

Original Article

Effects of tetrabutylammonium dihydrogen trifluoride etchant on bond strengths of resin composites with Ti-6Al-4V and Co-Cr alloysKohji Kamada¹⁾, Yohsuke Taira²⁾, Takafumi Egoshi²⁾, and Kei Kaida²⁾¹⁾Oral Management Center, Nagasaki University Hospital, Nagasaki, Japan²⁾Division of Cariology and Restorative Dentistry, Department of Prosthetic Dentistry, Graduate School of Biomedical Sciences, Nagasaki University, Nagasaki, Japan

Received January 5, 2023; Accepted April 5, 2023; J-STAGE Advance Publication: May 31, 2023

Abstract

Purpose: This study aimed at evaluating the effects of surface treatments with tetrabutylammonium dihydrogen trifluoride (TDTF) on the bond strengths of indirect resin composites with titanium-aluminum-vanadium (Ti-6Al-4V) and cobalt-chromium (Co-Cr) alloys.

Methods: Disk-shaped Ti-6Al-4V and Co-Cr alloy specimens were air-abraded with alumina, treated with an etchant (MEP) containing TDTF for 10 s (MEP10) or 30 s (MEP30), and rinsed with water. Subsequently, a primer containing 6-methacryloyloxyhexyl phosphonoacetate was applied to the surfaces, and the specimens were veneered with a light-curing indirect resin composite. Specimens without MEP were prepared as controls (no-MEP). Shear bond strengths were determined before or after 100,000 thermocycles, and the data were analyzed using the Steel-Dwass test ($\alpha = 0.05$, $n = 10$).

Results: No significant difference was found in the bond strengths between the Ti-6Al-4V and Co-Cr alloys. In each metal alloy, the MEP10 and MEP30 specimens exhibited higher bond strengths than the no-MEP controls after 100,000 thermocycles. Scanning electron microscopy observations revealed that submicron-pits and crevices were formed on both the metal alloys upon applying the MEP etchant.

Conclusion: Surface treatments with TDTF following air abrasion are useful for improving bonding durability while veneering resin composites on Ti-6Al-4V or Co-Cr alloy frameworks.

Keywords: adhesion, base metal alloy, surface modification

Introduction

Titanium-aluminum-vanadium (Ti-6Al-4V) and cobalt-chromium (Co-Cr) alloys are used for metal frameworks of implant-supported prostheses, fixed partial dentures, and removable dentures [1-5]. In addition to conventional casting techniques, computer-aided design-computer-assisted manufacturing (CAD-CAM) technologies with machine-milling or laser-sintering systems have facilitated the fabrication of these prostheses. The fabricated metal frameworks are often veneered with tooth- or gingiva-colored resin-based materials to satisfy the esthetic demands of patients. However, conventional bonding systems are not always sufficient to prevent local detachments of resin-based materials from metal frameworks, microleakages, discolorations, or staining at metal-resin interfaces [6,7]. Therefore, durable bonding is indispensable for prostheses to function in long-term intraoral use.

The application of primers containing functional monomers following air abrasion is a standard technique to improve the bonding of resins to metal alloys for dental applications. Certain acidic functional monomers, such as 4-methacryloyloxyethyl trimellitate anhydride (4-META) [8], 10-methacryloyloxydecyl dihydrogen phosphate (MDP) [9], and 6-meth-

acryloyloxyhexyl phosphonoacetate (6-MHPA) [10], have been developed for bonding base metal alloys. With regard to the modification techniques of Ti-6Al-4V alloy surfaces, primer applications following air abrasion [6,11], silica coating [12,13], plasma exposure [14,15], and chemical etching with alkaline [16] or fluoride-gel [17] have been evaluated. As for Co-Cr alloys, primer applications following air abrasion [9,18-20], silica coating [21,22], spark erosion [23], Nd:YAG laser irradiation [24], and plasma exposure [15] have been reported. Among these surface treatment methods, chemical etching and primer applications have the advantage of easy handling, low cost, short treatment time, and no requirement for special equipment.

