



2004

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**COMPARATIVE TENSILE STRENGTHS OF PRECERAMIC AND
POSTCERAMIC SOLDER CONNECTORS USING HIGH-PALLADIUM ALLOY**

A Thesis submitted in partial fulfillment of the requirements for the degree of Master of Science at Virginia Commonwealth University.

by

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July 2004

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Abstract

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Purpose: To evaluate the tensile strength properties of Rx Naturelle Plus and Option (high-palladium) alloys on soldered connectors under simulated preceramic and postceramic soldering conditions.

Materials and methods: Eighty cylindrical castings were fabricated (40 using Rx Naturelle Plus alloy and 40 using Option alloy). The 40 castings for each alloy were subdivided into 2 groups of 20 each. In the first group, castings for each alloy were randomly paired and soldered with SMG2 solder to produce 10 preceramic test connector specimens for each alloy. In the second group, castings were similarly paired and soldered with 490 fine solder to produce 10 postceramic test connector specimens for each alloy. Each solder connector was subjected to tensile force until failure using an Instron testing machine. Statistical analysis using a Wilcoxon Rank Sums Test was performed.

Results: No significant difference was found between the mean connector strength for Rx Naturelle Plus preceramic solder (mean tensile failure stress, 50,000 Psi; STD, 11,250) compared to Option preceramic solder (mean tensile failure stress, 59,700.4 Psi; STD, 16,350) ($p= 0.1202$). However, the connector strength for Rx Naturelle Plus postceramic solder (mean tensile failure stress, 37,800 Psi; STD, 32,450) was significantly lower than the Option postceramic solder (mean tensile failure stress, 45,300 Psi; STD, 17,550) ($p= 0.0407$). Not only did Rx Naturelle Plus postceramic solder connectors show evidence of lower strength, but also an increased variation among specimens.

Conclusions: Rx Naturelle Plus solder connectors provided better strength with the preceramic are opposed with the postceramic connectors. For postsolder connectors, Rx Naturelle Plus alloy had less acceptable strength and a wider variation, suggesting it is more technique sensitive than Option alloy connectors.

INTRODUCTION

Soldering is the most common and practical procedure used to unite fixed partial denture (FPD) components using an intermediary metal (solder) that has a lower melting point than the parent alloy.¹ High-palladium alloys have become popular for the fabrication of metal-ceramic restorations due to the excellent mechanical properties, cost, and bonding with dental porcelain.² Soldering is the preferred method of joining FPD units. Gegauff et al.³ found that best margin adaptation was produced by soldering when compared to one-piece castings.

Controversy exists in the literature concerning the solder connector strength of fixed partial denture components. Problems related to soldering procedures vary according to the different methods and materials used. There are significant factors associated with successful soldering that include alloy composition, solder material, surface contamination, gap distance, indexing method and material, wetting angle, the length and shape of solder connectors, surface to be soldered, soldering temperature, and the thickness of the retainers.⁴⁻⁸

Gap distance is one of the variables reported by several authors to influence the accuracy of soldering. It has been reported that the ideal gap distance ranges from 0.1 mm to 0.75 mm.⁹⁻¹³ Rasmussen et al.¹⁴ found that increasing the gap width will increase the strength of the solder connectors. Another study found that bond strength of a solder connector may be stronger using narrower gaps.¹¹ One study stated that gap distance doesn't affect solder connector strength.⁴ According to Stade et al.,¹⁵ the method of soldering was more significant to connector strength than gap width.

Hollenback et al.⁸ found that there is no appreciable effect of the shape of a solder surface on soldering distortion. Skinner et al.⁴ stated that a well-formed solder connector is one in which the solder wets the cast metal surface and creates adhesion by primary metallic bonding, with the interface between the metal and solder clearly visible. There should be no sharp angles to localize stress and initiate crack propagation.

