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Effects of alumina air-abrasion and acidic priming agents on bonding between SUS XM27 steel and auto-polymerizing acrylic resin

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Abstract: The purpose of this study was to evaluate the effects of functional monomers contained in the primers, as well as alumina particle abrasion on bonding between stainless steel and acrylic resin. SUS XM27 steel was primed with one of the following materials; Alloy Primer, Estenia Opaque Primer, M. L. Primer, and Super-Bond Liquid. Steel disks were either ground flat or alumina-blasted, primed with one of the four agents, and bonded with an acrylic resin (Unifast Trad). Bond strength was determined both before and after thermocycling (2,000 or 20,000 cycles). Among the four priming agents, the Alloy Primer and Estenia Opaque Primer, both of which contain 10-methacryloyloxydecyl dihydrogen phosphate (MDP), exhibited better bonding performance than the others. Alumina air-borne particle abrasion considerably improved the durability of bonding between the steel and the resin material. It can be concluded that alumina blasting followed by priming with an MDP agent is recommended for bonding the resin and SUS XM27 steel. (J. Oral Sci. 49, 191-195, 2007)

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Introduction

Over the last decade, the application of magnetic attachment systems to prosthodontic appliances has increased substantially, probably due to development of high-quality magnetic assemblies and keepers (1-3). The keepers are currently either cast-bonded or bonded with adhesive to root caps. Also, magnetic assemblies surrounded by magnetized steel are embedded in acrylic denture bases with resin-based materials. SUS XM27 (XM27) steel is used for making both magnetic assemblies and keepers due to its proper balance of attractive force and corrosion resistance.

The adhesive bonding of magnetic steels have been reported in literature (4-8). Acidic primers proved to be effective in bonding various steels (4-8), whereas other compounds such as thione, were ineffective in bonding the XM27 steel (8). The attachment and the denture base material should be strongly bonded to avoid detachment of the magnetic assembly from dentures. Although various adhesive techniques have been introduced for bonding steels (9-13), limited information is available concerning the combined effect of alumina air-abrasion and application of acidic primers for retaining magnetic attachments with acrylic resins. This study evaluated the effect of alumina air-borne particle abrasion as well as acidic priming agents on bonding between XM27 steel and auto-polymerizing acrylic resin using four acidic primers with different functional monomers.

Materials and Methods

A stainless steel designed for prosthodontic magnetic attachment systems (SUS XM27; abbreviated as XM27, Hitachi Metals Ltd., Tokyo, Japan) was selected as the adherend material. Four metal priming agents were used; Alloy Primer (Kuraray Medical Inc., Tokyo, Japan), Estenia Opaque Primer (Kuraray Medical Inc.), M. L. Primer (Shofu Inc., Kyoto, Japan), and Super-Bond Liquid (Sun Medical Co., Ltd., Moriyama, Japan), all of which were single liquid systems with functional monomer. An auto-polymerizing acrylic resin (Unifast Trad, GC Corp., Tokyo, Japan) was selected as the resin material. Information on the materials is summarized in Table 1.

A total of 160 steel disks (10 mm in diameter by 2.5 mm thick) were prepared, wet-ground with 1,500 grit silicon-carbide abrasive paper, and ultrasonically cleaned in acetone. One half of the ground disks (80) were subsequently air-abraded with alumina (Hi-Aluminas, Shufu Inc.). Both groups of 80 disks were divided into five sets (four primers and an unprimed control) of 16 specimens each. A piece of double-coated tape with a circular hole of diameter 5 mm was placed on the disk specimen surface to define the bond area. The four sets of 16 pairs were primed with one of the four priming agents, whereas the remaining 16 pairs were left unprimed and considered as the controls. A brass ring of 6 mm inner diameter, with 2 mm height and a 1-mm-thick wall, was positioned over the 5-mm-diameter circular hole. The brass ring was filled with Unifast Trad auto-polymerizing resin using a brush-dip technique.

After 30 min, the prepared specimens were stored in distilled water at 37°C for 24 h. This state was defined as 0 thermocycle. Forty of the ground specimens as well as forty of the alumina-blasted specimens (five priming conditions of eight specimens each) were tested at this stage. The remaining 40 of the ground specimens (five sets of eight specimens each) were subsequently thermocycled between 5°C and 55°C in a water bath for 2,000 cycles with a dwell time of 60 sec per bath (Thermal Shock Tester TTS-1 LM, Thomas Kagaku Co., Ltd., Tokyo, Japan). The remaining 40 of the alumina-blasted specimens (five sets of eight specimens each) were also thermocycled in identical conditions except that the number of thermocycles was set at 20,000. Each specimen was placed in a steel mold and seated in a shear test jig (ISO TR 11405) (14). Shear bond strengths were determined with a mechanical testing device (Type 5567, Instron Corp., Canton, MA, USA) at a crosshead speed of 0.5 mm/min. After the shear bond testing, the debonded surfaces were observed through an optical microscope (8×; SZX9, Olympus Corp., Tokyo, Japan).