A marketed etchant (Monobond Etch & Prime, Ivoclar Vivadent AG, Schaan, Liechtenstein) (designated as MEP) containing tetrabutylammonium dihydrogen trifluoride (TDTF, Fig. 1) is a clinically acceptable product used for bonding glass ceramics. In previous studies, the chemical etching of commercially pure titanium with the MEP etchant [25] or Ti-6Al-4V alloy with ammonium hydrogen fluoride [26] significantly improved bond strengths. The MEP etchant is considered to be effective in bonding Ti-6Al-4V alloys because both the ammonium hydrogen fluoride and TDTF are hydrogen fluoride derivatives. The surface of base metal alloys can be regarded as metal oxides rather than a metallic phase in the atmosphere. Additionally, it is assumed that hydrogen fluoride ions released from the fluorides dissolve the metal oxide layer as well as ceramics. However, further information is required regarding whether surface treatments using TDTF improve the bonding between resin-based materials and various metal alloys.

Therefore, the present study aimed to evaluate the effects of TDTF on the durability of the bond strengths between indirect resin composites and Ti-6Al-4V or Co-Cr alloys. The null hypothesis is that the surface treatments of these metal alloys with TDTF do not affect bond strengths.

Material and Methods**Materials**

The metal alloys, etchant, primer, and resin composite used in this study are summarized in Table 1. Sixty Ti-6Al-4V alloy disks (diameter = 10 mm; thickness = 2.5 mm) were cut from a rod using a diamond saw (IsoMet Low Speed Saw, Buehler, Lake Bluff, IL, USA). In addition, 60 Co-Cr alloy disks (diameter = 10 mm; thickness = 2.5 mm) were prepared using a laser sintering system (EOSINT M270, EOS GmdH, München, Germany) and Co-Cr powder (EOS CobaltChrome SP2, EOS GmdH). All metal specimens were ground with #600 and #1200 silicon carbide papers (Buehler) and air-abraded (Pen-Blaster, Shofu Inc., Kyoto, Japan) with alumina (Hi-Alumina, Shofu Inc., average grain size 50-70 μm) for 10 s. The air pressure for the air abrasion was 0.4 MPa, and the distance from the nozzle to the metal surface was 10 mm.

The Ti-6Al-4V and Co-Cr alloys were divided into three groups (MEP10, MEP30, and no-MEP) of 20 specimens each. For the MEP10 and MEP30 groups, the MEP etchant was applied to the specimen surface with disposable brush applicators (Shofu Inc.) for 10 s (MEP10) and 30 s (MEP30), respectively. This was followed by rinsing with a water spray for 30 s, ultrasonic cleaning with distilled water for 5 min, and air-drying. The specimens not treated with the MEP etchant were prepared as controls (no-MEP).

Preparation of bonded specimens

After surface treatment, a piece of 50- μm -thick masking tape with a 4-mm-diameter circular hole was attached to the metal specimen to define

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the bonding area. A primer (Metal Link, Shofu Inc.) and opaque resins (Ceramage Duo Pre-Opaque and Opaque, Shofu Inc.) were applied to the metal specimen in this order. The applied opaque resins were light-cured for 30 and 90 s, respectively, using a laboratory light-emitting diode unit (Labocure L, GC Corp., Tokyo, Japan). An acrylic ring (inner diameter = 6 mm; height = 2 mm) was placed around the bonding area. The acrylic ring was filled with a resin composite (Ceramage Duo Body resin, Shofu Inc.), followed by light-curing for 150 s.

Shear bond strength test

After 30 min from preparation, the bonded specimens were immersed in distilled water at 37°C for 24 h. The shear bond strengths of the 60 specimens (30 specimens × 2 metal alloys) were determined (designated as thermocycle 0) using a shear testing jig (No. ISO/TR11405, Wago Ind., Nagasaki, Japan) and universal testing machine (AGS-10kNG, Shimadzu Corp., Kyoto, Japan) at a cross-head speed of 0.5 mm/min. The remaining 60 specimens (30 specimens × 2 metal alloys) were subjected to 100,000 thermocycles using a thermocycling machine (Higuchi Corp., Nagasaki, Japan) and subsequently subjected to the shear bond strength test (designated as thermocycle 100,000). The thermocycle comprised two sequential 1-min water baths held at 4 and 60°C, respectively.