With the stress concentrations found in FPD connectors, their potential for failure is high, and these stresses are amplified by the presence of porosity.¹⁵ Staffanou et al.¹⁶ attributed solder joint failures to overheating of the solder, which can create atomic diffusion with the connectors and promote grain growth, and therefore weaken connectors. Lautenschlager et al.¹⁷ indicated that the amount of porosity in a solder connector was responsible for a decrease in connector strength. Voids can result from flux entrapment due to overfluxing or underheating. Janus et al.⁶ found that soldered connectors of low-gold alloy solders have more voids than those with high-gold alloy solders. Gap space has also been reported to have an effect on connector porosity.^{11,15} Usually, visual examination is used to evaluate a connector's size and voids. However, only after the central area of a connector is separated, can problems (internal voids, flux inclusion and oxidation) generally be detected.

Presoldered connectors have been found to be slightly stronger than postsoldered, but more failures occur with presolder connectors because of the closer melting temperature of the solder relative to the parent metal.¹⁸ It was reported, however, that the higher fusing solders have lower surface tensions and flow more easily.¹⁴ Other studies have found the opposite, that postsolder connectors are stronger than presolder^{14,16}, and one author reported no significant difference.¹⁹

Cattaneo et al.²⁰ found that there was no significant difference in tensile strength and connector porosity between conventional torch and infrared heating techniques. Bench cooling from a high soldering temperature to room temperature can promote grain growth that can weaken the soldered connector.²⁰

To have an acceptable solder connector, the metal alloy surfaces must be free of oxides and other impurities. According to Rassmussen et al.¹⁴, metals when properly fluxed and heated, will yield a proper wetting and bonding with the soldering alloys. Fluxes dissolve surface impurities and protect the surface from oxidation during the heating process. Too much flux can cause an inclusion of flux in the connector, leading to a decrease in the strength, while an insufficient amount of flux could lead to unsatisfactory metal protection.¹⁴ Meanwhile, the presence of voids may not be due to flux alone, but enough metal fluidity to flow must be considered too.

The purpose of this in vitro study is to evaluate the tensile strength properties of Rx Naturelle Plus (Pentron, Wallingford, CT) and Option (Dentsply-Ceramco, Bloomfield, CT) high-palladium alloys at soldered connectors using simulated preceramic and postceramic soldering conditions. The hypothesis of this project is that Rx Naturelle Plus alloy solder connectors have comparable tensile strength to Option high-palladium alloy solder connectors due to their similar compositions (Table I).

MATERIALS AND METHODS

Eighty cylindrical test castings, 14.1 mm long, 3 mm in diameter with a 6 mm sphere on one end were made using Rx Naturelle Plus and Option alloys. Forty castings were made using Rx Naturelle Plus alloy, and the other forty were made using Option alloy (Figure 1)(Table I).

All castings were fabricated using a centrifugal casting machine (Production Caster, J. F. Jelenko & Co., Armonk, NY.) and a gas-oxygen torch (Harris Calorific No. 16S, Dublin, Ireland). To avoid contamination, a separate ceramic crucible was used to melt each alloy. The castings were quenched after 5 minutes of bench cooling and before divesting. Small nodules were removed. The castings were then divested with a 50 micron non-recirculating aluminous oxide abrasive (Omega Micro Vac Work Station, Sterdent Corp., Stamford, CT). Castings were ultrasonically cleaned in general purpose cleaner (Jelenko All Purpose Cleaner, J. F. Jelenko & Co., Armonk, NY.) and distilled water. The castings were then considered ready for indexing and soldering. No polishing procedures were performed.

