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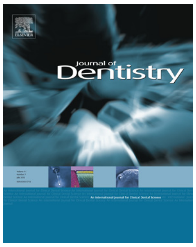

Effect of pretreatments on the metal–ceramic bonding strength of a Pd–Ag alloy

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ABSTRACT

Objectives: The purpose of this study was to evaluate the efficacy of surface treatments on the bonding properties between a metal and ceramic.

Methods: Sixty metal specimens were divided equally into four groups of 15 samples each. These groups received different treatments (Gr1: 250 μm Al_2O_3 + preoxidation; Gr2: 250 μm Al_2O_3 + degassing; Gr3: 120 μm Al_2O_3 + preoxidation; Gr4: 120 μm Al_2O_3 + degassing). Bond strengths were evaluated using a three-point bending test. The results were analyzed using 2-way ANOVA and Tukey's test. Scanning electron microscopy and energy dispersive spectroscopy were used to observe the microscopic features, elemental compositions and distributions, and diffusion in the specimens. Mechanical profiler was used to measure the roughness of metal surface.

Results: The bond strengths of the four groups ranged from 45.00 ± 3.63 MPa to 51.61 ± 5.91 MPa, with significant differences ($P < .05$). The specimen that received the pretreatment of 250 μm Al_2O_3 air-particle abrasion + degassing had the highest bond strength. Heating under different oxygen partial pressures caused the final Pd–Ag alloys to have varying degrees of internal oxidation and different quantities of metallic nodules. None of the elements in either the ceramic or the Pd–Ag alloy layer diffused into the other layer.

Conclusions: The metal–ceramic specimen subjected to air-particle abrasion with 250 μm Al_2O_3 and degassed before porcelain firing had significantly higher bond strength than specimens treated differently.

1. Introduction

The metal–ceramic (MC) bond is critical for the functional and aesthetic success of dental MC prostheses. Traditionally, four factors contribute to the strength of the MC bond: chemical bonding, mechanical bonding, van der Waals forces, and compressive forces. Chemical bonding and mechanical interlocking are believed to play the most prominent roles in the strength of the metal–ceramic bond.[1,2]

In an effort to improve the bond strength between the metal and ceramic, some surface treatments have been studied, such as oxidation of the metal prior to porcelain application, application of bonding agents, and airborne particle abrasion.[3–6] However, the literature has not come to a consensus on a standard pretreatment of the metal surface before applying the ceramic.

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Pa–Ag alloys were introduced in the 1970s and proved suitable for dental purposes.[7] They were specifically developed as economical alternatives to more expensive Au-based alloys.[8] Compared with the Ni–Cr alloy and other non-precious metals, Pa–Ag alloys have superior biocompatibility. Furthermore, these alloys are easy to machine and manufacture because of their satisfactory mechanical properties such as hardness and elastic modulus.[9,10]

The presence of an oxide layer on the surface of the metal alloy has been considered essential for porcelain adherence.[11,12] Oxide layers less than 1–2 nm thick have been found to promote high bond strength.[13] Compared with other casting alloys, Pd–Ag alloys are unique because of their ability at elevated temperatures to dissolve appreciable amounts of oxygen, leading to internal oxidization.[14] According to metallic internal oxidation theory, oxygen dissolves in the Pd–Ag alloy and then reacts with Sn and In contained in the alloy. The calculated molar volume of SnO_2 is 21.55 cm^3, 60% greater than that of Sn in an fcc (face centred cubic) solid solution. Similarly, 2 mol of In are required to form 1 mol of In_2O_3; thus, the increase in volume upon internal oxidation of In within an fcc structure is 48%. These volume increases induce strain in the alloy, which causes metallic nodules to form on the surface of the material.[15] Mechanical interlocking caused by the metallic nodules is believed to be the most important factor for increasing the bond strength of the MC bond in Pd–Ag alloys.[16]

Mackert et al.[15] found that metallic Pd–Ag nodules but no oxide film formed on the surface of the Pd–Ag–Sn–In alloy after a pre-porcelainizing oxidation treatment at 750 °C. They considered that the porcelain remained on the surface of this alloy primarily because of mechanical interlocking with the external nodules. They found that the quantity of Pd–Ag nodules depended on the temperature of the heat treatment. However, the relationship between the quantity of metallic nodules and MC bond strength has not been studied.