Failure mode observation

After the shear testing, the metal alloy and resin composite surfaces of the debonded specimens were observed using an optical microscope (SMZ-10, Nikon Corp., Tokyo, Japan) at a magnification of 20× to determine the type of bond failure. The failure mode was categorized as an adhesive failure at the metal alloy-resin composite interface (A), cohesive failure within the

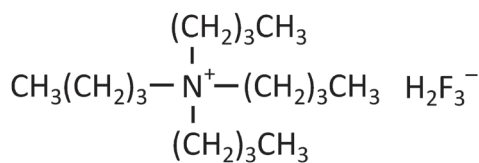


Fig. 1 Chemical structure of tetrabutylammonium dihydrogen trifluoride

resin composite (C), or a mixture of these failure modes (A/C).

Scanning electron microscopy (SEM)

An additional six metal specimens (three Ti-6Al-4V and Co-Cr alloys each) including two controls were prepared for micro-photographic observation. The specimens were air-abraded with alumina and treated with the MEP etchant for 0 s (no-MEP), 10 s (MEP10), or 30 s (MEP30) as described above. The surfaces were sputter-coated with gold (Ion Coater IB-3, Eiko Engineering Co., Ltd., Hitachinaka, Japan) and observed using a scanning electron microscope (JCM-6000Plus, JEOL Ltd., Tokyo, Japan) at 2,000× and 8,000× magnifications.

Statistical analysis

The arithmetic median and interquartile range of 10 specimens were calculated for each test group. The data were compared using the Steel-Dwass test at a statistical significance of 0.05. The statistical analyses were performed using a software system (JMP Pro Ver. 15.0, SAS Institute Japan Ltd., Tokyo, Japan).

Results

Shear bond strength

No significant difference was observed in the bond strengths between the Ti-6Al-4V and Co-Cr alloys. The arithmetic median ranged from 17.1 to 29.7 MPa (Table 2). In each metal alloy, no significant difference was observed among the no-MEP, MEP10, and MEP30 at thermocycle 0. The bond strength for the no-MEP group significantly decreased after 100,000 thermocycles, while the values for the MEP10 and MEP30 groups were maintained after 100,000 thermocycles. The *P*-values calculated for comparison of bond strengths at thermocycle 0 and 100,000 cycles were 0.0146 and 0.0044 for the no-MEP/Ti-6Al-4V and no-MEP/Co-Cr groups, respectively.

Failure mode

The failure modes observed after the shear bond strength tests are listed in Table 3. All specimens exhibited complete cohesive failures (C) at thermocycle 0, while most of the specimens shifted from the C mode to the A

Table 1 Metal alloys, etchant, primer, and resin composites used in this study

Name (Abbreviation)	Manufacturer	Composition	Batch No.
<Metal alloy>			
Ti-6Al-4V alloy	Kobe Steel Ltd., Kobe, Japan	Ti, ≥89.11%; Al, 6.13-6.15%; V, 4.16-4.30%; O, 0.15-0.18%; Fe, 0.18-0.22%; H, 0.0017-0.0089%; N, 0.003-0.004%; C, 0.027%	
Co-Cr alloy	EOS GmdH, München, Germany	EOS CobaltChrome SP2: Co, 62-66%; Cr, 24-26%; Mo, 5-7%; W, 4-6%; Si, <1.5%; Mn, <1.5%; Fe, <0.7%	
<Etchant>			
Monobond Etch & Prime (MEP)	Ivoclar Vivadent AG, Schaan, Liechtenstein	tetrabutylammonium dihydrogen trifluoride (TDTF), butanol, methacrylated phosphoric acid ester, bis-(triethoxysilyl)ethane	Y27773
<Primer>			
Metal Link	Shofu Inc., Kyoto, Japan	10-MDDT, 6-MHPA, acetone	101923
<Resin composite>			
Ceramage Duo	Shofu Inc.	Pre-Opaque: UDMA, silica powder, pigment, photoinitiator Opaque A3O: UDMA, silica powder, pigment, photoinitiator Body resin A3B: UDMA, urethane diacrylate, zirconium silicate, pigment	091905 081904 091907

10-MDDT, 10-methacryloyloxydecyl-6,8-dithiooctanate; 6-MHPA, 6-methacryloyloxyhexyl phosphonoacetate; UDMA, urethane dimethacrylate

