

A Scanning Electron Microscopy and Energy Diffraction X-Ray Spectroscopy Study to Evaluate the Effect of Firing Temperature at the Ceramic-Noble Metal Alloy Interface in Porcelain Fused to Metal Restorations

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ABSTRACT

Introduction: Porcelain Fused to Metal (PFM) restorations offer aesthetics of ceramics and the strength of metal hence are the gold standards of fixed partial dentures. However, the fracture of porcelain-metal interface still remains a matter of concern.

Aim: To study the porcelain-noble metal alloy interface of PFM restorations at different firing temperatures, using Scanning Electron Microscopy (SEM) and Energy Diffraction X-ray Spectroscopy (EDS).

Materials and Methods: This in-vitro study was conducted between November 2018 to October 2019 at Prosthodontics Department of Saraswati Dental College and Hospital and King George's Medical University Lucknow, Uttar Pradesh, India. A total of 75 strips of noble metal alloy were prepared and layered with 1 mm of porcelain on one surface, conforming to American National Standard/American Dental Association Specifications (ANSI/ADA) specification no. 38 for Metal-Ceramic Dental Restorative Systems: 2015. These were randomly divided equally into three groups. Specimens of each group (n=25) were fired at different temperatures that is 850°C, 900°C and

960°C, respectively. Scanning electron microscopy and Energy diffraction X-ray spectroscopy were performed at the noble metal alloy and ceramic interface of all specimens. Data was recorded and statistically analysed using one-way Analysis of Variance (ANOVA) and post-hoc Tukey HSD test.

Results: Irregularities/Coarseness (50 µm) was seen on the noble metal alloy surface adjacent to ceramic layer in SEM images at all the three chosen temperatures. The EDS study revealed intermingled zones of partial oxygen depletion at the interface region with formation of intermetallic compound, lead zirconate titanate and complete oxygen depletion zone near metal alloy end of interface. The mean value of Shear Bond Strength (SBS) was 16.31 MPa at 850°C, 24.33 MPa at 900°C and 19.41 MPa at 960°C (p-value <0.05).

Conclusion: Difference in properties and location of the intermetallic compound formed at the interface, as well as the location of the oxygen depletion zone could account for the weaker bond formed between noble alloy-porcelain interface compared to base metal-porcelain interface.

Keywords: Bonding, Intermetallic compound, Oxide layer, Oxygen depletion zone, Shear bond strength

INTRODUCTION

Metal ceramic restorations have been used since many years, and proved to be aesthetically pleasing, fracture resistant and bio-compatible. Although all ceramic restorations offer superior aesthetics compared to Porcelain Fused to Metal (PFM) restorations [1]; yet, the latter remain popularly used aesthetic restorations because of their resistance to fracture and are preferred especially, in a resource limited setting. In case of PFM restorations, coping can be made either with High Noble (precious), Noble (semi-precious) or Base metal alloys (non precious metal alloys). High noble alloys contain more than or equal to 40 weight% Au and more than or equal to 60 weight% of noble metal elements (Au, Pt, Pd, Rh, Ru, Ir, Os). Noble metal alloys must have more than or equal to 25 weight% of noble metal elements (Au, Pt, Pd, Rh, Ru, Ir, Os) and Base metal alloys contain less than 25 weight% of noble metal elements. High Noble metal alloys have been used historically while base metal alloys are used extensively at present because of being economical [2-6].

Noble metal alloys contain considerable amounts of silver and palladium and have properties intermediate to the other two groups. Noble metal alloys can be grouped into-

- Palladium-gold alloys- 57% Pd (palladium), 35% Au (gold), 5% Ga(gallium) , 2.8% Sn (tin), Zn (Zinc), In (Indium)

- Palladium-gold-silver alloys- 42% Pd, 32% Au, 14% Ag, 3% Pt (platinum)OR 74% Pd, 6.5% Ag (silver) , 5% Au
- Palladium-silver alloys- 60% Pd, 28% Ag, 12% Sn, Zn, In OR 54% Pd, 38% Ag), and
- Palladium-copper-gallium alloys - 75% Pd, 10% Cu (copper), 5% Ga, 2% Au OR 75% Pd, 10% Cu, 9% Ga.