For each group of eight replications, the average shear bond strength and standard deviation were calculated. The results were primarily analyzed by Levene test for evaluation of equality of variance (SPSS 14.0, SPSS Japan Inc., Tokyo, Japan). When the results of the Levene test did not show homoscedasticity in at least one category, Kruskal-Wallis test was performed separately for both the pre- and post-thermocycling groups to evaluate the difference among primer variations at $\alpha = 0.05$. On the basis of the results of Kruskal-Wallis test, Steel-Dwass multiple comparisons (KyPlot 4.0, KyensLab Inc., Tokyo, Japan) were further applied to compare the difference among five priming conditions in each of the pre- and post-

Table 1 Materials assessed

Material/Trade name	Manufacturer	Lot number	Composition
Stainless steel			
SUS XM27	Hitachi Metals Ltd., Tokyo, Japan		Fe 72, Cr 26, Others 2, mass%
Primer			
Alloy Primer	Kuraray Medical Inc., Tokyo, Japan	0214AA	MDP, VTD, Acetone
Estenia Opaque Primer	Kuraray Medical Inc.	0140AA	MDP, Solvent
M. L. Primer	Shofu Inc., Kyoto, Japan	080514	10-MDDT, 6-MHPA, Acetone
Super-Bond Liquid	Sun Medical Co., Ltd., Moriyama, Japan	GG5	4-META, MMA
Auto-polymerizing acrylic resin			
Unifast Trad	GC Corp., Tokyo, Japan	0507122	Powder; PMMA, PEMA
		0508051	Liquid; MMA, *N,N*-dimethyl-*p*-toluidine

MDP, 10-methacryloyloxydecyl dihydrogen phosphate; VTD, 6-(4-vinylbenzyl-*n*-propyl) amino-1,3,5-triazine-2,4-dithione, -dithiol tautomer; 10-MDDT, 10-methacryloyloxydecyl 6,8-dithiooctanoate; 6-MHPA, 6-methacryloyloxyhexyl phosphonoacetate; 4-META, 4-methacryloyloxyethyl trimellitate anhydride; MMA, Methyl methacrylate; PMMA, Poly(methyl methacrylate); PEMA, Poly(ethyl methacrylate).

thermocycling conditions with the value of statistical significance also set at 0.05. Difference between the pre- and post-thermocycling bond strengths within an identical surface preparation was analyzed with Mann-Whitney *U* test at $\alpha = 0.05$ level.

Results

Kruskal-Wallis test showed that χ^2 values were 27.442 (ground) and 19.254 (alumina-blasted) for the pre-thermocycling group and 35.575 (ground) and 35.305 (alumina-blasted) for the post-thermocycling group. The *P*-value was less than 0.05 for both pre- and post-thermocycling bond strengths, hence the pre- and post-thermocycling results for both surfaces were separately analyzed with the Steel-Dwass multiple comparisons.

The results of shear bond testing for the ground steel specimens are summarized in Table 2. The average bond strength varied from 0.2 to 33.7 MPa. Mann-Whitney *U* test run on the bond strength results showed that reduction in bond strength after thermocycling was statistically significant for all five priming conditions (Table 2). The unprimed control group resulted in the lowest bond strength both before (category a) and after (category d) thermocycling. Significant differences in bond strength were not found among the Alloy Primer, Estenia Opaque Primer, and M. L. Primer groups before thermocycling (category c). Post-thermocycling bond strengths, on the contrary, were divided into four categories (d, e, f, and g), although average values were less than 10.0 MPa.

The results of bond testing of the particle-abraded specimens are summarized in Table 3. The mean bond strength varied from a low of only 0.4 MPa to a high of 34.7 MPa. Mann-Whitney *U* test applied to the bond strength results showed that reduction in bond strength after thermocycling was again statistically significant for all five priming conditions (Table 3). Unprimed control group resulted in the lowest bond strength both pre- (category h) and post-thermocycling (category j). Before thermocycling, bond strengths were comparable (category i) except for the control group. Unlike the ground specimens, no significant difference was found between the Alloy Primer and Estenia Opaque Primer groups (category m), and these groups demonstrated the greatest post thermocycling bond strength.