Abrading the surface of the metal alloy with Al_2O_3 increases mechanical interlocking and enlarges the surface area of the metal in contact with the deposited porcelain. Although the literature recommends a larger Al_2O_3 particle size when blasting the surface of the alloy to increase the undercuts and cavities generated and to reduce Al_2O_3 contamination,[17] the particle size of Al_2O_3 used for Pd–Ag alloy blasting is generally 120 μm or less.[18,19] Hofstede et al.[20] contended that a rough metal surface can trap air pockets and contaminants, which may lead to gas formation during ceramic firing, producing porosity in the ceramic.

Heat treatment and air-particle abrasion have proven to be important procedures for increasing the bond strength at the MC interface.[21–23] However, further research must be performed to find the appropriate size of particles used for air-particle abrasion of Pd–Ag alloys and the best heat treatment methods after abrasion and ceramic deposition.

This study investigated the bond strength of Pd–Ag alloy specimens subjected to different heat treatments and air-particle abrasion treatments using different particles sizes. After performing these treatments, we observed the metal surfaces and MC bond interfaces with scanning electron microscopy (SEM) and energy-dispersive spectroscopy (EDS). The null hypothesis was that the bond strength of Pd–Ag alloys would be similar among the groups, regardless of pretreatment.

2. Materials and methods

A 3D modeller (Projet-DP 3000, 3D Systems, Rock Hill, SC, USA) was used to prepare 60 precision acrylic templates (VisiJet CP200, 3D Systems) (25 mm × 3 mm × 0.5 mm) of metallic frameworks according to the ISO9693 standard.[24] We mounted each template in a silicon ring and then poured in investment material (Bellavest SH, Bego, Bremen, Germany), following the manufacturer's recommendations. We then cast the metallic frameworks using a Pd–Ag alloy (W-1, Ivoclar Vivadent, Liechtenstein) in an electrical induction furnace (Nautilus, Kerr, Germany). The sprues were removed using carbide discs at low speeds. We then performed air-particle abrasion (Jalixing, Zhuhai, China) with 110 μm Al_2O_3 particles at a pressure of 0.3 MPa to remove the investment material. Afterwards, specimens were ultrasonically cleaned for 30 min in a distilled water bath (Ultrasonic Cleaner, Shenzhen, China). All metal samples were measured with a digital calliper (HengLiang, Shanghai, China).

The 60 metallized samples were divided into four groups of 15 samples according to the heat treatment (preoxidation or degassing) and surface treatment (air-particle abrasion with 250 μm or 120 μm Al_2O_3) used. The four groups were Gr1: 250 μm Al_2O_3 + preoxidation, Gr2: 250 μm Al_2O_3 + degassing, Gr3: 120 μm Al_2O_3 + preoxidation, and Gr4: 120 μm Al_2O_3 + -degassing. The metallized samples were air-abraded at an angle of 45° for 10 s from a distance of ~2 cm under a pressure of 0.3 MPa according to grouping. The surfaces of the metallized samples after air-abrasion were studied using SEM (JCX8100, JEOL, Tokyo, Japan). The metallized samples of Gr1 and Gr3 were then placed into a programmable dental porcelain furnace (Programat P3000; Ivoclar Vivadent, Liechtenstein) pre-heated to 600 °C. The temperature rose to 1010 °C at a rate of 70 °C/min; the air pressure was maintained at standard atmospheric pressure. Gr2 and Gr4 were heat treated according to the same procedures, except at a vacuum level of 0.0036 MPa.

One random metal samples from each group was taken to study its surface using SEM. Then, to examine cross-sections of the metal layers, those samples taken from each group were embedded in clear autopolymerizing acrylic resin (Weiyi, Laizhou, China). We then cut a section from each sample, ground it, and polished it. After these procedures, the specimens were ultrasonically cleaned in acetone for 10 min and then dried. SEM and EDS (Oxford-7412, Oxford Instruments, Abingdon, UK) were used to observe the morphologies, elemental compositions and distributions, and diffusion in the cross-sections. Another random metal samples from each group were measured surface roughness (Ra) by a mechanical profiler (E-35B, ACCRETECH, Tokyo, Japan) in microns. Each sample was measured 10 times and the average was obtained. The filtration was done using Gaussian filter with cut-off wavelength 0.25 mm and measuring length 4 mm.

We then used a furnace (Programat P3000; Ivoclar Vivadent, Liechtenstein) to apply a 8 mm × 3 mm × 1.1 mm ceramic