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Hyeongil Kim

State University of New York at Buffalo

Soni Prasad

Marquette University, soni.prasad@marquette.edu

Robert Dunford

State University of New York at Buffalo

Edward A. Monaco, Jr.

State University of New York at Buffalo

Marquette University

e-Publications@Marquette

Dentistry Faculty Research and Publications/School of Dentistry

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Strength Properties of Preceramic Brazed Joints of a Gold-Palladium Alloy with a Microwave-Assisted Oven and Gas/Oxygen Torch Technique

[Hyeongil Kim](#)

Associate Professor, Restorative Dentistry, University at Buffalo, Buffalo, NY

[Soni Prasad](#)

Assistant Professor, General Dental Sciences, Marquette University, Milwaukee, WI

[Robert Dunford](#)

Statistician, Department of Oral Sciences, University at Buffalo, Buffalo, NY

[Edward A. Monaco Jr.](#)

Assistant Professor, Restorative Dentistry, University at Buffalo, Buffalo, NY

Statement of problem

The effect of [microwave](#) brazing on the strength properties of dental casting alloys is not yet known.

Purpose

The purpose of this study was to compare the strength properties of preceramic brazed joints obtained by using a microwave oven and a conventional torch flame for a high noble alloy (Au-Pd).

Material and methods

A total of 18 tensile bars made of an Au-Pd ceramic alloy were fabricated. Six specimens were cut and joined with a high-fusing preceramic solder in a specially designed microwave oven, and 6 specimens were joined with a conventional natural gas/oxygen torch. The remaining 6 uncut specimens were tested as a control. All the specimens were subjected to testing with a universal testing machine. A 1-way ANOVA was performed for each strength property tested.

Results

The tensile strength of the uncut group was the highest (745 ± 19 MPa), followed by the microwave group (420 ± 68 MPa) and the conventional torch group (348 ± 103 MPa) ($P < .001$); however, no significant difference in tensile strength was found between the microwave group and gas torch group. The tensile strength of the microwave group exceeded ANSI/ADA Standard No. 88, Dental Brazing Alloys (a joint standard of the American National Standards Institute and the American Dental Association).

Conclusions

The [microwave heating](#) preceramic solder method demonstrated the excellent tensile strength of an Au-Pd alloy and may be an alternative way of joining alloys when a torch flame is [contraindicated](#).

Clinical Implications

The effect of [microwave](#) brazing on the strength properties of dental casting alloys is not yet known. This research suggests an alternative method of preceramic brazing that improves the physical properties of the Au-Pd alloy.

As defined by the Glossary of [Prosthodontic](#) Terms, the connector is a component of a partial fixed dental [prosthesis](#) (FDP) "that unites the retainer(s) and pontic(s)."¹ The rigid connector is commonly used in the partial FDP and can be made with a cast, brazed, or fused joint.

Numerous investigations on dental brazing have been done on the accuracy and strength of connectors made of various materials and with various methods. Gap distance plays an important role in achieving accurate and strong joints. Ryge² stated that a gap distance greater than 0.13 mm should be provided to prevent distortion during the heating and brazing of Type II gold alloys. He found that the more porous joints occurred with small gap distances when a 650 solder was used. Stade et al³ agreed that gap distance affected joint porosity, concluding that gaps greater than 0.30 mm resulted in increased strength under preceramic and postceramic conditions. However, Willis and Nicholls⁴ found that a narrow gap distance (0.15 mm) resulted in greater overall distortion than wider gap distances (0.3 and 0.45 mm). They concluded that a minimal gap distance without contact was desirable to minimize distortion when Type III gold alloys were joined with a gas-air torch and a 650 solder. Many studies have been done on the effect of different brazing methods before and after [porcelain](#) application. Stade³ reported that postceramic brazed joints were stronger than preceramic joints because of the higher copper content of postceramic solder. However, Staffanou et al⁵ reported no major differences in strength between preceramic and postceramic brazed connectors in various ceramic-metal combinations. Ziebert et al⁶ investigated the accuracy of 1-piece casting, preceramic brazing, and postceramic brazing. They concluded that preceramic and postceramic brazing adversely affected the marginal fit of the partial FDPs as the [edentulous](#) span of the prosthesis was increased. The accuracy of both preceramic and postceramic brazing did not differ. The 1-piece casting of the 3-unit FDPs was comparable with preceramic and postceramic brazing. The 4-unit and 5-unit partial FDPs joined by preceramic and postceramic brazing had better marginal fit than the 1-piece casting.⁶

