exceeds 0.2 μm[6], but the surface roughness was less than 0.1 μm even after 50,000 toothbrush abrasion tests (Figure 3-10).

In the gloss change by toothbrush abrasion test, the durability of “Nu:le Coat” was confirmed to be equal to or more than that of polished surfaces (Figure 3-11).

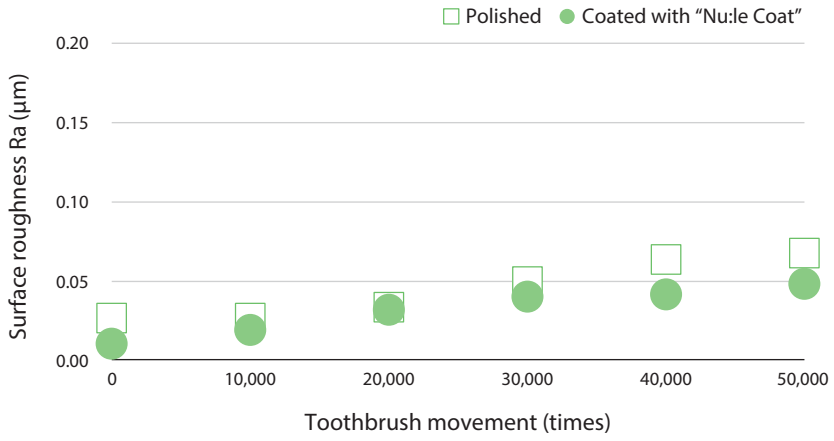


Figure 3-10 Scratch resistance by toothbrush abrasion test

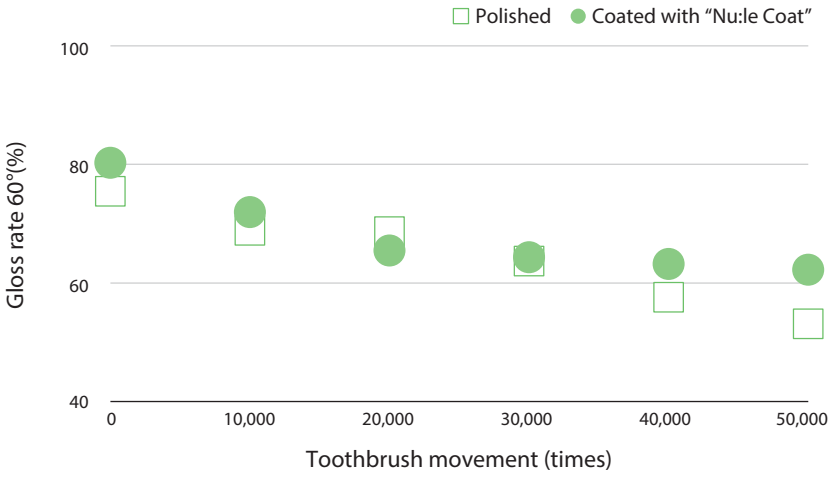


Figure 3-11 Change in gloss by toothbrush abrasion test

In addition, the “Nu:le Coat” liquid color type (blue and orange) for characterization was applied and was similarly subjected to the toothbrush abrasion test for 50,000 cycles. It was confirmed that it was fixed to the resin block surface without peeling (Figure 3-12).

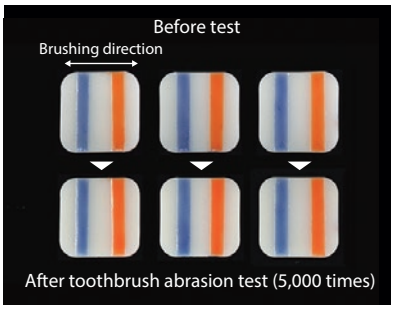


Figure 3-12 Peel resistance by toothbrush abrasion test

3.8. Adhesion to PEEK

Polyaryletherketone (PAEK)-based super engineering plastics (PEEK, PEKK, etc.) have been attracting attention in recent years for their high shock absorption properties, which are expected to mitigate the impact on bone. They have also been used as dental materials in recent years due to their lightness and durability. “Nu:le Coat” can be used as a pretreatment material for PEEK, and light-cured indirect composite resin (such as “Luna-Wing”) and light and heat-cured indirect composite resin (such as “TWiNY”) can be built up on PEEK frames. The bond strength was measured when “Nu:le Coat” was used as a pretreatment material for PEEK.

• Test condition

PEEK pellets of 14 mm in length and width and 2 mm in thickness made of PEEK material were prepared. The bonding surface of the PEEK pellets was polished with P1000 abrasive paper, sandblasted with 50 μm alumina particles for 6 seconds at a pressure of 0.2 MPa, and ultrasonically cleaned in an alcohol solution. After that, a masking tape with a hole of 5 mm in diameter was applied to define the adhesive area. Three test samples were prepared: two layers of Liquid Clear were applied to the masking area, a mixture of Liquid Clear and Gel at a volume ratio of 1:1 was applied, and sandblasting alone was used as a comparison sample. For each test specimen, TWiNY’s Opaque OA3 was applied to fit the holes in the masking tape, and after polymerization, TWiNY’s Dentin DA3 was used to build up a cylindrical shape with a diameter of 5 mm and a height of 2 mm. The test samples were then placed in distilled water at 37°C for one day, and shear bond strength was evaluated by applying a load parallel to the bonding surface to the cylindrical part using the EZ-Graph universal testing machine (SHIMADZU PRECISION INSTRUMENTS, INC.)

