Development of a primer containing a silane coupling agent for zirconia /Kimura H[1], Kato T[1, 2], Saigo K[2],Yamada B[1], Yamauchi J[1], Anraku T[1] ([1] Yamamoto Precious Metal Co. Ltd., [2] Kochi University of Technology) : For firm adhesion between a dental resin and a zirconia frame, sandblasting followed by suitable priming is required. γ-Methacryloyloxypropyltrimethoxysilane was employed after sandblasting with and without heat treatment (1000 ˚C). The use of silane coupling after sandblasting was found to significantly enhance the adhesive strength. The sandblasting/heat treatment, which might induce the phase transformation of zirconia, slightly reduced the adhesive strength in comparison to treatment by sandblasting only.

[Abstract]

In recent years, the clinical application of zirconia has been increasing due to considerations of biocompatibility. In order to achieve firm adhesion between zirconia and resin when building in resin on a zirconia frame, sandblasting of the frame, followed by priming using a primer specifically suitable for use with zirconia, is required. A body of literature states that adhesion with zirconia can be improved by the use of a primer which includes phosphoric-acid monomers; however, the effectiveness of using a silane coupling agent has yet to be clarified. In this investigation, the influence of sandblasting treatment and the use of silane coupling on adhesive strength between resin and zirconia is investigated.

[Materials and Methods]

A disc of zirconia (Aadva, manufactured by GC) was used for adhesion testing. The surface was (1) polished with #600 water-resistant paper, (2) subjected to sandblasting treatment (alumina sand, particle diameter 50 μm) after polishing and (3) subjected to heat treatment at 1000˚C for 15 min after sandblasting. Adhesive strength was evaluated under the conditions obtaining after the three types of treatment above. γ -Methacryloyloxypropyltrimethoxysilane.

An ethanol solution (TR) of γ-Methacryloyloxypropyltrimethoxysilane (manufactured by Momentive Performance Materials Japan) was used as a slilane coupling agent. After the bonding plane was provided with tape with φ5 mm perforations, an opaque resin (TWiNY IvO, manufactured by Yamamoto Precious Metal) was lightly applied and curing was conducted using a photopolymerization device (LED CURE Master, Yamamoto Precious Metal). Molds of 2 mm in thickness and with φ5 mm perforations were aligned with the bonding plane and fixed with double-sided tape. An indirect composite resin (TWiNY DA3, manufactured by Yamamoto Precious Metals) was supplied and curing was conducted by means of photopolymerization. The molds were removed from the cured test specimens, and after immersion for 24 hours in distilled water at 37˚C, the test specimens were placed in a small-sized universal tester (Ez-Graph, manufactured by Shimadzu). Load was applied to the cured test specimens in a shearing direction at a rate of 1.0 mm/min.; shear adhesive strength was ascertained from test strength values obtained on fracturing.

[Results and Discussion]

Figure 1 gives adhesive strength values for zirconia under the various surface treatments applied. While almost no effectiveness was displayed under sandblasting alone, adhesive strength increased significantly (0.1 MPa → 9.7 MPa) under TR treatment. However, heat treatment following sandblasting caused adhesive strength values to decline by 23% (9.7 MPa → 7.5 MPa).

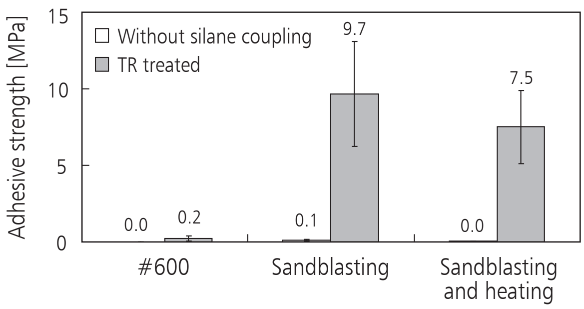

Fig. 1: Shear adhesive strength of each zirconia specimen

Table 1 shows shear adhesive strengths obtained between the bonding surface and TR after polishing and heat treatment.

Table 1. Surface area and adhesive strength

Treatment	Surface area* (μm^2)	Adhesive strength (S.D.) (MPa)
#600 sand paper	0.01690	0.2 (0.2)
Sandblasting	0.02947	9.7 (3.4)
Sandblasting/heating	0.02967	7.5 (2.4)

Surface area*= Relative to field of vision (128μm x 128μm = 0.01638μm^2)

While adhesive strength increases in line with the increase in surface area caused by sandblasting treatment, adhesive strength values decline under heat treatment regardless of the fact that the surface area undergoes no significant change. A surface hydroxyl group on the target bonding surface is necessary for silane coupling[2)] ; it is thought that heat treatment at high temperatures impacts zirconia in such a way as to decrease the surface hydroxyl volume. There is also the possibility that for partially stabilized zirconia, which is widely used as a dental material, the stresses resulting from sandblasting may induce a phase transformation of the surface crystals from tetragonal to monoclinic crystals, with reversion to tetragonal form under heat treatment at 1,000°C and over. It is thought that this kind of phase transformation has an influence on the adhesive strength.

[Conclusion]

This investigation demonstrates that, for zirconia which has undergone sandblasting treatment, silane coupling improves adhesion with resin.

[Reference]

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