Defects play an important role in the strength of the brazed joint. With the presence of porosity, [stress concentration](#) on the brazed joints could be amplified and result in failures.⁷ As Ryge² stated, less porosity resulted when the gap distance was wide and when the units to be joined were brought to brazing temperature before solder application. Lautenschlager et al⁷ also concluded that internal porosity weakened joint strength. Their conclusion was that internal porosity occurred with nonuniform heating and when a gap distance was excessively small.

In addition to gas/oxygen/air torch and oven brazing techniques, alternative approaches to heating metals have been investigated to minimize the oxidation of braze materials in protective environments. Several methods, including an infrared heating method and laser welding, have been investigated to join different metals such as high-palladium, cobalt-chromium, and titanium alloys.^{8, 9, 10, 11, 12, 13} Although these methods fulfill the need for mechanical strength, torch and oven brazing are still standard for joining [dental alloys](#).

[Microwaves](#) form a small portion of the electromagnetic spectrum, with wavelengths ranging from 1 m to 1 mm and frequencies between 0.3 and 300 GHz. These frequencies are above those of radio waves and just below those of visible light on the electromagnetic spectrum. Raytheon developed the first commercial microwave ovens for culinary use a half century ago. Microwave processing of materials has until recently been confined to [oxide ceramics](#), carbide semimetals, and polymeric materials.¹⁴ Roy et al¹⁵ were the first to report the sintering of pure metal powders to full density metal with a microwave oven. Microwave processing of materials, including heating and sintering, is fundamentally different from conventional processing. For many applications, conventional processing is slow, and considerable time is needed to

achieve thermal equilibrium. In [microwave heating](#), the [absorption](#) of microwave energy is followed by uniform heating involving a conversion of electromagnetic energy into thermal energy. In this process, the heat is generated internally within the material instead of originating from the external sources. Therefore, microwave processing provides rapid heating with uniform heat generation, as described in the work of Clark and Sutton.¹⁶

Although [microwave](#) technology is well known in industrial settings for its faster heating rate, shortened processing time, improved microstructure, and energy efficiency, the application of [microwave technology](#) in dentistry has been limited to polymeric materials and dental ceramics.^{17, 18} The microwave energy polymerization of denture base materials produced similar dimensional accuracy to conventional hot water bath polymerization.¹⁹ The polymerization of denture base materials with a microwave oven improved mechanical properties and decreased residual monomer.²⁰ Loh et al²¹ reported that denture resin bases were polymerized effectively by microwave energy, without adverse effects on resin hardness or porosity. Baysan et al²² concluded that the microwave polymerization of a soft denture lining material did not compromise bonding to acrylic resin. Polyzois et al²³ also concluded that the microwave disinfection method was a useful alternative to glutaraldehyde immersion and did not compromise flexural strength. Microwave technology has been used to sinter and glaze [dental ceramic](#) materials.^{24, 25} However, the application of microwave technology to dental brazing alloys has not yet been reported.

The purpose of this study was to evaluate [microwave](#) oven processing and the conventional preceramic torch method and to compare the strength properties of the gold-palladium ceramic alloy resulting from each treatment. The null hypothesis was that the strength properties would be the same for metal connectors joined in a microwave oven, a conventional gas torch, or as cast.