• Test result

As shown in Figure 3-13, it has been confirmed that the shear adhesion to the resin to be built up is improved when two layers of Liquid Clear were applied as a pretreatment material for PEEK. In addition, mixing Liquid Clear and Gel at a ratio of 1:1 in advance gives it the proper viscosity as a pretreatment material and allows pretreatment with only one layer for the pretreatment material. The shear bond strength was equivalent to that of two layers of Liquid Clear.

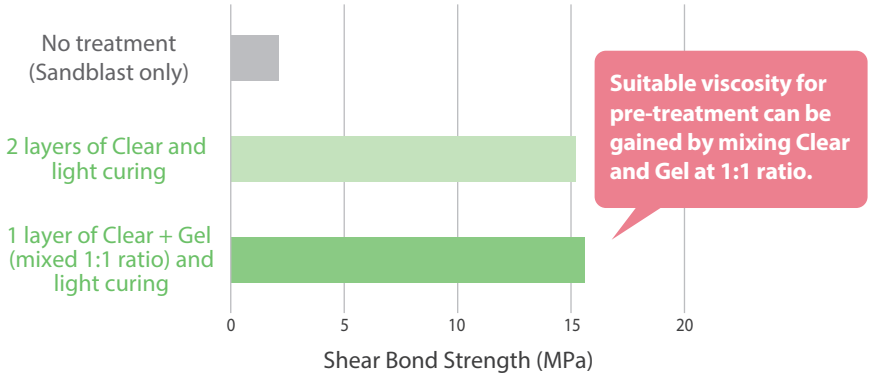


Figure 3-13 Shear bond strength of resin build-up on PEEK coated with “Nu:le Coat”

3.9. Adhesion to resin (various materials and adhesion mechanisms)

The durability of Nu:le Coat was evaluated using adhesion strength to various materials as an index.

• Test condition

The bonded surfaces of the various materials were polished with P1000 abrasive paper, sandblasted with 50 µm alumina particles at a pressure of 0.2 MPa for 6 seconds, and ultrasonically cleaned in alcohol. Then, Liquid Clear was applied and light-cured for 30 seconds, the area other than the bonded area was covered with masking tape with a 3 mm diameter hole to define the bonding area, indirect composite resin “Luna-Wing” was applied, and after light-curing, resin cement was bonded and fixed with a stainless steel rod. Figure 3-14 shows a schematic diagram of the tensile bond test. The samples were then placed in distilled water at 37°C for 1 day, and the tensile bond strength was evaluated by tensile bond test using “EZ-Graph,” the universal testing machine.

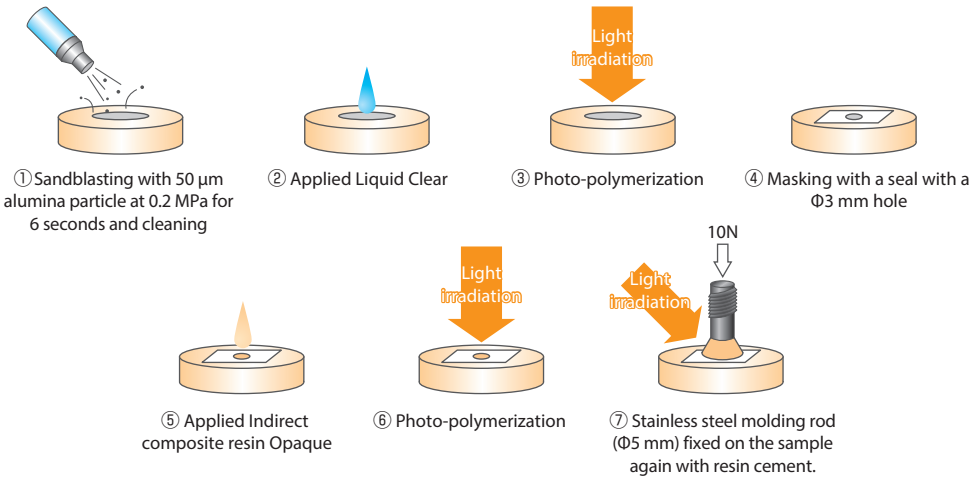


Figure 3-14 Schematic diagram of tensile bond test

To investigate the effect of MMA on various materials, the surface roughness and surface condition of the material surfaces were observed under a microscope (50x), after polishing with P1000 water-resistant abrasive paper and after immersion in MMA for 5 seconds and wiping off the MMA.

• Test result

As shown in Figure 3-15, Liquid Clear bonded to all the materials tested in this study and showed bond strengths of 5 MPa or higher, with the exception of polypropylene (PP). This indicates that the adhesive strength is durable enough.

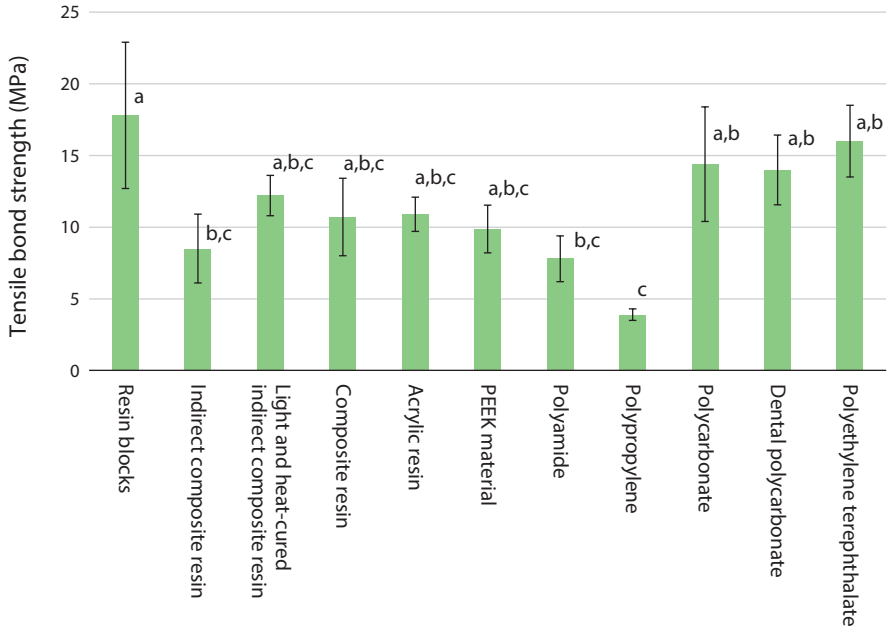


Figure 3-15 Tensile bond strength to various materials
(significant difference between different alphabets ($p<0.05$)