Table 2 Arithmetic median (interquartile range) of shear bond strength (MPa)

Group name	Ti-6Al-4V alloy			Co-Cr alloy		
	thermocycle 0	thermocycle 100,000	<i>P</i> -value**	thermocycle 0	thermocycle 100,000	<i>P</i> -value**
no-MEP	28.1 (26.0-31.2) ^{a*}	20.8 (19.4-22.3) ^b	0.0146	29.7 (25.1-31.1) ^{a*}	17.1 (10.0-20.9) ^b	0.0044
MEP10s	28.5 (25.4-31.5) ^a	25.2 (23.8-31.9) ^{ab}	0.9447	27.6 (25.5-31.0) ^a	24.4 (22.0-28.2) ^a	0.5817
MEP30s	27.2 (25.7-28.1) ^a	29.0 (25.7-30.2) ^a	0.8127	28.1 (25.6-32.0) ^a	26.6 (21.7-29.3) ^a	0.7045

*Identical small or capital letters indicate values that are not statistically different (*P* ≥ 0.05). ***P*-values calculated for comparison of shear bond strengths at thermocycle 0 and 100,000 cycles

Table 3 Failure mode (number of specimens)

Group name	Ti-6Al-4V alloy		Co-Cr alloy	
	thermocycle 0	thermocycle 100,000	thermocycle 0	thermocycle 100,000
no-MEP	C (10)	A (1), A/C (9)	C (10)	A (1), A/C (9)
MEP10s	C (10)	A/C (4), C (6)	C (10)	A/C (5), C (5)
MEP30s	C (10)	A/C (2), C (8)	C (10)	A/C (6), C (4)

A, adhesive failure at metal-opaque resin interface; C, cohesive failure within resin composite; A/C, mixed failure of A and C

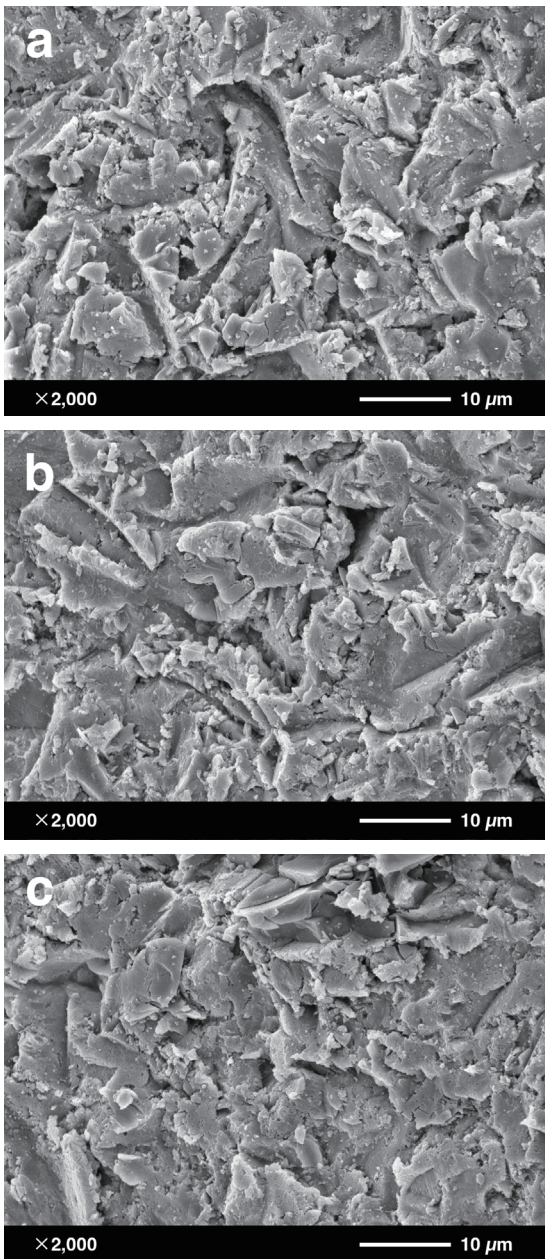


Fig. 2 Scanning electron microscopic images of the Ti-6Al-4V alloy specimen surfaces: (a) air-abraded with alumina; (b) modified with MEP etchant for 10 s after air abrasion; (c) modified with MEP etchant for 30 s after air abrasion (Original magnification $\times 2,000$)

or A/C mode after 100,000 thermocycles. For the no-MEP at thermocycle 100,000, no specimen exhibited the C mode.