Material and methods

A brass split mold was used to fabricate 18 dumbbell-shaped wax patterns with a circular cross-section in accordance with ANSI/ADA Standard No. 88, Dental Brazing Alloys (a joint standard of the American National Standards Institute and the American Dental Association).²⁶ The pattern dimensions are shown in [Figure 1](#). The alloy used in this study was a high noble ceramic alloy ([Table I](#)) (Lodestar [Au, 51.5%; Pd, 38.5%; In, 8.5%; Ga, 1.5%; Ru, <1.0%; Re, <1.0%]; Ivoclar Vivadent USA). To prepare the cast rods, wax patterns were invested with a phosphate bonded investment (Sure-Vest High Heat Investment; Ivoclar Vivadent). All the specimens were cast with a centrifugal casting machine (Production Caster; J. F. Jelenko & Co) and a gas-oxygen torch (No. 16S; Harris Calorific). The cast specimens were finished and treated with airborne-particle abrasion with 50- μ m alumina particles.

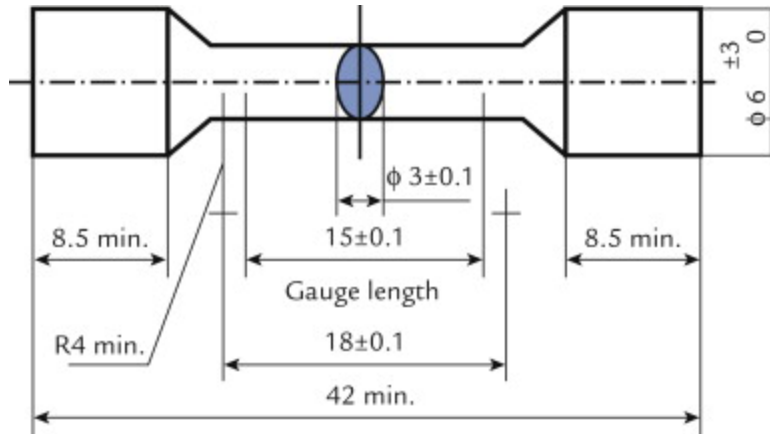


Fig. 1. Test specimen with radius shoulders (dimensions in millimeters).

Table I. Materials tested

Materials	Name	Manufacturer	Composition	Melting Range
Ceramic alloy	Lodestar	Ivoclar	Au, 51.5%; Pd, 38.5%; Ga, 1.5%;	1215°C-
		Vivadent	In, 8.5%; Re, <1.0; Ru, <1.0	1290°C
Braze alloy	High Fusing White Ceramic (HFWC)	Ivoclar	Au, 45%; Pd, 12.4%; Ag, 41.5;	1100°C-
		Vivadent	In, 1.0%; Li, <1.0; Ru, <1.0	1165°C

Data from Ivoclar Vivadent.

After the 12 castings were made, they were sectioned in half perpendicular to the long axis of the casting. A customized positioning jig (made of aluminum) was used to facilitate the alignment of the sectioned specimens for brazing (Fig. 2). The gap between each half was standardized with a 0.3-mm leaf gauge. The test specimens were then indexed with an acrylic resin (Pattern resin LS; GC America Inc) and divided into 2 groups. Six specimens were brazed in a microwave oven with a preceramic solder (High Fusing White Ceramic [HFWC] solder [Au, 45%; Pd, 12.4%; Ag, 41.5%; In, 1.0%; Li, <1.0%; Ru, <1.0%]; Ivoclar Vivadent), and 6 specimens were brazed with a conventional torch. Torch-brazed specimens were subsequently quenched in room temperature water 5 minutes after brazing. The remaining 6 unsectioned castings of the same dumbbell design were randomly selected to serve as an 'as cast' control group.

