




ORIGINAL ARTICLE

Effect of different firing atmospheres on debonding strength of dental porcelain fused to commercially pure titanium

Jen-Chyan Wang^{1,2,5}  | Po-Sung Fu^{1,3} | Pei-Ling Lai² | Chen-Shih Liu¹ |
Wen-Cheng Chen^{4,5}  | Chun-Cheng Hung^{1,2,5} 

¹School of Dentistry, College of Dental Medicine, Kaohsiung Medical University, Kaohsiung, Taiwan

²Division of Prosthodontics, Department of Dentistry, Kaohsiung Medical University Hospital, Kaohsiung, Taiwan

³Department of Dentistry, Kaohsiung Municipal Ta-Tung Hospital, Kaohsiung, Taiwan

⁴Advanced Medical Devices and Composites Laboratory, Department of Fiber and Composite Materials, College of Engineering, Feng Chia University, Taichung, Taiwan

⁵Dental Medical Devices and Materials Research Center, College of Dental Medicine, Kaohsiung Medical University, Kaohsiung, Taiwan

Correspondence

Wen-Cheng Chen, Department of Fiber and Composite Materials, Feng Chia University, No. 100, Wenhwa Rd., Seatwen, Taichung 40724, Taiwan.
Email: wencchen@fcu.edu.tw

Chun-Cheng Hung, School of Dentistry, College of Dental Medicine, Kaohsiung Medical University, No. 100, Shih-Chuan 1st Road, Kaohsiung 80756, Taiwan.
Email: yuchali@ms21.hinet.net

Funding information

Ministry of Science and Technology, Taiwan, Grant/Award Number: MOST 99-2314-B-037-051-MY3 and contract of CZ-02-07-22-98

Abstract

An in vitro investigation was performed to evaluate the bonding characteristics of porcelain fused to metal (PFM)/commercially pure titanium (cp Ti, grade II) in three firing atmospheres of under vacuum and using two noble gases argon (Ar) and helium (He). Three groups of porcelain veneers firing under vacuum, Ar, and He were prepared to evaluate the bonding of porcelain fused to the cold-rolled cp Ti. The bond strength of PFM durability by a three-point bending test, phases, microhardness of cp Ti after firing processes, and fractures were measured and evaluated. Results show the microhardness of cp Ti in group of porcelain firing under He atmosphere was significantly lower than that of the two other groups, which were in vacuum and Ar ($P < .05$). X-ray diffraction showed the He group produced in relatively small amounts of TiO_2 and TiO oxides than other groups but featured relatively high quantity of airhole defects in the porcelain body leading to the lowest bond strength. The Ar group presented the highest bond strength of comparing with the groups under vacuum and using He ($P < .05$). Although the firing processes in He could efficiently prevent the diffusion of oxygen into Ti, the porcelain-cp Ti bond strength using Ar protective atmosphere presented the advantage to achieve clinical requirement because porcelain firing under He revealed prominent voids and defects within the body of porcelain.

KEYWORDS

bending, bonding strength, commercially pure titanium (cp Ti), noble gases, porcelain fused to metal (PFM)

1 | INTRODUCTION

The production of metallic restorations, such as crowns, bridges, inlays, cast posts and cores, and partial dentures, has been carried out

for decades. Since the 1960s, metal-ceramic restorations have been popularly used in intraoral repair of ceramic fractures. Different approaches have been explored to improve the bond between metals and ceramics. The requirement for esthetic restorations has led to the

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development of ceramic systems that can be associated with alloys. The alloys of choice were originally high-gold alloys, but gold prices soared in the 1970s; thus, the low-gold alloys were gradually developed, and the gold alloys were finally replaced by palladium and cobalt-chromium alloys for the construction of removable partial dentures.¹ Commercially pure titanium (cp Ti) has become a popular dental material because of its excellent physiochemical properties, good corrosion resistance, and biocompatibility.^{2–4} The excellent properties of cp Ti include relatively light density (density 4.5 g/cm³) and low heat transfer; thus, this material is less irritating to the pulp for dental use, features excellent corrosion resistance and good biocompatibility, and is cheaper than noble alloys.^{5,6}