SEM observation

Representative SEM images of the Ti-6Al-4V and Co-Cr surfaces are shown in Figs. 2-5. Although there were no clear differences in the surface texture for all images (Figs. 2a-c, 3a-c) at $2,000\times$ magnification, characteristic submicron features were observed at higher magnification of $8,000\times$ (Figs. 4a-c, 5a-c). In the Ti-6Al-4V alloy, the no-MEP specimen (Fig. 4a) was scratched with the alumina particles to form V-shaped smooth grooves on the surface. When treated with the MEP etchant (Fig. 4b, c), the Ti-6Al-4V alloy exhibited sponge-like surface irregularities in addition to localized pits and deepened crevices. As for the Co-Cr alloy, as well as the Ti-6Al-4V alloy (Fig. 4a), no-MEP specimen showed V-shaped smooth grooves (Fig. 5a), whereas the MEP10 and MEP30 specimens (Fig. 5b, c) exhibited deepened crevices and microcavities forming undercuts.

Discussion

The results of this study revealed that the application of an etchant,

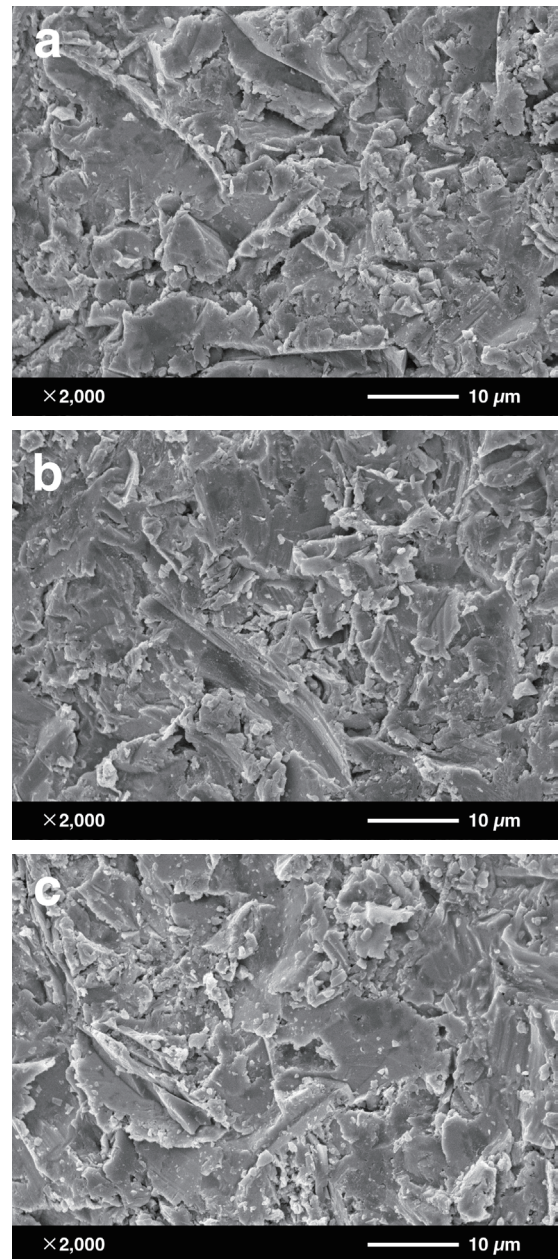


Fig. 3 Scanning electron microscopic images of Co-Cr alloy specimen surfaces: (a) air-abraded with alumina; (b) modified with MEP etchant for 10 s after air abrasion; (c) modified with MEP etchant for 30 s after air abrasion (Original magnification $\times 2,000$)

MEP, containing TDTF significantly improved the durability of the bond strengths between the resins and the Ti-6Al-4V and Co-Cr alloys. After 100,000 thermocycles, the observed failure modes for the MEP10 and MEP30 specimens suggest higher bond strengths than the no-MEP specimens. Therefore, the null hypothesis was rejected.