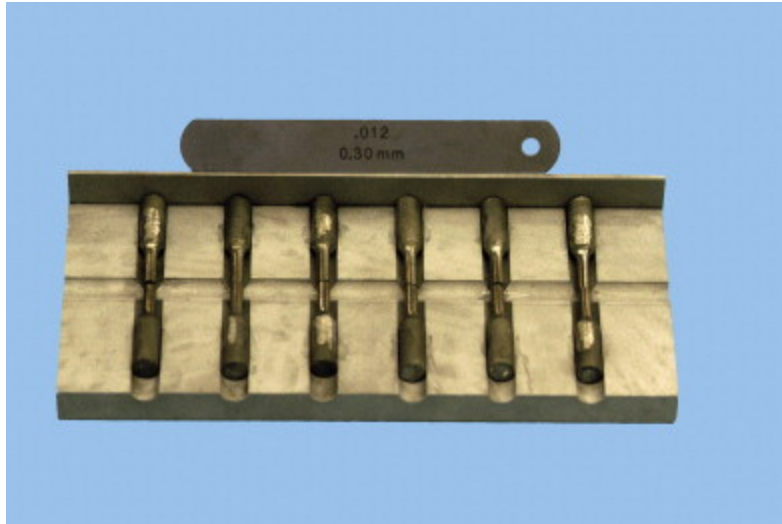


Fig. 2. Assembling jig and leaf gauge.

Indexed units were invested in brazing investment (Soldervest Quick; GC America Inc) according to the manufacturer's directions and placed in a cold wax-elimination oven at a 15°C/min temperature rise to 538°C. After the GC pattern resin had vaporized, the specimens were allowed to bench cool. All joints to be brazed were fluxed (High Fusing Bondal Flux; Ivoclar Vivadent), and a premeasured solder strip was placed onto the joint space. The solder strip was extended 1 to 2 mm beyond the joint surface to ensure enough solder to fill the gap.

Microwave oven brazing

A microwave oven (ThermWave 1.3; EPL Ceramic Materials LLC) was used to braze the gold-palladium alloy ([Fig. 3](#)). This custom-built microwave oven incorporated a water jacket to cool the system and could easily generate and sustain heat up to approximately 1500°C. A pyrometer/thermocouple was inserted from the top of the oven to read the temperature inside the oven accurately. A control box was attached to the microwave oven to control the temperature inside the heating chamber precisely. The invested and fluxed units with solder in position were placed in an insulation box made from alumina insulation board (Eco25B; Zircar Ceramics Inc). Three silicon carbide susceptors (Susceptor; EPL Ceramic Materials LLC) were placed at the far ends of the insulation box. A cover made of the same insulation board was placed on the box. The box assembly was placed on small square insulation blocks on the base of the microwave turntable. This allowed air ventilation and reduced the amount of heat absorbed by the microwave oven.



Fig. 3. ThermWave [Microwave Heating](#) System.

It took approximately 15 minutes to reach the set temperature (1200°C). When the pyrometer indicated that the temperature had reached 1200°C, the unit was shut down manually. The insulation box was removed by using insulated gloves and subsequently bench cooled to room temperature. The specimens were removed and devested with airborne-particle abrasion with 50- μ m alumina particles. After each joint was brazed, excess braze material was removed on a machinist's lathe, and the diameter of the brazed joint was recorded.

All the brazed specimens were heat treated following the manufacturer's instructions for the metal ceramic [porcelain](#) (IPS d.SIGN; Ivoclar Vivadent) to simulate normal porcelain build-up procedures, including oxidation, opaque, [dentin](#), enamel, and glaze porcelain applications. A tensile strength test ([Table II](#)) was performed on a universal testing machine (Instron Model 4202; Instron) at a crosshead speed of 2.0 mm/min until the specimen fractured. The fractured joint surface was subsequently evaluated for fracture mode analysis with a field emission scanning [electron microscope](#) (SEM) (Hitachi S4000, Hitachi High Technologies America Inc). SEM images of the fractured surfaces were made (Figs. [4](#), [5](#)).