Figure 3-16 shows the surface roughness of various materials before and after immersion in MMA. The surface roughness of polycarbonate, Acrylic resin, and Polyethylene terephthalate was increased by immersion in MMA. All of these three materials exhibit high tensile bond strength in Figure 3-15, and it is assumed that the roughening by MMA contained in Nu:le Coat is affected by this. Resin blocks, light and heat-cured indirect composite resins, and composite resins also exhibited high tensile bond strengths of 10 MPa or higher, but no change in surface roughness by MMA was observed. For these materials, the flexural strength of the materials themselves was greater than 150 MPa, and the high strength of the materials is thought to have influenced the high bond strength by making it difficult for the materials to fracture at the bond interface. It is also suggested that the use of methacrylic acid monomers in these resin-based materials may have slightly impregnated MMA, resulting in higher adhesive strength.

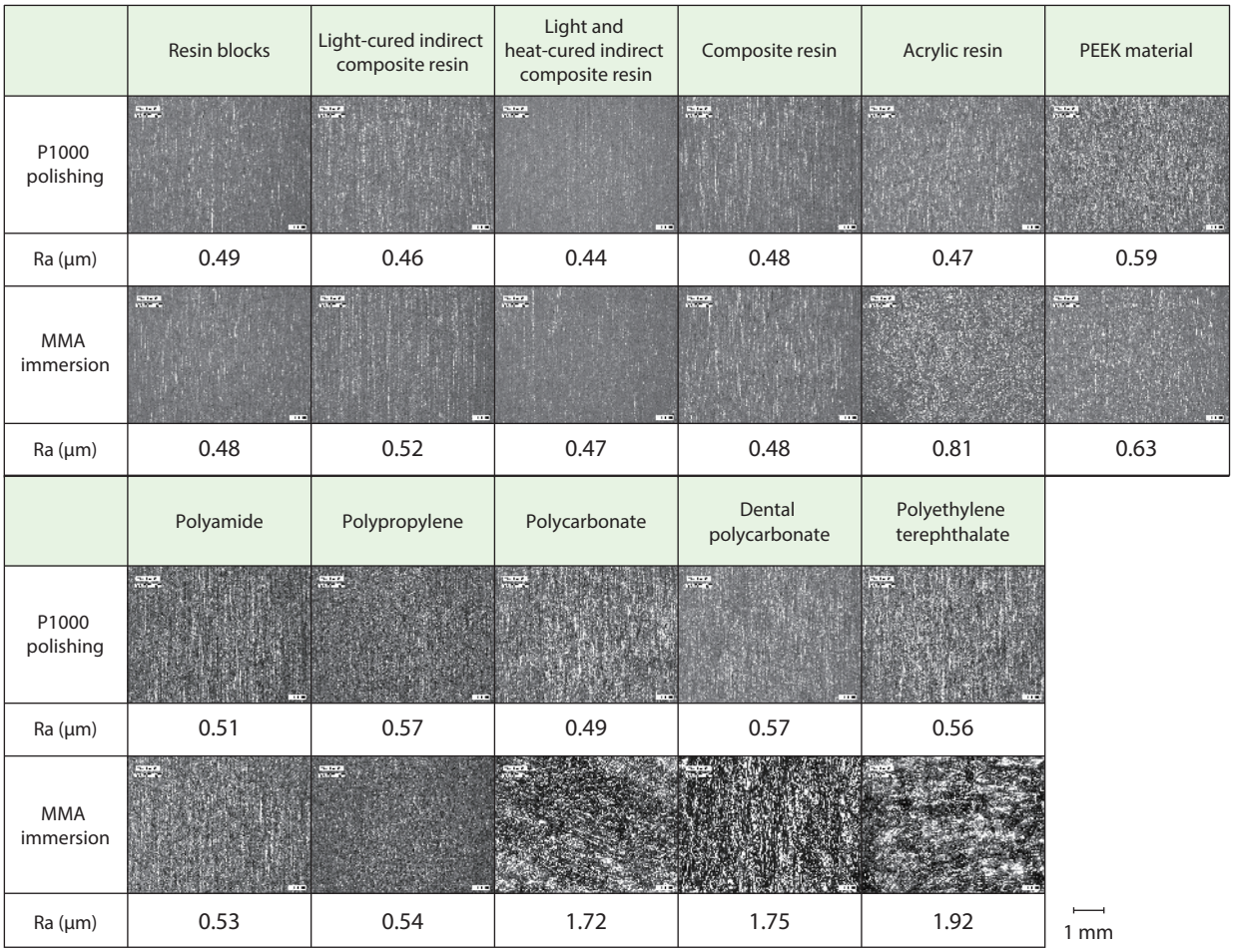


Figure 3-16 Photographs of various material surfaces before and after MMA immersion and surface roughness (Ra)

3.10. Conditions of light irradiation (wavelength, LED, halogen)

As an initiator for dental glazing materials, TMDPO with an absorption peak around 400 nm wavelength is used, as described in section 2-2. Since there are several light sources for light curing and light irradiators used in dentistry, as described in Table 3-2, commercially available light irradiators: LED (with 400 nm peak (with purple LED) and without 400 nm peak (without purple LED)) and halogen lamp (wide wavelength range) were evaluated for their adaptation to “Nu:le Coat”.