Primers containing phosphoric or phosphonic monomers (i.e., MDP or 6-MHPA) promote bonding to air-abraded Ti-6Al-4V and Co-Cr alloys [6,7,9,11,19,20,26,27]. The MEP etchant and the Metal Link primer used contained a phosphate monomer and 6-MHPA, respectively. However, the present and previous findings indicate that no durable bonding was achieved using the phosphoric or phosphonic monomer-based primers without additional surface modifications [26,27].

The air-abraded Ti-6Al-4V alloy surface should be a contaminated surface containing titanium oxides and aluminum oxides [6]. In the presence of fluorides, titanium-bound oxygen replaces fluorine to form titanium-fluoride compounds [28]. As reported previously, acidulated phosphate fluoride or ammonium hydrogen fluoride reacts with Ti-6Al-4V alloys to generate micro-roughened surfaces [17,26]. When the concentration of hydrogen fluoride in an aqueous solution was higher than 30 ppm, the passive film of the titanium was destroyed [29]. Taking the aforementioned

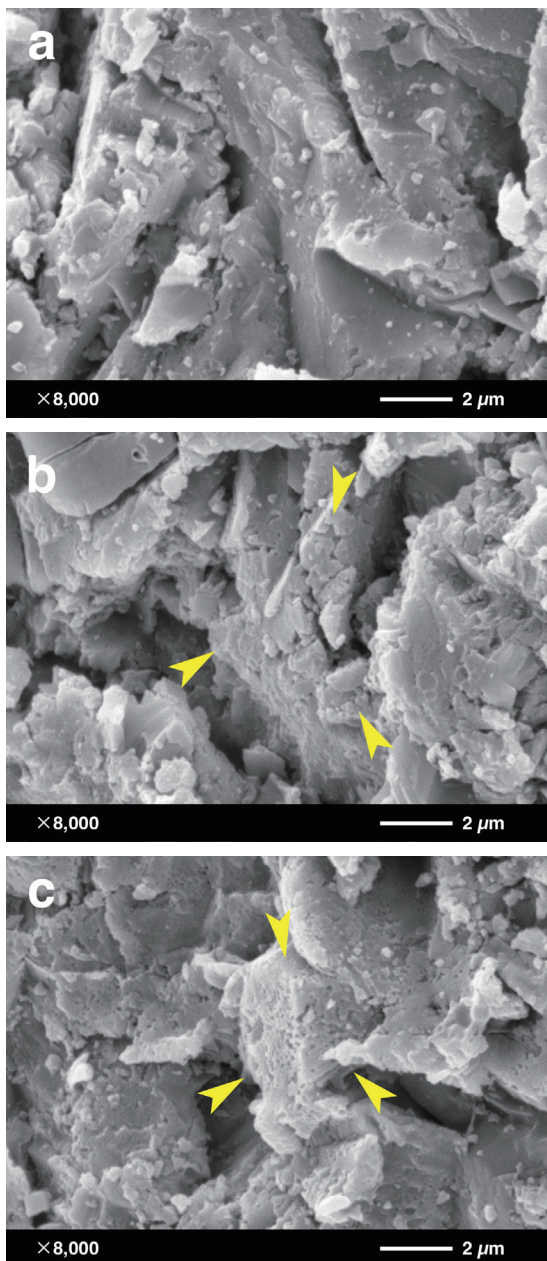


Fig. 4 Scanning electron microscopic images of the Ti-6Al-4V alloy specimen surfaces: (a) air-abraded with alumina; (b) modified with MEP etchant for 10 s after air abrasion; (c) modified with MEP etchant for 30 s after air abrasion. Arrows indicate sponge-like surface irregularities (Original magnification $\times 8,000$).

reports into consideration, it is thought that H_2F_3^- ion released from TDTF was chemically etched onto the Ti-6Al-4V alloy surface.

On the other hand, when fabricating the disk-shaped Co-Cr alloy specimen, the Co-Cr alloy microparticles were sintered with approximately 20- μm layer thickness and repeatedly layered using the Yb-fiber laser according to the manufacturer's instructions. Although the Co-Cr alloy surface is known to be covered with a passive oxide film in the atmosphere [30], the observed deepened crevices and microcavities on the Co-Cr alloy specimens (Fig. 5b, c) enable us to speculate that the sintered microparticle interface was etched by TDTF, thereby increasing the actual surface area and micro-mechanical retention.