The rapid development rate of dental restorative application using Ti substrates combined with low-fusing ceramics began in the last decade. However, firing of porcelain over Ti needs a special protocol because Ti features several disadvantages, such as low deformability and wear resistance, difficulty in manufacturing, welding, and machining. Moreover, the melting point of Ti reaches as high as 1700°C, resulting in an intense reaction with the embedded casting materials or the surrounding impurities of oxygen (O₂) and nitrogen (N₂) gases at elevated temperatures. The formation of alpha case (α -case), which not only includes an oxide layer on the surface but also an oxygen interstitial layer beneath the metal surface, implies that a hard and brittle oxygen-enriched transition layer exerts a detrimental effect on the mechanical properties.^{7,8} The hardness of α -case, which is due to the Ti phase transformation during cooling processes of Ti through high heat treatment, is higher than that of cp Ti, causing ductility and fatigue resistance of cp Ti deterioration after fusing dental ceramic (also known as dental porcelain) to cp Ti. The bond strength of metal-ceramic can be considerably improved by removing α -case using chemical milling or pickling before fusing the ceramic once α -case is already formed.⁹ However, completely removing the α -case is difficult because it needs considerable time and leads to other harmful issues in hydrogen uptake.¹⁰ Therefore, the formation of α -case remains the largest obstacle to the bonding of titanium with ceramic and must be avoided as much as possible.

The porcelain-cp Ti bond strength is affected by three factors: embedding material, dental porcelains with various composition and properties, and using atmospheres during porcelain firing for ceramic fused to cp Ti.^{11,12} Contrary to conventional alloys through heat treatment, Ti is tensely oxidized during porcelain firing at temperature above 800°C, providing a thick and nonadherent layer of TiO₂. Therefore, the firing procedure of porcelain fused to metal (PFM) generally operates in vacuum suction. To advance the protection, firing procedure operating in the protective atmosphere of noble gases, such as argon (Ar), has been used.¹³ The phenomenon of α -case development in casting can be effectively reduced by Ar insertion during firing, Ti can still react with ceramics during high-temperature firing. Even though evacuation reading of the vacuum system of a vacuum casting machine uses full vacuum and is refilled with protective noble gases, completely eliminating the problem of residual minimal reactive oxygen remains difficult.¹⁴ In the dental technical field, Ar is the most used protective gas in the cp Ti casting procedure; however, helium (He) is another preferred choice.⁴ Several manufacturer protocols use He as gas for casting Ti,

and the operating instructions indicate that using He in cp Ti casting procedures would increase the success rate. The use of different noble gases for investigating porcelain-cp Ti bond strength compared with PFM procedures general operating under vacuum remains rare.

The dentist selects the most suitable alloy for proper application in esthetic requirements to operate the prosthesis. However, the generally used dental porcelain furnaces for the firing porcelain fused to cp Ti have been limited. Using different atmospheres is believed to affect the bond strengths of firing porcelain fused to cp Ti. In this study, three porcelain firing atmospheres, namely, under vacuum and using protective noble gases (Ar and He), were used to investigate the effects of porcelain-fused cp Ti reactions on bonding strengths, morphologies, and interfaces.

2 | MATERIALS AND METHODS

2.1 | Sample preparation and veneering with recommended layering ceramics

The commercial Ti (ASTM grade II, Kobe Steel Co., Japan) plate samples underwent the cold rolling process and were cut with length \times width \times thickness dimensions of 25 mm \times 3 mm \times 0.5 mm according to ISO 9693 protocol.¹⁵ The specimens were initially grounded using 240-grit sandpaper to standardize the surface conditions. The specimens were sandblasted at an angle of 45° with Al₂O₃ particles (mean: 110 μ m) by utilizing an air compressor under 2–3 bar pressure blasted over a 3 cm distance for 10 seconds. All specimens were washed in acetone for 10 minutes in the ultrasonic cleaner and dried under vacuum overnight before firing the porcelain.