• Test condition

The 15 mm diameter, 1 mm thick molds were filled with “Luna-Wing” (E3) light-cured indirect composite resin for crowns, sandwiched between transparent PET film on both sides and light-cured from both sides for 90 seconds to prepare resin pellets. The resin pellets were then coated with a sample, and the pencil hardness was evaluated after light irradiation using “LED CURE Master”, a curing light for indirect composite resin, “ a dental LED light curing equipment(effective wavelength: 400±20 nm and 460±20 nm)”, and a dental halogen light curing unit.

• Test result

Table 3-2 Pencil hardness of “Nu:le Coat” Liquid Clear under various light exposure conditions

Type	Product name	Place of use	Polymerization time	Pencil hardness
LED (with 400 nm)	LED CURE Master	Dental laboratory	60 sec	9H
	Dental LED light curing equipment (effective wavelength: 400±20 nm and 460±20 nm)	Dental clinics	20 sec	9H
LED (without 400 nm)	Dental LED light curing equipment (effective wavelength: 460 nm)	Dental clinics	60 sec	No polymerization
Halogen lamp	Dental halogen light irradiator	Dental clinics	20 sec	No polymerization
			40 sec	9H

As a result of the test, it was confirmed that the pencil hardness of “LED CURE Master” and “a dental LED light curing equipment(effective wavelength: 400±20 nm and 460±20 nm)” became 9H by light irradiation for the specified time (60 seconds and 20 seconds, respectively) as described in the electronic attachment of “Nu:le Coat”. It was also confirmed that the halogen light irradiator did not polymerize in 20 seconds, and that light irradiation for 40 seconds resulted in a pencil hardness of 9H. The possible reason for this is that the light intensity of the halogen lamp was weaker than that of the LED lamp, and polymerization took longer. Polymerization was not confirmed even after 60 seconds of light-curing using the LED light-curing unit with an effective wavelength of 460 nm. Since “Nu:le Coat” cannot be polymerized with a light illuminator without the wavelength (400 nm) of the purple LED, it is necessary to confirm that the light illuminator has an effective wavelength and light intensity for polymerization in advance.

3.11. Viscosity and thickness

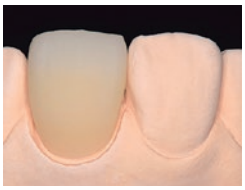
Thickness of 1 layer
- Liquid : 5-15 μm
- Gel : 20-50 μm

“Nu:le Coat” Liquid is pursued an optimal viscosity that is easy to apply. Thanks to its low viscosity design, it is hard to remain traces of brush and easy to apply thinly and evenly. It can produce a thin hard coat layer of approximately 5 μm per layer, allowing coating and characterization without affecting the surface shape of dental restorations. Also, the gel type is suitable for thicker layers. Dark brown HV (20~50 μm) is designed to be suitable for the application on the pit and fissure of crowns.

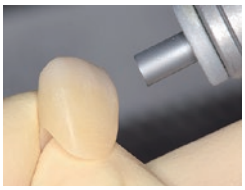
※Procedure for use at the laboratory and chairside (for use as dental glazing material)

The procedures for laboratory-side and chairside use as dental glazing material are as follows.

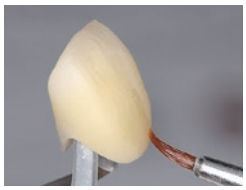
For Laboratory Use



After shape modification

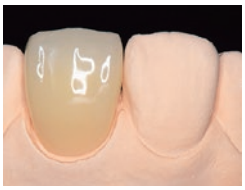


Roughen the surface
(Alumina Sandblast etc.)



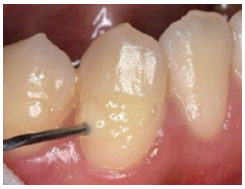
Application of Nu:le Coat
(characterizing and glazing)

Light Curing
(60 sec. by LED CURE Master)

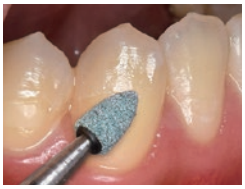


Completion

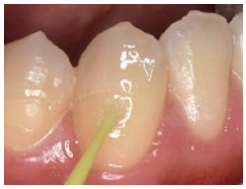
For Chairside Use



Filling composite



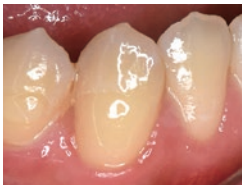
After light irradiation, shape modification and roughening the surface.



Application of Nu:le Coat
(glazing)

Light irradiation

*Light irradiators should have an output peak wavelength of 400±20 nm, and also should contain the purple LED



Completion

Point Gel is recommended to use for the parts where need thickness.

1 Apply one layer of Clear as base coat and light curing.

2 Apply Gel and light curing

For Color types, shake the bottle well before use as the colouring material may settle.
Please avoid using this product for occlusal parts where pressure is applied.

Figure 3-17 Procedure for laboratory use and chairside use

4. Usage of color types

There are White, Gray, Brown, Yellow, Orange, Blue, Red, Pink, Black, Gum, and Dark Brown HV in Nu:le Coat Liquid color types that allow for free characterization. There are A plus, B plus, C plus, and D plus lineups that allow fine adjustment of the shade. (Figure 4-1, lineup as of June 2023.)