Table II. Simulated firing parameters for metal ceramic restoration

	High Temp	Low Temp	Rate	Holding Time	Vacuum
Oxidation	1010°C	403°C	80°C/min	5 min	Yes
Wash opaque	900°C	403°C	80°C/min	1 min	Yes
Second opaque	890°C	403°C	80°C/min	1 min	Yes
First dentin/incisal	870°C	403°C	60°C/min	1 min	Yes
Second dentin/incisal	870°C	403°C	60°C/min	1 min	Yes
Glaze	870°C	403°C	60°C/min	1 min	Yes

Data from Ivoclar Vivadent.

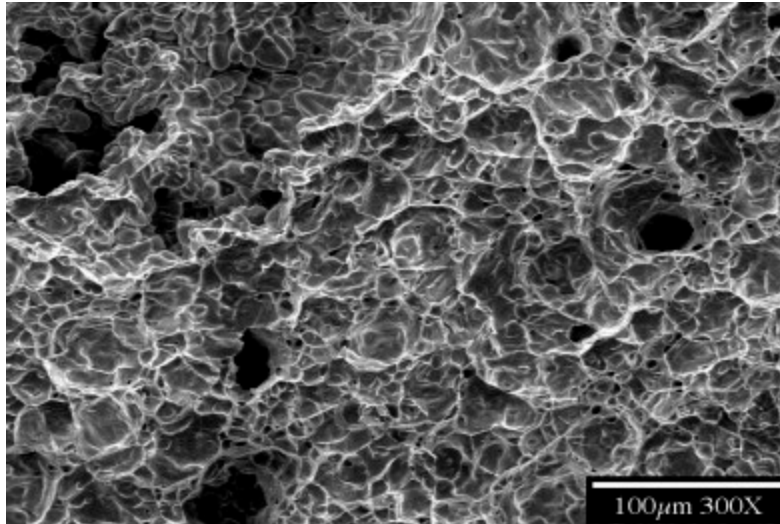


Fig. 4. Tensile specimen of torch group.

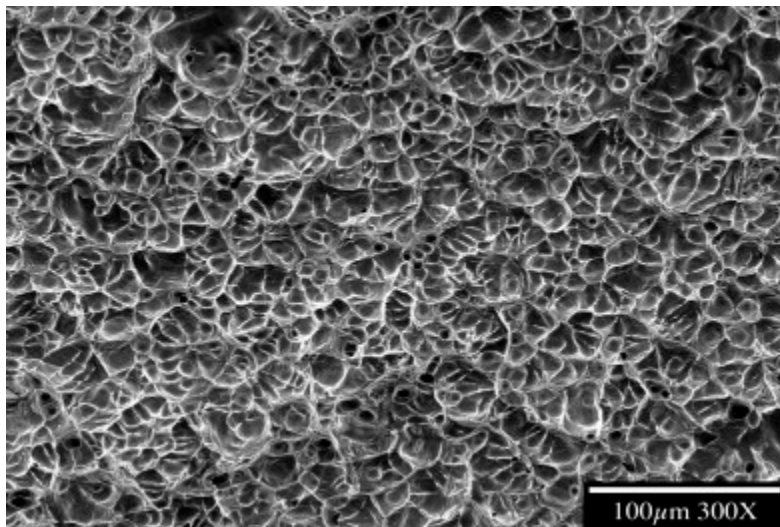


Fig. 5. Tensile specimen of [microwave](#) group.

The strength property data on tensile strength, modulus of elasticity, yield strength, and elongation were analyzed statistically with ANOVA. The post hoc procedure for homogeneous subsets with multiple Tukey comparisons was used when a statistically significant result was obtained from ANOVA ($\alpha=.05$). An analysis was performed to evaluate the power of the tests for the selected sample sizes to estimate the size needed for future experiments to detect statistical difference.