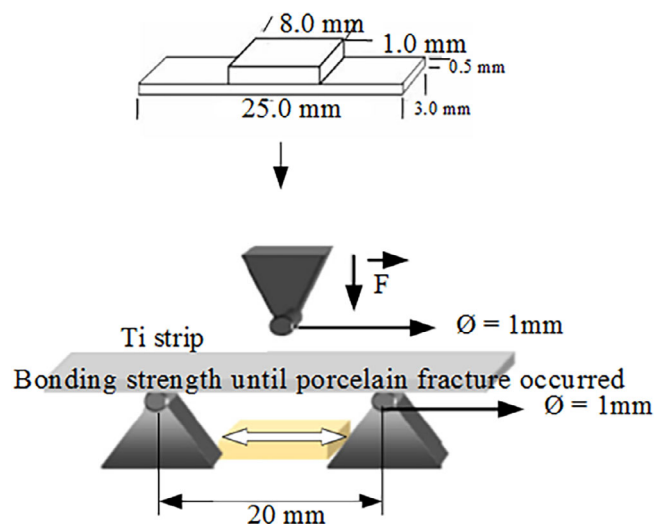
To measure the bond strength between Ti and veneering porcelain, the coats of opaque low-fusing porcelain of Vita Titankeramik (Vita Zahnfabrik, Bad Säckingen, Germany) condensed by vibration and blotting procedures were applied on the specimen surface. The porcelain veneer (8.0 mm \times 3.0 mm \times 1.0 mm) was prepared using a custom designed mold. The built up and firing parameters of the porcelain followed the manufacturer's instructions after subsequently applying each approximately 0.1 mm-thick porcelain bonding agent (bonder), opaque powder mixed with opaque fluid, and dentine powder mixed with modeling liquid (Table 1). The body porcelain on the metal specimen surface was fired in the porcelain furnace (Whip Mix Pro 100, Whip Mix, Louisville) through three different atmospheres, under vacuum and using the noble gases Ar and He, to fire the porcelain fused on cp Ti. After firing the porcelain veneer under vacuum or using 0.5 atm pressure of Ar and He, a 1 mm-thick uniform porcelain with controlled deviation (\pm 0.05 mm) was selected and applied along an 8 mm length in the central portion of the metal specimen surface. The testing configuration shown in Figure 1 illustrates the relationship between the specimen and the rollers of the three-point bending jig.

2.2 | Evaluation of metallic surface conditions

To evaluate the surface conditions after different firing porcelain environments, the microhardness test was performed on the metallic part

TABLE 1 Firing schemes used for commercial fusing porcelains (Vita Titankeramik): Firing technological parameters

Firing cycle/compositions	Base/preheat temperature (°C)	Heat raising time (min)	Heat raising rate (°C/min)	End/firing temperature (°C)	Holding/firing time (min)
Paste bonder/first	400	6	67	800	6
Opaque/second	400	4	98	790	2
Dentine firing/body porcelain	400	7	53	770	6

**FIGURE 1** Bonding test configuration and three-point bending of porcelain fused to cp Ti substrate according to ISO 9693:1999. Loading was applied on the metallic strip with a crosshead speed of 0.5 mm/min, and the direction of crack propagation is shown by a blank double-headed arrow

of the Ti specimens with porcelain with 0.1 mm intervals from 0.5 mm under the outermost surface to 1.0 mm from the surface of each specimen ($n = 5$). A microhardness tester (MZT-50, Matsuzawa Seiki Co., Ltd, Japan) was used at 200g for 20 seconds.

Surface phase characterization was conducted using an X-ray diffractometer (D5000, Siemens, Munich, Germany) operating at 30 kV and 20 mA with Ni-filtered Cu K_{α} (1.54 Å) radiation. The X-ray was operated with a scan angle range of 2θ from 20° to 90° and a scan rate of 4.0°/min. Phases were identified by matching each characteristic peak with those of the Joint Committee on Powder Diffraction Standards (JCPDS) files.

2.3 | Bonding strength, morphology, and element mapping measurements

The specimens with porcelain were subjected to three-point bending tests using a universal testing machine (Shimadzu AG-A ; Shimadzu Crop, Japan). The specimens were loaded at the center with a crosshead speed of 0.5 mm/min (Figure 1). The deflection and load were continuously recorded. The porcelain bonding strengths were determined by a sudden decrease in the load-deflection curves

($n = 6$). The peak load before obvious fracture of the veneered porcelain was recorded, and the metal-to-porcelain bond strength (in MPa) was calculated according to the following formula provided by ISO 9693¹⁵:

$$\text{Porcelain bonding strength} = F \times k,$$

where F is the strength load (N) until debonding occurred, and k is a coefficient calculated from the ISO specification equal to the value of 4.6 mm^{-2} for cp. Ti is dependent on the modulus of elasticity of the alloy and height of each specimen.

The ceramic bond profile of cp Ti with firing porcelain groups under three atmospheres was studied through its topographic and cross-sectional fracture images obtained via scanning electron microscopy (SEM, JEOL JSM-6700F, Japan) coupled with energy-dispersive spectroscopy (EDS).