Figure 4-2 shows an example of shade adjustment of CAD/CAM resin block “E-VA” for anterior teeth, and Figure 4-3 and Figure 4-4 show examples of characterization using color type. Dark Brown HV used in Figure 4-4 is designed to have high viscosity and can be applied with a viscosity suitable to adjust the shade of the pit and fissure.

Figure 4-1 The color line-up of Nu:le Coat and examples of shade plus use

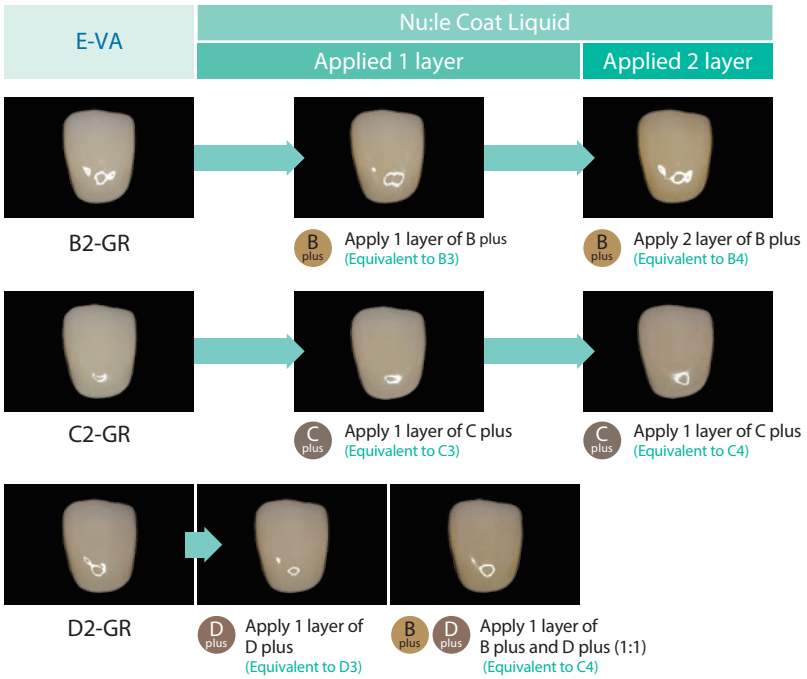


Figure 4-2 Shade adjustment of “E-VA” by shade plus

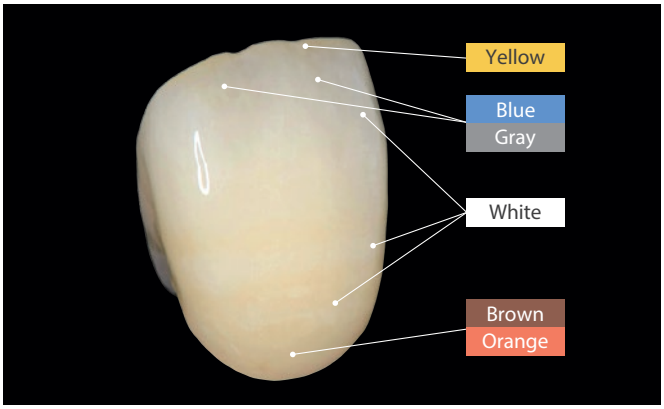


Figure 4-3 Characterization by color type

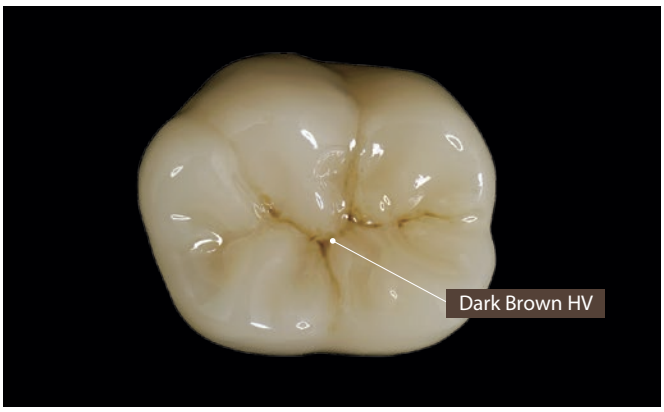


Figure 4-4 Reproduction of pit and fissure of a molar by Dark Brown HV

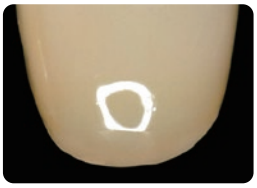
Nu:le Coat

Key points for use ①

It is possible to express a various color tone because each Liquid can be mixed. Also, by mixing Liquid Clear, you can dilute the color.

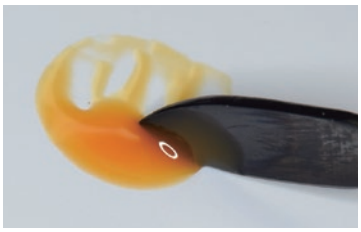
A plus
+
Liquid Clear

A plus



A plus
+
Orange

Key points for use ②

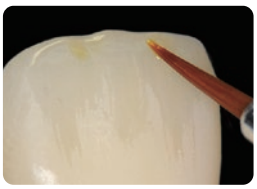


You can apply Liquid with suitable viscosity by leaving it on the mixing pad for a certain period of time.

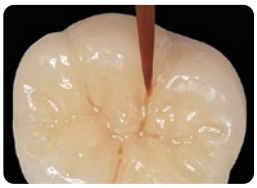
*If coloring components are separated, mix before use.
Also, it is necessary to apply and light cure (10 sec.) Liquid Clear on the material surface in advance when using Liquid with high viscosity because the adhesion to material surface is reduced. Regarding Dark Brown HV, it is designed to have high viscosity, and can be applied with viscosity suitable to adjust the shade of the pit and fissure.