Observation of debonded surfaces revealed that adhesive failure between the steel surface and the Unifast resin material was predominant for all specimens.

Table 2 Shear bond strength (MPa) between ground SUS XM27 steel and the Unifast Trad acrylic resin with/without priming

	0 thermocycle				2,000 thermocycles				
Primer	Median	Mean	SD	Category	Median	Mean	SD	Category	M-W *U* test
None (Control)	4.6	5.3	1.6	a	0.2	0.2	0.1	d	Significant
Super-Bond Liquid	29.9	30.2	1.5	b	0.3	0.3	0.1	d	Significant
M. L. Primer	33.2	33.0	1.8	b c	0.6	0.7	0.2	e	Significant
Estenia Opaque Primer	33.1	33.6	1.7	c	1.8	1.7	0.3	f	Significant
Alloy Primer	32.7	33.7	2.7	c	8.8	7.9	3.6	g	Significant

SD, Standard deviation. Category, Identical lower case letters indicate that they are not statistically different (Steel-Dwass test, $P > 0.05$).
M-W *U* test, The term 'Significant' indicates that the difference between the pre- and post-thermocycling bond strengths is significant (Mann-Whitney *U* test, $P < 0.05$).

Table 3 Shear bond strength (MPa) between alumina-blasted SUS XM27 steel and the Unifast Trad acrylic resin with/without priming

	0 thermocycle				20,000 thermocycles				
Primer	Median	Mean	SD	Category	Median	Mean	SD	Category	M-W *U* test
None (Control)	19.7	19.6	3.6	h	0.2	0.4	0.3	j	Significant
Super-Bond Liquid	33.9	34.0	1.3	i	4.0	4.3	1.3	k	Significant
M. L. Primer	34.4	34.5	1.7	i	7.8	7.5	1.7	l	Significant
Estenia Opaque Primer	35.4	34.6	2.0	i	15.0	15.1	3.8	m	Significant
Alloy Primer	34.3	34.7	1.9	i	18.0	16.5	4.1	m	Significant

SD, Standard deviation. Category, Identical lower case letters indicate that they are not statistically different (Steel-Dwass test, $P > 0.05$).
M-W *U* test, The term 'Significant' indicates that the difference between the pre- and post-thermocycling bond strengths is significant (Mann-Whitney *U* test, $P < 0.05$).

Discussion

This study evaluated the effects of air-borne particle abrasion as well as priming agents on bonding between XM27 steel and auto-polymerizing acrylic resin. Bonding characteristics of priming agents were evaluated primarily using four single liquid compositions and ground steel specimens. Although the reduction in bond strength after 2,000 thermocycles was remarkable, post-thermocycling bond strength results revealed effectiveness of the Alloy Primer material in which a hydrophobic phosphate monomer (MDP) was added. Effectiveness of the MDP monomer in bonding stainless steel has been previously reported (4,5,7-9), and the current result agrees with these findings, although the composition of resins and steel, and testing assembly were not identical. The authors speculate that the improved bond strength to XM27 steel is derived from the interaction between the dihydrogen phosphate in the MDP monomer and passive chromium oxide layer (15) on the XM27 steel, because the steel contains 26% chromium.

The effect of alumina air-abrasion was next evaluated by extending the thermocycling period. The results again demonstrated the effectiveness of the two priming agents that contain MDP, i.e., the Alloy Primer and Estenia Opaque Primer materials. It is notable that the ranking of the priming agents was almost identical regardless of the application of particle abrasion and extension of thermocycling. The advantages of a particle-abraded surface over a ground surface are; 1) contamination can be mechanically removed by alumina particles, and 2) the bonding surface area can be increased microscopically by roughening the surface by alumina particles.

Reduction in bond strength after thermocycling was obvious in this study as compared with a previous study (8), in which both XM27 steel and the Unifast Trad resin were employed. The difference between the current study and previous study (8) was specimen assembly. The current study used metal-to-bulk-resin bonded specimens, whereas the previous study (8) employed metal-to-metal luted specimens. The influence of thermal stress during thermocycling may be more apparent in metal-to-resin bonded specimens than in metal-to-metal bonded specimens. This is due to the considerable difference in coefficient of thermal expansion between steel and acrylic resin (4).

On the basis of the current experiment results, the magnetic assemblies and keepers should be air-abraded with alumina, followed by priming with one of the MDP primers before bonding with resin material. Also, it is beneficial for assemblies and keepers to be luted to the metal skeleton or root cap with adhesive resin to avoid detachment of the assemblies and keepers as well as fracture of acrylic denture bases within a short period.

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