For the presolder specimens, pairs of prepared castings were arranged in a custom soldering index jig modified after Rasmussen et al.¹⁴ which is an aluminum jig designed to hold the casting units in a precise standardized position using a uniform gap distance (0.5 mm) (Figure 2). Connectors were prefluxed with fluoride flux (Dentsply-Ceramco, Bloomfield, CT) and joined with autopolymerizing acrylic resin (Duralay, Reliance Dental Mfg. Co. Worth, Il.). The soldering gap was produced by means of a feeler gauge (Figure 3). Each pair of castings was invested for soldering with gypsum-bonded soldering investment (Hi-Heat, Whip-Mix Corp., Louisville, Ky.) and was allowed to set

for at least 1 hour before soldering. The soldering blocks were trimmed to a uniform 2x4 inches size and square shape, placed in an oven, and heated to 1200°F to eliminate the acrylic resin. The assemblies were removed from the oven and immediately torch soldered with the same gas-oxygen torch with a single orifice soldering tip and fluoride flux (Dentsply-Ceramco). Both alloys were preceramic soldered with SMG2 solder (Dentsply-Ceramco, Bloomfield, CT.). The investments were quenched in water after 5 minutes of bench cooling and before divesting. Each assembly was subjected to the following heat and cooling cycles to simulate porcelain application: one degassing, one opaque porcelain, two body porcelain, and one glazing, all in a single porcelain furnace (Ney 660 Modular, J. M. Ney Co. Bloomfield, CT.)

The postsolder specimens were cleaned ultrasonically and heat-treated to simulate porcelain addition. Each casting was subjected to the following heat and cooling cycles: one degassing, one opaque porcelain, two body porcelain, and one glazing, all in a single porcelain furnace (Ney 660 Modular, J. M. Ney Co.). Pairs of prepared castings were arranged in the custom soldering index jig as before (Figure 2). Connectors were prefluxed with Borax flux (Dentsply-Ceramco) and joined with autopolymerizing acrylic resin (Duralay, Reliance Dental Mfg. Co.) adding powder and liquid sequentially. A precise 0.5 mm soldering gap was produced by means of a feeler gauge (Figure 3). Each pair of castings were invested for soldering with gypsum-bonded soldering investment (Hi-Heat, Whip-Mix Corp.) and were allowed to set for at least 1 hour before soldering. The soldering blocks were trimmed to a uniform 2x4 inches size and square shape; placed in an oven, and heated to 1200°F to eliminate the acrylic resin. The assemblies were removed from the oven and immediately torch soldered with the same gas oxygen torch,

with a single orifice soldering tip and Borax flux (Dentsply-Ceramco). Alloys were soldered with 490 Fine solder (Dentsply-Ceramco). The investments were quenched after 5 minutes of bench cooling before divesting.

After completion of all soldering procedures, all tensile test specimens were cleaned of debris and machined to a uniform diameter (original casting measurements) using a precision jig on a machinist's lathe (Unimat, American Edelstaal Inc., Tenefly, NJ.). Tensile testing was performed with the Instron universal testing machine (Instron Corp., Canton, Mass.). A custom-made holding device was used to firmly hold the test specimens to insure uniaxial tensile loading along the long-axis of the specimens at a crosshead speed of 0.02 inches/minute (Figure 5). Each specimen was pulled until solder connector failure occurred.

RESULTS

Using statistical analysis software (JMP Version 5.1, SAS Institute Inc, Cary, NC.), Figure 6 and 7 show the distribution of data for all four groups of solder connectors, including means and standard deviations. Table II shows relatively low mean tensile strength data and relatively high standard deviation for the postsolder connectors of the Rx Naturelle Plus alloy (mean tensile failure stress, 37,800 Psi; STD, 32,450). Log transformation of the skewed data for each alloy did not normalize it sufficiently to apply a Student's t-Test or ANOVA. Therefore, the Wilcoxon Rank Sums test was used to determine whether there were significant differences between the test groups.

Wilcoxon Rank Sums Test found no significant difference between the mean connector strength for Rx Naturelle Plus preceramic solder (mean tensile failure stress,

50,000 Psi; STD, 11,250) compared to Option preceramic solder (mean tensile failure stress, 59,700.4 Psi; STD, 16,350) ($p= 0.1202$). However, the connector strength for Rx Naturelle Plus postceramic solder (mean tensile failure stress, 37,800 Psi; STD, 32,450) was significantly lower than the Option postceramic solder (mean tensile failure stress, 45,300 Psi; STD, 17,550) ($p= 0.0407$).