2.4 | Statistical analysis

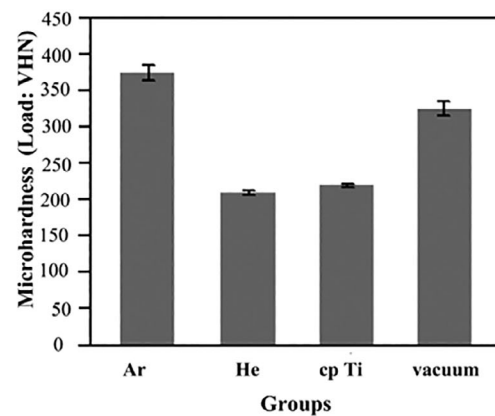
Statistical analyses were conducted by one-way analysis of variance (ANOVA)/Tukey-Kramer honestly significant difference test at a 5% significance level.

3 | RESULTS AND DISCUSSIONS

3.1 | Metallic surface conditions and microhardness

Compared to smooth surfaces, porcelain interfaces can be physical locked on rough surfaces, which were fabricated in various methods, one of the most important manufacturing parameters of cp Ti is cold working and sandblast to roughen of the surface for increasing the effective surface area to adjacent to the porcelain interface, thereby improving the anchored force. In this study, the formed oxidation layer on the surfaces of cp Ti through cold working and sandblast after firing porcelain fused to Ti under Ar protective atmosphere yielded the highest hardness value, whereas the hardness value in He group was the lowest and showed comparable hardness with untreated cp Ti (Figure 2). The high-temperature firing of cp Ti under different atmospheres showed a significant effect on the surface hardness ($P < .001$, Figure 2). Post hoc analysis by Tukey-Kramer test exhibited no significant differences when comparing the values of cp

FIGURE 2 Microhardness of cp Ti under different atmospheres under vacuum and using noble gases Ar and He ($n = 5$) and the statistical results based on one-way ANOVA for comparison



Source	df	SS	MS	F Ratio	Probability > F statistic
Atmospheres (vacuum, Ar, and He)	3	586998.72	195666	76.19	<.0001**
Error	96	246547.31	2568		
Total	99	833546.03			

df, degree of freedom; SS, sum of squares; MS, mean squares; ** indicates that the test groups were significantly different ($p < 0.0001$)

Ti between the He group and untreated cp Ti. However, the statistical significance is evident among the four groups.

Titanium can form different chemical equivalent oxides, such as TiO_{1+X} ($0 < X < 1$), TiO , Ti_2O_3 , Ti_3O_5 , and especially TiO_2 . The phase existing in the outermost oxide layer, which is also the most abundant oxygen layer, is usually TiO_2 . When the temperature is higher than 883°C , the cp Ti will change its phases from α to β ,¹⁶ leading to numerous oxygen atoms entering the grains. Once the temperature is cooled, the β phase of Ti will return to α phase, and an outer layer named “ α -case layer” will be generated.^{8,17} The hardness of the α -case layer is about three times that of the Ti oxides (600 vs 200 KHN) due to the oxygen diffusion results with the formation of an oxygen enriched layer beneath the oxide scale which occurs in oxide/Ti interface with the high solubility of interstitial oxygen diffused in α -Ti.¹⁸ This layer of oxygen-rich surface lacks ductility and shows poor fatigue resistance. The presence of this α -case layer will also deteriorate the bonding force between the porcelain and cp Ti.¹⁹

By performing Ti under heat treatment in vacuum or noble gas atmospheres, α -case layer can be limited and even under the high temperature porcelain-fused firing processes, could be used to prevent oxygen from ceramic to enter the base Ti lattices. The proper surface hardness indicate the appropriate oxide interfaces formation could inference cp Ti bonding to porcelain. That is important for the restorative materials and fillers used in dental applications. This parameter reflects the material's surface abrasion resistance, impact resistance, and the ease of polishing.²⁰ In this experiment, the surface hardness of the Ar and vacuum groups was significantly higher than that of the He and untreated cp Ti groups under porcelain-fused firing processes.