Expression of white band

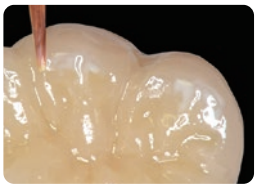
Expression of cracks
and hairline



Fine shade adjustment
around the incisal



Shade adjustment of the pit
and fissure



Shade adjustment of the
cusp apex and the margin

5. Joint use with polycarbonate

“KZR-CAD Provisional PC” (YAMAKIN CO., LTD., hereinafter referred to as Provisional PC) is a material for dental milling and machining made of polycarbonate to make a provisional restoration. Provisional restoration materials made of polycarbonate are already used in many countries, and it is recognized for their high usefulness. On the other hand, PMMA is the mainstream in Japan. Polycarbonate exhibits the highest impact resistance of all plastics.

Polycarbonate has excellent characteristics listed below[7].

1. Impact resistance: exhibits the highest impact resistance of all plastics.
2. Operate temperature range: exhibits stable characteristics in a wide temperature range (-100°C ~130°C).
3. Electrical characteristics: there is no change in the wide operating temperature range, and exhibits excellent characteristics as an insulation material.
4. Transparency and weatherability: it is the only one that has transparency among general-purpose engineering plastics, and can be used outdoors for a long time.
5. Dimension stability: it has excellent creep properties, and has very little dimension change by moisture absorption, temperature, and time.
6. Heat resistance: it has excellent heat resistance equivalent to UL Certification.

It is possible to improve the aesthetics by using “Nu:le Coat” and “Provisional PC” together. Figure 5-1 shows Provisional PC (anterior teeth) applied Liquid Clear, A plus, B plus, C plus, and D plus of shade plus.

In the usual polished finish, the workpiece is roughly polished with a paper cone, sandblasted only at the junction with the lathe, and polished with an abrasive and deerskin buff. By applying Liquid Clear, it is possible to omit the polishing process. Especially in long bridge cases, as it takes time to polish, it is expected to improve work efficiency by using Nu:le Coat. Also, it allows us to adjust the shade by using color types together. A2 of Provisional PC is designed to have a shade close to A2 of zirconia disk for cutting “KZR-CAD Zirconia” (YAMAKIN CO., LTD.). However, it is possible to shift shades or recreate gradation by using shade plus. With A plus, A2 can be adjusted to A3, and with B plus, to B3. For example, in cases of long-term wearing is required, the use of Nu:le Coat provides a more esthetics provisional restoration by adjusting shades and adding esthetics.

However, care should be taken when using Nu:le Coat on flexible materials such as Polycarbonate because of the flexibility differences between the flexible area and Nu:le Coat layer by applying various layers and making the surface thick may cause cracks and increase the risk of fracture of the material itself.

As cited below, it is specified to apply less than 3 layers in cases of a single crown and less than 2 layers in areas that are easily deformed in the Instruction for Use of Nu:le Coat.

(1) Note that when using this product on crowns and resin materials for dentures (PMMA, Polycarbonate, etc.) without fillers, applying thick layers may cause cracks (coating thickness less than 40 µm, approximately up to 3 layers).

(2) Note that when using this product on bridges made of PMMA and Polycarbonate without fillers, applying thick layers may cause cracks (coating thickness less than 30 µm, approximately up to 2 layers). Especially in areas where the product tends to accumulate.

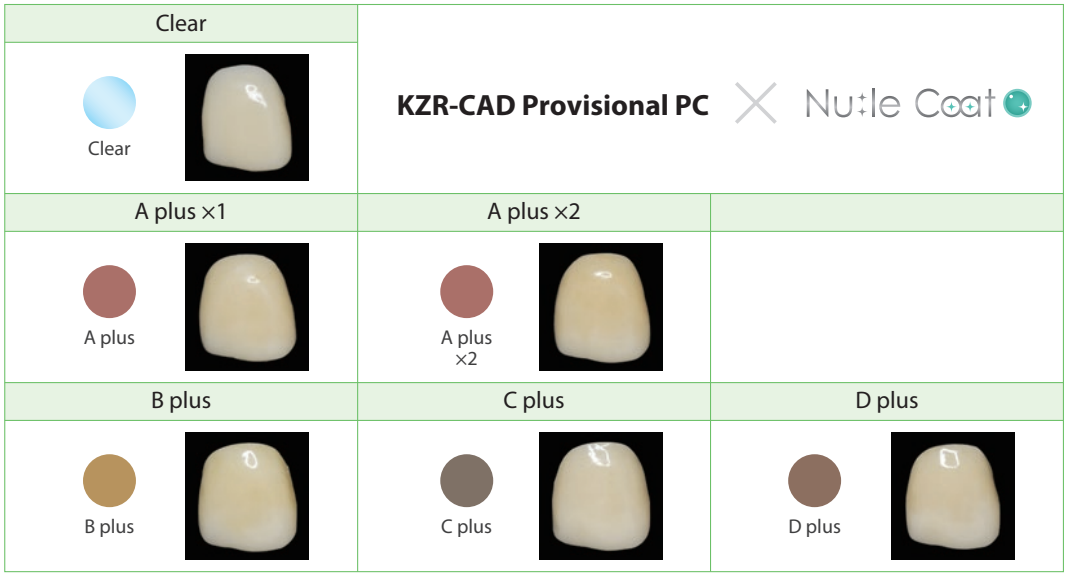


Figure 5-1 Image of shade shift of Provisional PC (A2) by Nu:le Coat Liquid

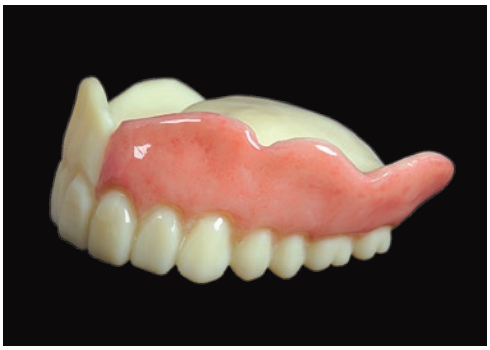


Figure 5-2 Reproduction of Gum Shade of copied denture made from Provisional PC by using Nu:le Coat Liquid Gum