DISCUSSION

The development of strength in a solder connector involves the melting of solder, solder flow, and wetting between the parent metal and the solder. The precision of solder flow without overheating or melting the parent alloy requires accurate control of the soldering temperature. Technique-related problems could be a contributor to the low reproducibility of high-strength solder connectors.

In this study, many (80%) of the specimens soldered adequately produced connectors that were acceptable. However, their mean strengths were somewhat lower than the strengths reported in previous studies using high-palladium alloy with a different preceramic solder.^{5,6} Only 2 solder joints fractured at the interface between the solder and parent alloy, so it is assumed that the wetting of the remaining cast solder alloys by the solders was sufficient. It was observed that a few of the weak specimens had “ringed connectors” which had the solder flow around the circumference of the connector leaving the center void of solder. The lower tensile strength for the low fusing postsolder may be due to their higher surface tension as discussed by Rassmussen et al.¹⁴, which may potentially result in a weaker solder connection. A limitation of this study was that more control of soldering connectors could have been achieved by using a closed vacuum

furnace or protective atmosphere instead of the connectors formed using gas/oxygen or gas/air torch techniques.

Since the purpose of this study was to determine the tensile strength of preceramic and postceramic solder connectors of high-palladium alloy, it was found that acceptable connectors resulted for the preceramic solder connectors only. Further research is needed to evaluate failure under fatigue stresses to simulate the expected mode of failure in the oral environment.

CLINICAL IMPLICATIONS

Alloys are widely used in the dentistry and comparable alloy materials are available from different manufacturers. It is of interest to determine the comparative tensile strengths of solder connectors of available dental alloys. Postsolder comparisons were found to be especially variable between the two high-palladium alloy materials examined.

CONCLUSION

An experiment was conducted to evaluate the connector strength of Rx Naturelle Plus and Option (high-palladium) alloys under preceramic and postceramic simulations. Statistical analysis indicated that Rx Naturelle Plus postceramic solder connectors had significantly lower strengths. Preceramic solder joints for both Rx Naturelle Plus and Option alloys were higher than postsolder joints.