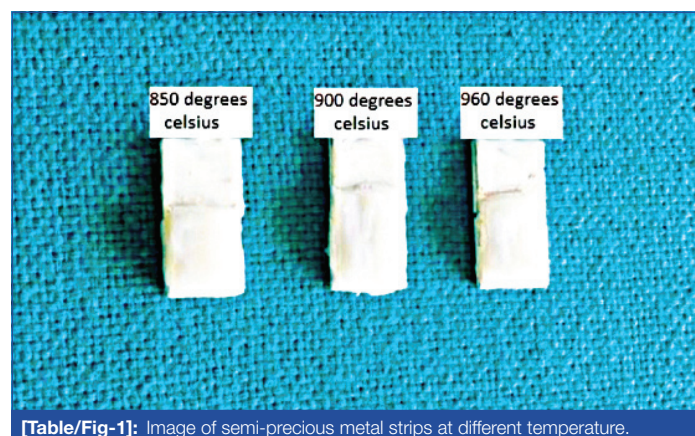
The majority of nickel-chromium alloys for crowns and Fixed Dental Prosthesis (FPD) contain nickel, chromium and molybdenum. The cobalt-chromium alloys typically contain cobalt, chromium, and molybdenum, which probably affect the metal-ceramic bond. The longevity of PFM restoration depends mainly on metal-ceramic bonding. Porcelain fracture is considered as the main reason for failure of metal-ceramic prosthesis. Based on the placement of fracture it can be: 1) Adhesive fracture: failure of bonding between the veneered porcelain and the metal base; 2) Cohesive fracture: occurs within the porcelain. The porcelain is chipped off and some of it remains adhered to the metal framework; 3) Mixed fracture: it is combination of adhesive and cohesive fracture [7]. Even in the case of zirconia, the core needs pretreatment with silica [8] or hydrofluoric acid and silane [9], to alter surface topography and enhance wettability in order to improve veneer bonding. It is vital to understand the mechanism of metal alloy-ceramic bonding in order to minimize failures of restorations due to fracture or chipping of porcelain.

Various studies [10,11] have evaluated the micro structural changes in ceramo-base metal alloy interface. Earlier explanations of ceramo-metal (predominantly base metals) bonding were based on van der Waal's forces and other modes of chemical-ionic bonding, besides, micromechanical bonding due to surface roughness of alloy [2,10-16]. Newer bonding concepts based on Scanning Electron Microscopy (SEM) and Energy Dispersion Spectroscopy (EDS) studies, postulate that a zone of oxygen depletion and intermetallic compounds plays a pivotal role in base metal alloy and porcelain bonding [1,10,15,16,17]. SEM produces images of surface topography of a given sample by focusing an electron beam. Interaction of electrons with surface atoms produces signal containing information about surface topography and composition of the sample. The EDS is used for elemental analysis and chemical characterization of a sample by focusing a beam of X-ray which emits characteristic X-rays. It can determine the chemical elements present in a sample and their relative abundance. The nature of alloy and firing temperature decides the type of intermetallic compounds formed [18]. Based on the firing temperature, dental porcelains are classified as high fusing (1300°C), medium fusing (1101-1300°C), low fusing (850-1100°C), ultra-low fusing (less than 850°C). Low fusing and ultra-low fusing porcelains are used for construction of FPDs [18]. Firing temperature has effect on both ceramo-metal interface [10], and on the structural and chemical changes of porcelain [19]. There are few studies focusing on ceramic-noble metal alloy interface [19,20].