6. Biological safety assessment of “Nu:le Coat”

Biological safety assessment evaluates the risk of adverse effects on human health. All medical device that comes into contact with the human body is proven to the risk is acceptable to be used in medical treatment by the evaluation of biological safety.

As shown in Table 6-1, the biological safety evaluation items to be considered for medical devices are established on the basis of the site of contact with the body (surface-contacting medical devices, externally communicating medical devices, and implant medical devices) and the contact period (limited exposure, prolonged exposure, long-term exposure).

In this chapter, test examples when the acute systemic toxicity and genotoxicity were evaluated using test samples made of raw materials as same as “Nu:le Coat” is introduced.

Table 6-1 Endpoints to be addressed in a biological risk assessment

Contact duration (accumulation): A-limited(≦24h), B-prolonged (>24h to 30 days), C-Long term (>30 days)			Endpoints of biological evaluation															
			Physical and/or chemical information	Cytotoxicity	Sensitization	Irritation or intracutaneous reactivity	Material mediated pyrogenicity	Acute systemic toxicity	Subacute toxicity	Subchronic toxicity	Chronic toxicity	Implantation effects	Hemocompatibility	Genotoxicity	Carcinogenicity	Reproductive/developmental toxicity	Degradation
Non-contact medical devices			None of the evaluations are required														
Surface medical device	Intact skin	A	×	E	E	E											
		B	×	E	E	E											
		C	×	E	E	E											
	Mucosal membrane	A	×	E	E	E											
		B	×	E	E	E		E	E			E					
		C	×	E	E	E		E	E	E	E	E		E			
	Breached or compromised surface	A	×	E	E	E	E	E									
		B	×	E	E	E	E	E	E			E					
		C	×	E	E	E	E	E	E	E	E	E		E	E		
Externally communicating medical device	Blood path, indirect	A	×	E	E	E	E	E					E				
		B	×	E	E	E	E	E	E				E				
		C	×	E	E	E	E	E	E	E	E	E	E	E	E		
	Tissue/bone/dentine	A	×	E	E	E	E	E									
		B	×	E	E	E	E	E	E			E		E			
		C	×	E	E	E	E	E	E	E	E	E		E	E		
	Circulating blood	A	×	E	E	E	E	E					E	E			
		B	×	E	E	E	E	E	E			E	E	E			
		C	×	E	E	E	E	E	E	E	E	E	E	E	E		
Implant medical device	Tissue/bone	A	×	E	E	E	E	E									
		B	×	E	E	E	E	E	E			E		E			
		C	×	E	E	E	E	E	E	E	E	E		E	E		
	Blood	A	×	E	E	E	E	E				E	E	E			
		B	×	E	E	E	E	E	E			E	E	E			
		C	×	E	E	E	E	E	E	E	E	E	E	E	E		

E means endpoints to be evaluated in the risk assessment (either through the use of existing data, additional endpoint-specific testing, or a rationale for why assessment of the endpoint does not require an additional data set). If a medical device is manufactured from novel materials, not previously used in medical device applications, and no toxicology data exists in the literature, additional endpoints beyond those marked “E” in this table should be considered. For particular medical devices, there is a possibility that it will be appropriate to include additional or fewer endpoints than indicated.

6.1. Acute systemic toxicity

Systemic toxicity test evaluates the toxicities in the organs all over the body when exposed to medical devices or raw materials. It is classified into 1) Acute systemic toxicity: adverse effects occurring at any time within 72 hours after single, multiple or continuous exposures of a test sample for 24 hours, 2) Subacute systemic toxicity: adverse effects occurring after or continuous exposure between 24 hours and 28 days, 3) Subchronic systemic toxicity: adverse effects occurring after the repeated or continuous administration of a test sample for a part of the lifespan, 4) Chronic systemic toxicity: adverse effects occurring after the repeated or continuous administration of a test sample for a major of the lifespan. In this section, acute toxicity test (intravenous/ intraperitoneal administration) in accordance with ISO 10993-11:2017, *Biological evaluation of medical devices --Part 11: Tests for systemic toxicity* is explained. Extracts of test samples were prepared by extracting at 121°C for an hour in physiological saline or sesame oil. Empty extracts were prepared in the same method for each extraction solvent. After a single dose of each extract was administered to mice, changes to general condition and body weight were measured after 72 hours, and the results were determined according to Table 6-2.

Table 6-2 Evaluation table

Acute systemic toxicity	Through the period of observation, all animals in the extract-treated group had strong biological reactions compared to the animals in the empty extract-treated group.
No acute systemic toxicity	More than 2 animals in extract treated animals die, more than 2 test animals show significant toxic symptoms such as convulsion and weakness, or more than 3 animals lost more than 10% of body weight.
Reexamination	Any of the animals in the extract-treated group show a slight biological reaction compared to the animals in the empty extract-treated group, or only one animal shows a strong biological response.

Based on the evaluation results, physiological saline or sesame oil extracts were determined as “no acute systemic toxicity” because all animals in the extract-treated group did not show strong biological reactions compared to the animals in the empty extract-treated group throughout the observation period.