FIGURES

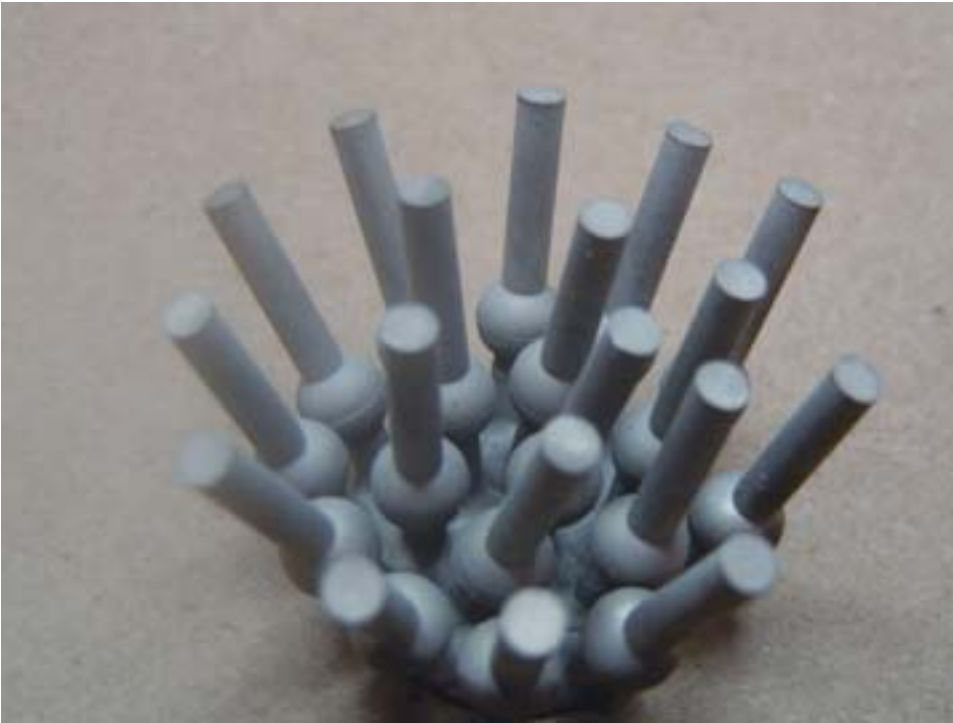


Figure 1. Castings after divesting and cleaning before cutting off button.



Figure 2. Custom aluminum jig for aligning castings to index for soldering. The jig consists of 2 structures that fit precisely together to hold 10 castings (5 on each side) with a gap distance produced by a leaf gauge.

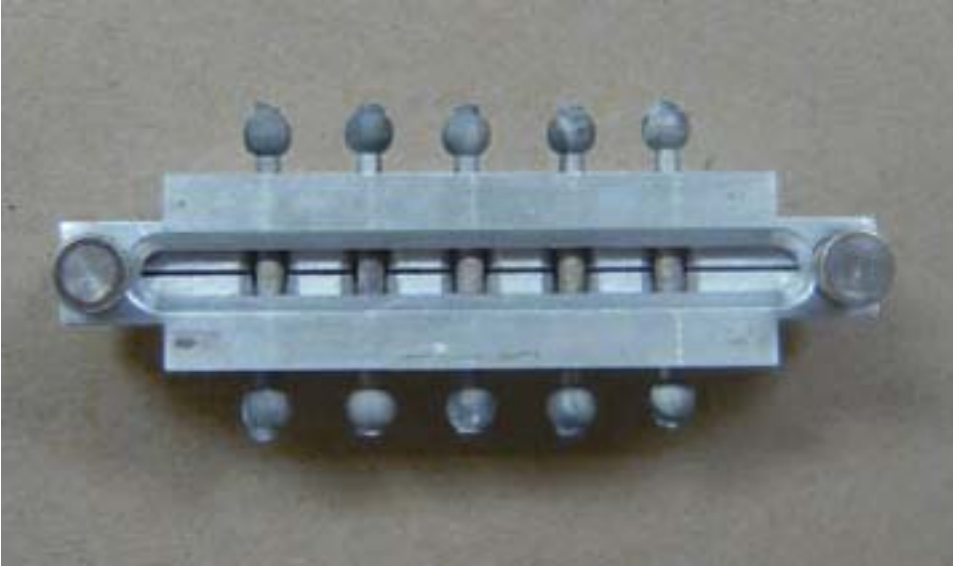


Figure 3. Castings arranged in jig with leaf gauge in place to produce standard gap. The leaf gauge placed between the castings to create a gap distance for soldering.



Figure 4. Typical tensile test specimen before machining.



Figure 5. Tensile test specimen in holding apparatus on Instron testing machine.