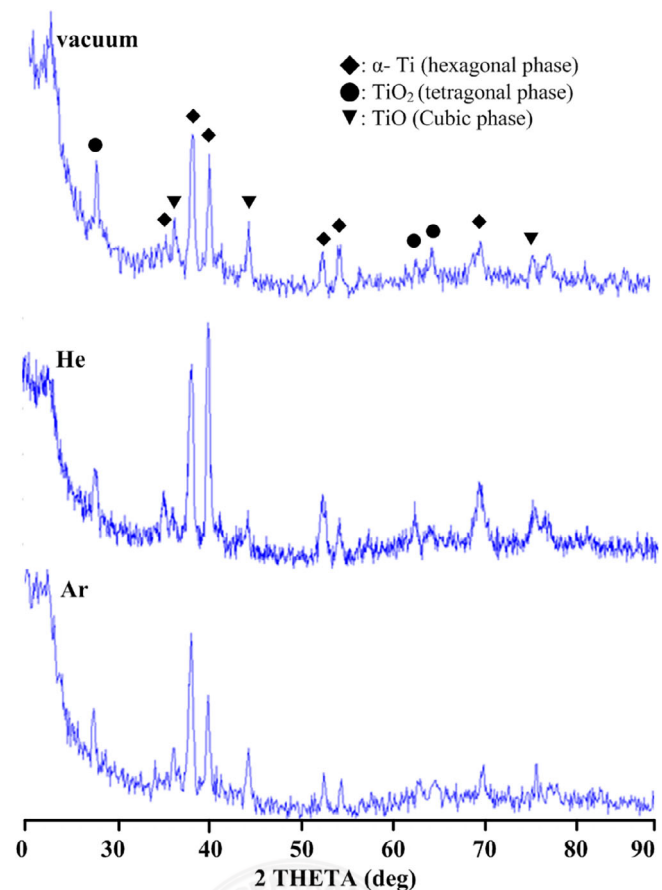


FIGURE 3 XRD patterns of cp Ti under vacuum and noble firing gases Ar and He

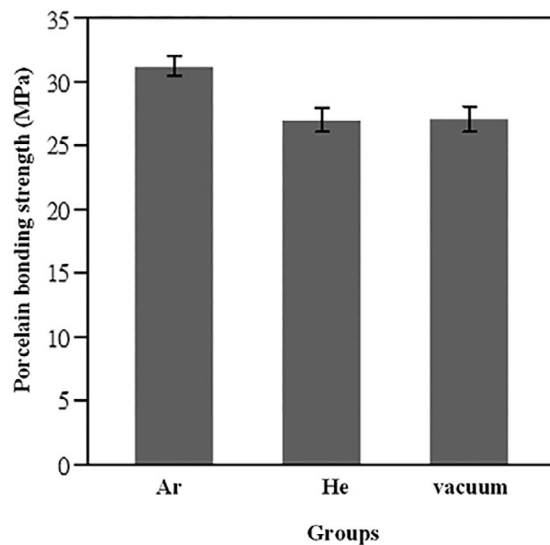


FIGURE 4 Porcelain bonding strengths and statistical results based on one-way ANOVA for comparison of different firing atmospheres

Source	<i>df</i>	SS	MS	F Ratio	Probability > F statistic
Atmospheres (vacuum, Ar and He)	2	58.31	29.16	7.37	0.008*
Error	12	47.41	3.95		
Total	14	105.72			

df, degree of freedom; SS, sum of squares; MS, mean squares; * significantly different ($p < 0.05$).

3.2 | Surface phase characterization by X-ray diffraction

Figure 3 shows the X-ray diffraction (XRD) patterns of the cp Ti after firing porcelain fused to Ti under three protective atmospheres. Based on JCPDS file comparisons, no significant differences were observed in the diffraction angles. The cp Ti pattern generally matched the diffraction data of α -Ti (hexagonal phase), and a considerable quantity of the compact oxide layer of TiO_2 (tetragonal phase) and TiO (cubic phase) was observed in all three experimental groups.

XRD patterns were used to observe the lattice structure changes in the cp Ti after firing porcelain fused to Ti under different atmospheres. Despite the lack of significant differences in the relative high peak angles of the three experimental groups, the oxidation rutile TiO_2 peaks in Ar and vacuum groups were higher than those in He group. As the oxide phase of rutile TiO_2 is the most stable and the most abundant within the three cp Ti oxidation of TiO_2 (rutile, brookite, and anatase),²¹ Oxidation in the vacuum and Ar groups was slightly more evident than that in the group, resulting in the reasonably higher hardness of the Ar and vacuum groups than that of the He group. Residual oxygen and water vapor are the major atmospheric constituents that can create oxidation within the PFM processes under the vacuum system. In addition to the residual stress generated by cold rolling, metallic defects, such as vacancies and stacking

defects, would be generated during the firing processes. As the cumulative amount of deformation increased, the grains deformed along the direction of the force and became elongated crystal grains, changing the relative intensities of the XRD patterns (Figure 3).