6.2. Genotoxicity

Genotoxicity test determines the direct or indirect effects of medical devices or raw materials on the structure or function of DNA, or general disorders caused as a result. Genetic disorder in DNA means genetic mutation or chromosomal abnormalities or recombination, and cancer can be caused by it. Therefore, the genotoxicity test is a screening test for carcinogenic. In principle, more than 2 types of tests are required because one test cannot detect all mechanisms of genotoxicity.

In this section, it is explained reverse mutation test using bacteria and chromosome abnormality test using mammalian cultured cells compliant with ISO 10993-3:2014, *Biological evaluation of medical devices--Part 3: Tests for genotoxicity, carcinogenicity and reproductive toxicity*.

1) Reverse mutation test using bacteria

Reverse mutation test analyses if there is a property of causing genetic mutation (mutagenicity) caused by medical devices or raw materials. It used Salmonella and Escherichia coli (amino acid auxotrophic strains) with mutations in genes involved in amino acid metabolism in the test. Normally, these bacteria cannot grow on a medium that has not added amino acid since it is impossible to synthesize the amino acid necessary for growth in the bacteria. However, in cases of genetic mutation, it becomes possible to synthesize amino acids, multiply, and form colonies. Using this principle, mutagenicity is assessed by comparing the test sample with negative control (control sample without mutagenicity) colony formation.

Test samples were extracted with dimethyl sulfoxide at 37°C for 48 hours with shaking, and the resulting extract was used as the test stock solution. Substances that exhibit genotoxicity when metabolized were assessed by test conditions that do not involve the metabolic activation method that directly interacts with the test sample and the genotoxicity of the sample that was evaluated under metabolic activation conditions using a metabolic enzyme (S9mix) to detect substances that exhibit genotoxicity when metabolized.

The results showed that the extracts of the samples did not increase the number of reversion mutant colonies compared to the negative control under any of the test conditions, and therefore, the gene mutagenicity of the samples was concluded to be negative.

2) Chromosome abnormality test using mammalian cultured cells

The chromosome abnormality test evaluates mutations caused by medical devices or raw materials at the chromosome level. Chromosomes are string-like structures that store multiple pieces of generic information, and there are 46 in a normal human. Chromosome abnormalities can be divided into structural abnormalities, in which the shape of the chromosomes is abnormal, and numerical abnormalities in which the number of chromosomes changes, and both can be evaluated by observation using an optical microscope. In this test, the frequency of cells with chromosomal aberrations less than 5% was considered negative, between 5% and 10% was considered suspicious-positive, and 10% or more was considered positive. For the test, the sample were extracted with shaking for 48 hours at 37°C in the test medium, and the resulting extract was used as the test stock solution. The cells used were Chinese hamster lung fibroblasts (CHL/IU). Tests were performed with three doses of the test stock solution at 25, 50, and 100% by short-time and continuous treatment methods. The cells were cultured for 3 days after seeding and then treated with and without the metabolic enzyme (S9mix). In the short-term culture treatment method, each plate was cultured for 6 hours, then the medium in the plates was replaced with a test medium, and cultured for another 18 hours. In the continuous treatment method, the cells were cultured for 24 hours. Chromosome samples were prepared after culturing. In addition, the cells in the plates were counted at the start and at the end of the culture, and the relative increase in cell counts (RICC) was calculated.

$$\text{RICC (\%)} = \frac{\text{Increase in cell number at each test dose in test groups treated with sample and positive control}}{\text{Increase in cell number in test group treated with negative control}} \times 100$$

Chromosomal abnormalities were recorded according to the classification in Table 6-3.

Table 6-3 Classification of chromosomal abnormalities

Chromosome structural abnormalities	ctb: chromatid break cte: chromatid exchange csb: chromosome break cse: chromosome exchange Others (fragmentation, compound anomaly)
Chromosome numerical abnormalities (polyploidy)	Polyploidy* Others (endoreduplication)
Remarks	*Cells with 38 or more chromosomes were recorded as polyploidy.

As a result, the frequency of cells with chromosomal structural abnormalities and the frequency of cells with chromosomal numerical abnormalities (ploidy) in the test samples were both less than 5%, and it was concluded that the samples were negative for inducing chromosome abnormalities under the test conditions.

The above is a part of the biological safety evaluation of the safety required by laws and systems (Pharmaceuticals and Medical Devices Act) using actual samples mixed with the ingredients contained in the Liquid and Gel types of “Nu:le Coat”. In addition to the biological safety tests introduced here, “Nu:le Coat” has been evaluated for biological safety which is considered necessary in consideration of the usage form of the material, and it has been confirmed that there are no problems in any of the evaluation items.

7. Conclusion

“Nu:le Coat“ is a dental glazing material for finishing dental restorations with reduced curing strain, improved transparency, durability, flexibility in color tone with color types, and ease of application with low viscosity.

It can be also used as a pretreatment material for resin build-up on PEEK as an additional application. The aesthetic element of CAD/CAM crown materials used for anterior teeth is defined as “a layered structure of multiple shades including enamel (incisal color), dentine (cervical color), and transitional colors (intermediate colors)” in Japan[8]. However, the number of shades is limited even with a stacked structure of multiple shades that meets this definition, making it difficult to achieve optimal color expression for each individual patient. The use of “Nu:le Coat” color type enables characterization that more closely resembles natural teeth.

CAD/CAM crowns are expected to become increasingly popular in the future, and this product can streamline the finishing process. Of course, this product can be used not only for CAD/CAM crowns but also for indirect composite resin and intraoral composite resin treatment. We hope that this report will encourage the use of dental glazing material.

References

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