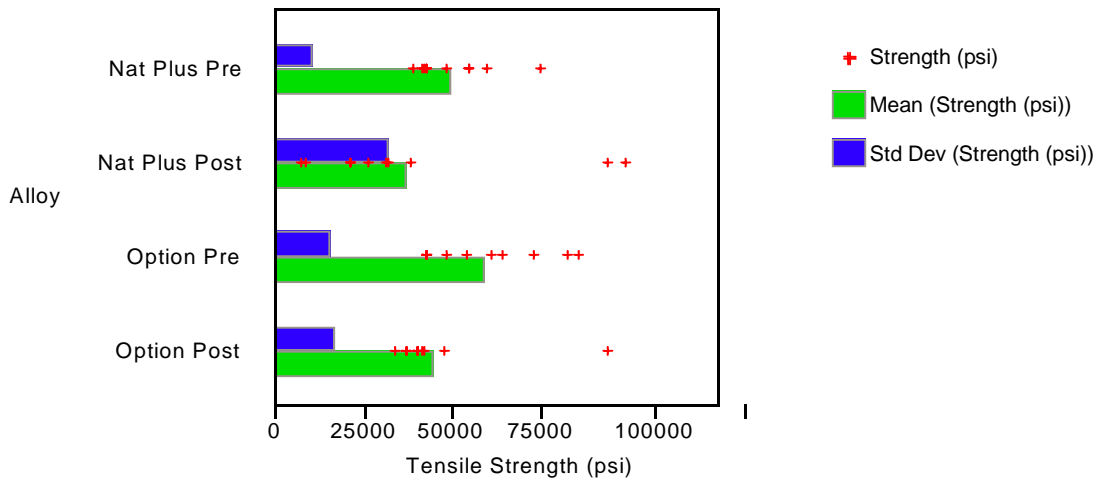


Figure 6. Strength of high-palladium alloy, pre and postsolder connectors.

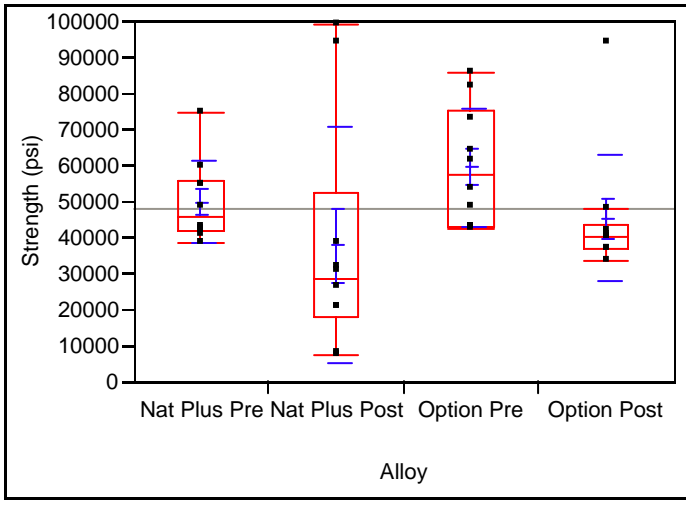


Figure 7. Analysis of Strength for Rx Naturelle Plus and Option alloy solder connectors.

TABLES

Table I. Composition by percentage of Rx Naturelle Plus alloy, Option alloy, SMG2 for presolder, and 490 Fine for postsolder

ALLOY OR SOLDER	ELEMENTS												
	<i>Melting Range</i> (°F)	<i>Au</i>	<i>Pt</i>	<i>Pd</i>	<i>Ag</i>	<i>Cu</i>	<i>Zn</i>	<i>Fe</i>	<i>Ir</i>	<i>In</i>	<i>Ga</i>	<i>B</i>	<i>Sn</i>
NATURLLE PLUS	2,100-2,250	2		77		10				4	7		
OPTION	2,174-2,354	2		78.8		10			1		9	.02	
SMG2	2,030	85.5	4	3	5		.5	.5		1			.5
490 FINE	1,409	49			24.6	19.5	5.7						1.2

Table II. Mean and standard deviation for tensile strength data (Psi).

Solder	Mean	SD
Rx Naturelle Plus Presolder	50,000	11,250
Rx Naturelle Plus Postsolder	37,800	32,550
Option Presolder	59,700	16,450
Option Postsolder	45,300	17,550

LITERATURE REVIEW

Soldering is the most common and practical procedure used to unite fixed partial denture (FPD) components using an intermediary metal (solder) that has a lower melting point than the parent alloy.¹ High-palladium alloys have become popular for the fabrication of metal-ceramic restorations due to the excellent mechanical properties, cost, and bonding with dental porcelain.² Soldering is the preferred method of joining FPD units. Gegauff et al.³ found that best margin adaptation was produced by soldering when compared to one-piece castings.

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