3.3 | Porcelain bonding strength

After the three-point bending test, the porcelain bonding strength obtained by firing the group under Ar was significantly better than that of groups under He and vacuum atmospheres (Figure 4). The statistical results of one-way ANOVA showed that considering the use of different atmospheres to porcelain fused to cp Ti, and a significant difference was noted in the effect of flexural bonding strength ($F = 7.37$, $P < .05$, Figure 4). The bonding strength of porcelain to cp Ti under the Ar group was significantly higher than that of other groups, whereas no significant difference was noted between the vacuum and He groups ($P > .05$). According to specifications of DIN 13927 and ISO 9693 criteria, the required bond strength for clinical dentistry should meet a minimal requirement of 25 MPa for PFM.^{15,22} In this experiment, the porcelains fused to cp Ti under three different atmospheres all exceeded such standards. The bond strength in the Ar group provided the best capability and potential application for the enhanced restoration potential of porcelain bonding strength.

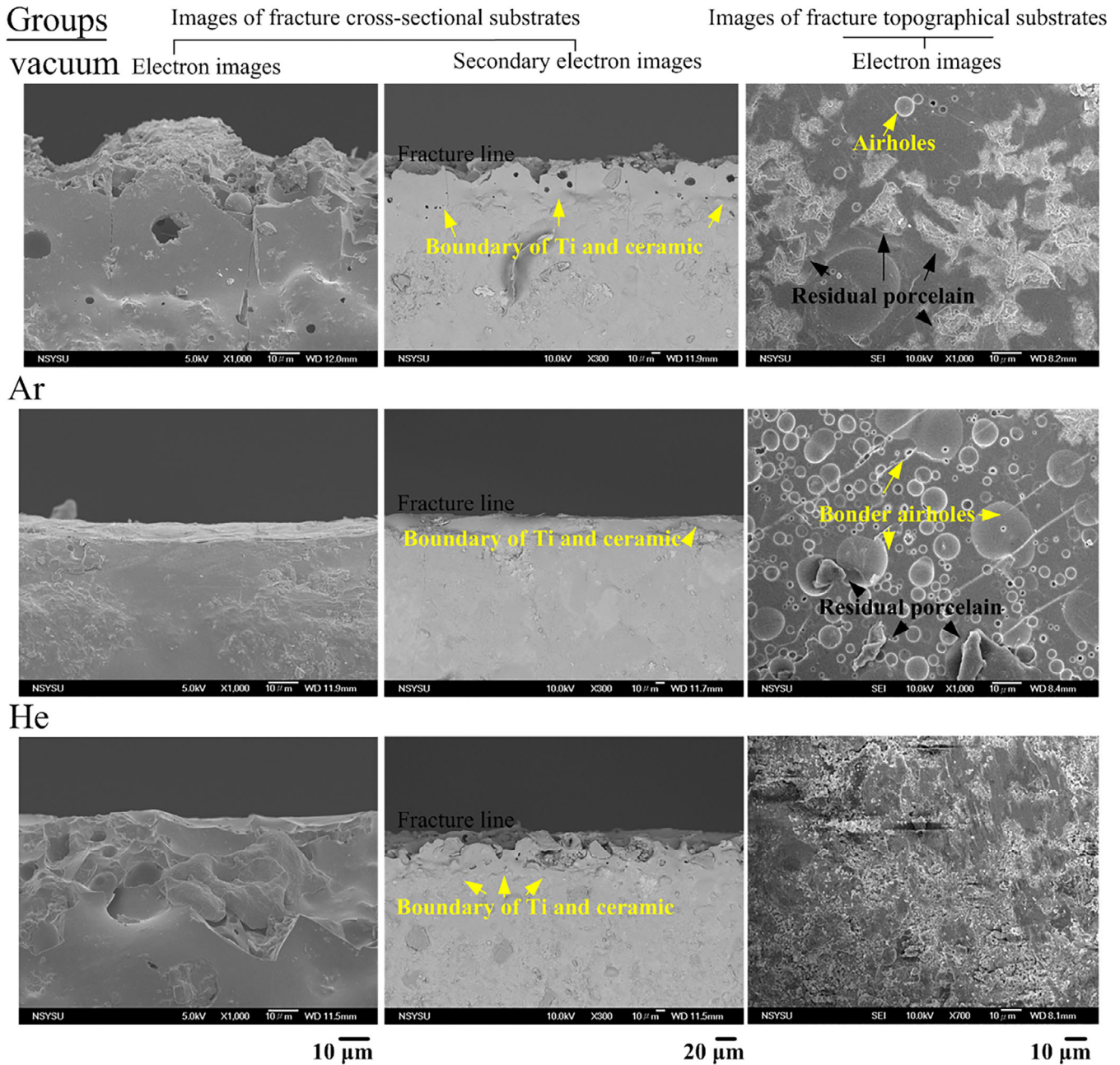


FIGURE 5 SEM electron images (left) and secondary electron images (middle) of fracture cross-sectional substrates and fracture topographical images (right). A distinct zone of the boundary is pointed by arrows in the middle images

3.4 | Fracture surface characterization-morphology and element mapping measurements

After the bending test, all specimens in the three groups contained residual porcelain fragments on the fracture surface of cp Ti. However, the highest residual porcelain was observed on the Ti surfaces of the He group, and the least residual porcelain was observed in the Ar group (Figure 5). The marginal line of cross-sectional fracture surfaces between the cp Ti and porcelain showed almost no trace of residual porcelain in the Ar group, and the topographical surfaces and cross-sectional/marginal fracture line were