However, the evidence in this study does not exclude the contribution of chemical bonding. For Co-Cr alloy, three bonding mechanisms related to functional monomers have been proposed in the order of descending adhesive force: 1) ionic bonding with the metallic phase, 2) hydrogen bonding to the hydroxyl groups of the passive film, and 3) dispersion bonding to the hydrated components of the oxide layer [30]. If the excess oxide layer is removed by the pre-treatment, chemical bonding could be an additional explanation for the increased bond strength. Therefore, further studies are

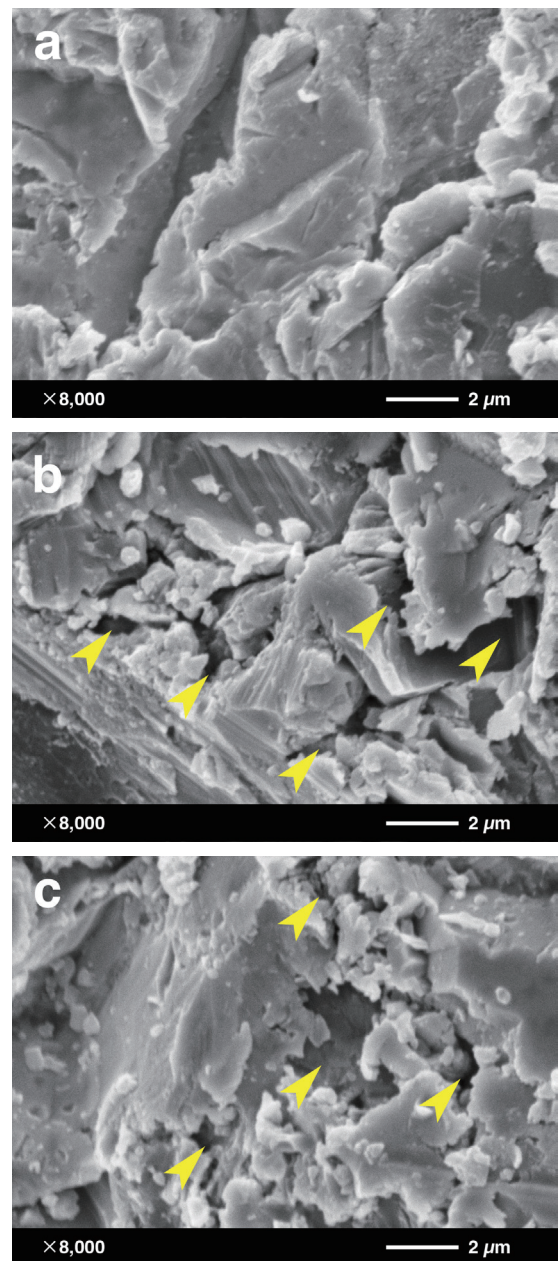


Fig. 5 Scanning electron microscopic images of Co-Cr alloy specimen surfaces: (a) air-abraded with alumina; (b) modified with MEP etchant for 10 s after air abrasion; (c) modified with MEP etchant for 30 s after air abrasion. Arrows indicate deepened crevices and microcavities (Original magnification $\times 8,000$).

required to reveal the details of the chemical interactions between TDTF and the Ti-6Al-4V and Co-Cr alloy surfaces.

The thermal cycling test of 10,000 cycles, which is 1/10th of that conducted in this study, corresponds to approximately one year of evaluation in the oral cavity [31]. The combined use of air abrasion, the MEP etchant, and the primer containing 6-MHPA may reduce the detachments of veneered resin composites from metal frameworks and microleakages over a longer term. When fabricating resin-composite-veneered prostheses, the applied MEP etchant should be thoroughly rinsed from the substrate surfaces in the laboratory and never used directly in the oral cavity as TDTF is a deleterious substance.

Within the scope of this study, it is concluded that the durability of the bond strengths between indirect resin composites and air-abraded Ti-6Al-4V and Co-Cr alloys were significantly improved with the use of an etchant containing TDTF.

Conflict of interest

The authors declare no conflicts of interest.

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