Results

[Table III](#) compares the tensile strength, modulus of elasticity, yield strength, and elongation results of the [microwave](#), torch, and as-cast groups. The tensile strength of the control group was the highest (745 ± 19 MPa), followed by the microwave group (420 ± 68 MPa) and the

conventional torch group (348 ± 103 MPa). The microwave group and the torch group were not statistically different with regard to the other strength properties, except for the modulus of elasticity. SEM images demonstrated that the specimen brazed with the torch flame (Fig. 4) and microwave oven (Fig. 5) displayed patterns of intergranular ductile fracture at the brazed joint.

Table III. Ultimate tensile strength, modulus, yield strength, and elongation of prebrazed and nonbrazed specimens

Variable	N	Mean	SD	Min	Max
UTS (MPa)					
Microwave*	6	420	68	332	499
Torch*	6	348	103	250	542
As cast†	6	745	19	715	764
Modulus (MPa)					
Microwave*	6	127 995	15 746	102 060	147 270
Torch*	6	152 631	41 424	96 708	199 041
As cast*	6	140 623	13 621	129 052	164 366
0.2% offset (MPa)					
Microwave*	6	398	52	325	448
Torch*	6	331	85	241	483
As cast†	6	507	12	489	519
Elongation (%)					
Microwave*	6	6	1	4	8
Torch*	6	5	2	3	10
As cast†	6	31	3	26	33

UTS, ultimate tensile strength; SD, standard deviation.

*No significant difference.

†Significant difference.

As seen in Table III, Table IV, statistically significant differences existed among the groups ($P < .001$), except for the modulus of elasticity. The 'as cast' group results were significantly different from the other 2 test groups (torch and microwave). However, the torch and microwave groups were not significantly different from each other.

Table IV. ANOVA

Variable	SS	df	MS	F	P
UTS					
Between	536 836.00	2	268 418.00	51.20	<.001

Variable	SS	df	MS	F	P
Within	78 632.00	15	5242.13		
Total	615 468.00	17			
Modulus					
Between	1.82	2	9.11	1.27	.309
Within	1.08	15	7.17		
Total	1.26	17			
2% offset					
Between	94 748.44	2	47 374.22	14.02	<.001
Within	50 682.50	15	3378.83		
Total	145 430.94	17			
Elongation					
Between	2501.33	2	1250.67	225.57	<.001
Within	83.17	15	5.54		
Total	2584.50	17			

SS, sum of squares; MS, mean of squares; UTS, ultimate tensile strength.

Discussion

The null hypothesis (that the distribution of strength properties would be the same across all categories of the test group) was rejected.

[Microwave](#) energy was developed primarily for communications and some areas of processing, for example, culinary preparation and the polymerization of rubber materials. Based on their interaction with the microwave field, materials can be divided into 3 categories: opaque (conductor), absorbers (high dielectric loss materials), and transparent (low dielectric loss materials).²⁷ Bulk metals are excellent examples of opaque reflectors of microwave energy. They are not generally heated by microwaves, because they reflect them. Pure water, which is a high dielectric loss material, absorbs well over a wide range of microwave frequencies. Through the [absorption](#) of microwaves, heat energy is generated during the microwave-water interaction.¹⁶ Many ceramics and polymers do not absorb microwaves at room temperature. However, their absorption can be enhanced by increasing the temperature, by adding microwave-absorbing materials, or by changing their form (such as bulk to powder). Increasing the temperature with radiant heat is commonly used to induce materials to better absorb microwaves. Once the material reaches its critical temperature, microwave absorption is coupled with radiant heat sufficient to cause self-heating.²⁷

The microwave processing of materials has until recently been limited to ceramics, semimetals, and inorganic and polymeric materials. Few detailed reports on the microwave processing of metallic materials are available. Metallic materials in powder form absorb microwaves efficiently.¹⁵ Bulk materials preheated to moderate temperatures will begin

coupling in a microwave field, resulting in rapid heating. The lack of research in the microwave-assisted sintering of metals stems from the misconception that all metals reflect microwaves or cause plasma formation (or both). On the contrary, placing a bulk metal in a microwave oven does not create plasma formation. However, a highly reflective sheet of aluminum foil would interact with microwaves, causing them to travel back to the microwave generator (Magnetron) and damage it. Likewise, sparks would likely be created with the presence of fork-like, sharp metal tips placed closely together.