smooth. By contrast, a considerable amount of porcelain attached without binder lenticular voids was observed on the fracture topography of the He group (Figure 5). Although observation in the vacuum group also revealed voids within the porcelain, the degree of void content and distribution was between that of the Ar and He groups. Applied secondary electron image is useful for observing the marginal composition difference. Therefore, the interface between the porcelain and cp Ti can be specified (Figure 5, middle images). The representative secondary electron images from fractured surfaces, as represented by indicated parts of relative brightness and darkness, were referred to as cp Ti and porcelain, respectively. Minimal residual

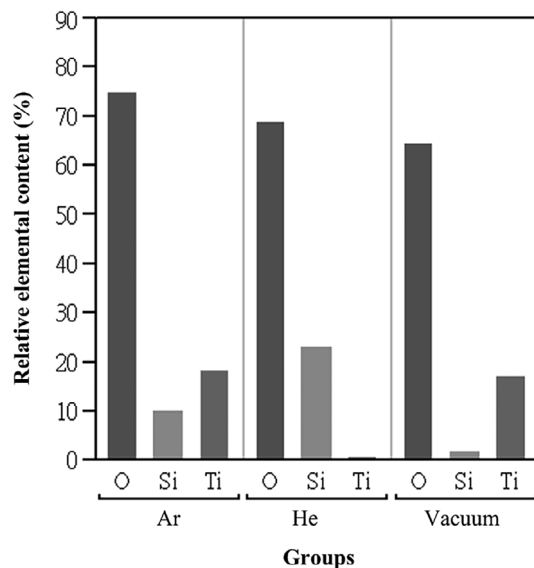


FIGURE 6 Semi-quantitative elemental Ti, Si, and O analysis of the fracture surfaces of cp Ti under different firing atmospheres, that is, under vacuum and using noble gases Ar and He

amounts of porcelain were found on the surface in the Ar group. Moreover, additional porcelain internal bubbles resulted in holes in the He group. The use of Ar atmosphere in firing PFM ceramics showed fewer holes between the metal and ceramic interfaces and therefore the bonding is strongest. It is considered that firing ceramic under Ar atmosphere can reduce the defects on oxidation phenomenon when the oxygen atom of ceramic diffused to base metal of cp Ti and improve the bonding of Ti and ceramics relative to firing in a vacuum and in the He (Figure 5). It was concluded that PFM ceramics firing in the Ar exhibited significant higher bonding strength than He group.

3.5 | Element mapping measurements on fracture cp Ti surfaces

The elemental mapping of the fracture surfaces in the three experimental groups was analyzed by SEM equipped with an EDS system for microanalysis (Figure 6). The evidently greater density of Si and O mapping indicates that fracture surfaces occurred in the porcelain. Elemental mapping of Ti on the topographical fracture surface showed an extremely minimal quantity especially in the He group, but the proportion of Si was considerably higher than that of Ar and vacuum groups, indicating that the amount of residual porcelains on the surface was highest in the He group.