In this study, joining solid metals with [microwave heating](#) in a specially designed microwave oven was tested. This custom-built microwave oven used silicon carbide susceptors (microwave absorbers) enclosed in a ceramic insulating chamber placed on the turntable. As microwaves passed through the thermal insulating chamber, they were immediately absorbed by the susceptors, generating radiant heat. The material in the oven was exposed simultaneously to heat and microwaves. This oven incorporated a water jacket to cool the system and could rapidly generate and sustain heat up to approximately 1500°C at a rate of 100°C per minute. The melting range of the solder used in this study (HFWC) was 1100°C to 1165°C. The solder assembly was heated to 1200°C for approximately 15 minutes to ensure the complete flow of the solder. As the specimens reached the definitive temperature, the unit was turned off manually. All specimens were subsequently bench cooled.

Joining and brazing in a microwave oven produced strong joints (see [Table III](#)). The highest tensile strengths were placed in the as-cast group (745 ±19 MPa), followed by microwave-brazed specimens (420 ±68 MPa) and the conventional torch-brazed joints (348 ±103 MPa). The microwave group exceeded the ADA requirement. The tensile strength of the conventional torch group was slightly below that required by the ADA requirement (350 MPa).

To achieve statistically significant differences between the torch and microwave groups, the necessary sample size can be determined based on the differences seen in the current study. For elongation, the observed mean difference was 0.66% with a standard deviation of 2.36. A power analysis estimated that an experiment with 720 specimens (240 for each of 3 groups) would be required to have 80% power to achieve statistical significance at the 5% level. For tensile strength, the observed mean difference was 72 MPa with a standard deviation of 72. In this case, an experiment with 63 specimens (21 per group) would have 80% power to detect statistical significance. Based on the results of the current experiment, in a larger experiment, the microwave technique would outperform the torch technique on each measurement.

On the SEM images, voids, porosities, and flux inclusions appeared on all the fractured interfaces. In torch-brazed specimens, incomplete sintering of the braze material at the gap center and the presence of irregular-shaped porosities were observed (see [Fig. 4](#)). Microwave specimens demonstrated more even sintering, uniform joints, and the presence of the round-edged porosity in the brazed joints (see [Fig. 5](#)). The grain size of all specimens was not determined from the SEM images in this study. One of the significant advantages of microwave heating over conventional heating is that the microwave-sintered specimen exhibits a finer grain size and more even sintering owing to its volumetric and internal heating. A finer grain size is believed to be responsible for the mechanical strength of ceramic materials as described by Katz.¹⁴ A further investigation of the grain size with microwave sintering is

indicated. Microwave heating may also reduce hydrogen and oxygen content in a gold-palladium alloy by eliminating the use of the gas torch. The high hydrogen content found in cast alloys with gas-torch heating often results in defect precipitation, unlike with electric heating, as proposed by Engström et al.²⁸ Further studies on a postceramic brazing with a low-fusing solder with or without an inert gas environment will follow.

Conclusion

This investigation evaluated an experimental method of brazing alloys with a [microwave](#) oven. Within the limitations of this study, the following conclusions were drawn. The sintering of a dental brazing alloy with a microwave-assisted oven is feasible. The microwave-assisted brazed joints demonstrated high values in tensile strength exceeding ANSI/ADA Standard No. 88, although the microwave method was not statistically different from the conventional torch method. The microwave-assisted brazing method may be an effective technique for joining a [dental ceramic](#) alloy.

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