As early as 1967, Leone compared PFM bonding in the atmospheres of Ar and vacuum in which the porcelain was fused to gold alloys.²³ Results showed the weaker bonding capability of the PFM prepared in Ar than that of the specimen in vacuum. The opposite results were revealed in the studies of Pask and Tomsia, who used 80Ni20Cr alloy as the PFM base metal: the surface oxide layer reduced under Ar, and the bonding force of alloy with porcelain increased.²⁴ They observed that the commercial alloy would form a

porous and buckled single-layer oxide scale with lenticular voids at the porcelain-to-metal interfaces. The scale was penetrated by silica, which formed a chemical bond at the alloy/porcelain interface, resulting in excellent adherence with fracture at the interface boundary, consistent with the findings of this study. Bonding was satisfactory because fracture occurred in the binder phase (Figure 5). The voids within the bonding interface between the metal and porcelain using Ar firing atmosphere was evidently less than that of the PFM using vacuum firing in relevant literature.²⁵ Due to it is well known that oxygen diffusion plays the most important role in the increase of hardness as with the measures of hardness, PFM firing in Ar atmosphere was therefore effectively reduce the formation of ceramic defects and exhibited higher bonding strength than other groups. Below the base metal of Ti surface, the grains would try to restore the surface to its original shape and phase during the cooling, thereby producing the airholes of the defects within the boundary. Reducing defects within ceramic exists a higher interfacial strength of adhesion level of porcelain to Ti was realized.

In addition to Ar, He is a popular noble gas used in the casting of Ti. He features the following advantages: easily obtainable and can be operated at low injection velocity during casting. He also possesses a small atomic radius (radius: He = 0.05 nm; Ar = 0.192 nm). Thus, this atom easily traverses the holes in the embedded material, reducing back pressure and eliminating the holes or defects of casting metals.^{25,26} However, the mechanical properties of casting metals using different gas atmospheres are generally inconsistent with the bonding strength of PFM because strength would be directly affected by the surface condition, oxidized layers, firing lenticular voids, and heat treatment during veneering porcelains.^{27–30} Therefore, a critical understanding of the mechanical properties of the different alloys used in practical processes is essential to ensure and simulate the maintenance and life of fixed partial dentures with veneering porcelains. It is worth mentioning that the bonding of the oxide layer with PFM is very sensitive to the defects of the oxidation layers of cp Ti, that is, the sintering process using different firing atmospheres.

4 | CONCLUSIONS

The three-point bending test was used to study the porcelain-cp Ti bonding strength under three different atmospheres. The conclusions obtained include the followings:

1. In the group of Ar atmosphere, the porcelain-cp Ti bonding strength was significantly higher than that of the groups under vacuum and He atmosphere.
2. The cross-sectional fractures showed numerous lenticular voids in the interior of the porcelains. The least residual porcelain content was evident in the Ar group, and the largest residual porcelain content was observed in the He group, which also featured relatively high quantity of defects in the porcelain body because the extremely small He molecules might lead to rapid diffusion rate, resulting in the easy occupancy and entrapment in the porcelain body space during firing. Therefore, the fracture occurred inside the porcelain body when bending force was applied.

3. In the comparison of base metal hardness of cp Ti, the Ar group showed the highest value, and no significant difference existed between the He group and the untreated cp Ti. The results of phase diffraction analysis showed that the oxidation reaction of the Ar and vacuum groups after high-temperature firing was more evident than that of the He group.
4. No evident relationship was observed between the porcelain and oxidation layers of cp Ti for the porcelain-cp Ti interfacial bonding strength possibly due the extremely complex chemical compositions of porcelain. The porcelain-cp Ti bonding strength in the firing process using Ar atmosphere exhibited the best condition, and such bonding strength meets the clinical requirement.

CONFLICT OF INTEREST

The authors declare no potential conflict of interest.

ORCID

Jen-Chyan Wang  <https://orcid.org/0000-0001-6318-233X>

Wen-Cheng Chen  <https://orcid.org/0000-0002-5581-9991>

Chun-Cheng Hung  <https://orcid.org/0000-0001-7461-7095>

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How to cite this article: Wang J-C, Fu P-S, Lai P-L, Liu C-S, Chen W-C, Hung C-C. Effect of different firing atmospheres on debonding strength of dental porcelain fused to commercially pure titanium. *Kaohsiung J Med Sci.* 2020;36:212–219. <https://doi.org/10.1002/kjm2.12157>