Therefore, this study was planned to evaluate the effect of firing temperature at porcelain-noble metal alloy interface in PFM restorations and the mechanism of bonding between metal and porcelain using EDS and SEM. A correlation of these changes with the SBS of porcelain to noble alloy was also derived. Shear load tends to produce sliding failure on a material [15], so the SBS test will help in assessing the strength of bond formed between the metal alloy and porcelain. In an earlier study involving base metal alloys maximum SBS was assessed at 900°C, and the ceramic layer never completely separated, but fractured under shear load [10]. Such a study could help in understanding similarity or difference in bonding mechanism of base metal and noble metal alloys to ceramic.

MATERIALS AND METHODS

This in-vitro study was conducted at Prosthodontics Departments of Saraswati Dental College and Hospital and King George's Medical University Lucknow, Uttar Pradesh, India, between November 2018 to October 2019. In accordance with American National Standard/ American Dental Association Specifications (ANSI/ADA) no. 38, (For Metal-Ceramic Dental Restorative Systems:2015) 75 strips of semi-precious alloy [Table/Fig-1] (Begopal-S, BEGO GmbH & Co. KG, Germany) were prepared.



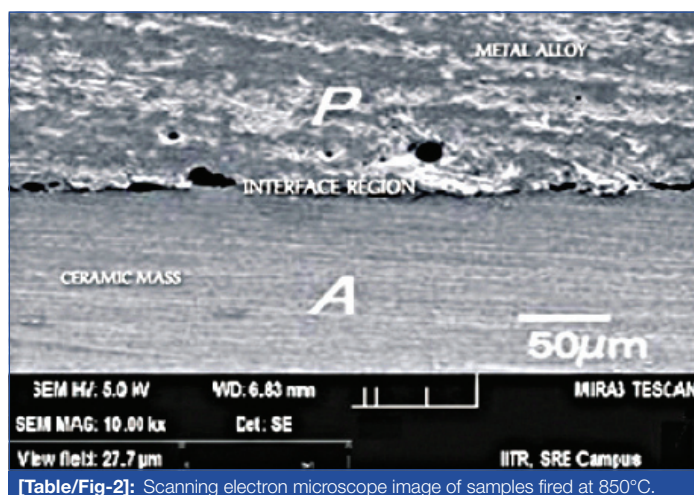
[Table/Fig-1]: Image of semi-precious metal strips at different temperature.

Study Procedure

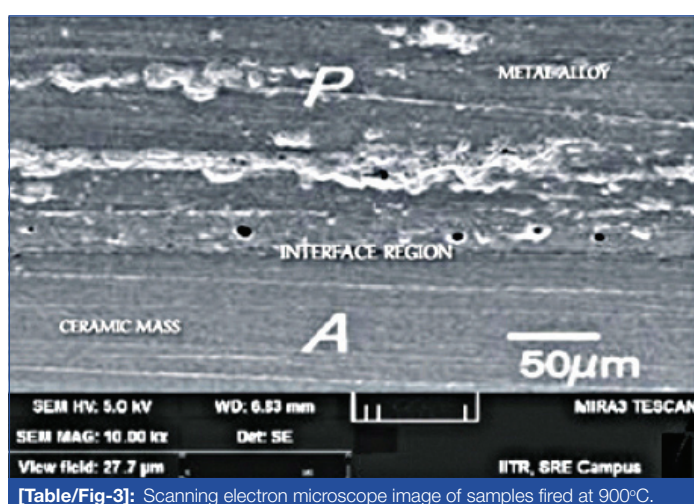
Composition of this alloy is Gold 6%, Palladium 75.4%, Silver 6.2%, Lead and Titanium 6.3%, Zirconium and Copper 6%, Calcium, Sodium and Niobium <1%, with dimensions of 20 mm length, 5 mm width and 3 mm thickness were prepared and surface was finished

similar to a metal coping. These strips were sand blasted with 50 µm Aluminum oxide particles (Sandstorm 2, Vaniman Sandstorm Expert, USA) and then oxidised by open atmosphere heating at 400°C for 10 minutes. Porcelain layering (Ceramco, Dentsply, USA) was done on these oxidised semiprecious alloy strips to a length of 8 mm, a width of 3 mm and a thickness of 1 mm (ANSI/ADA specification no.38).

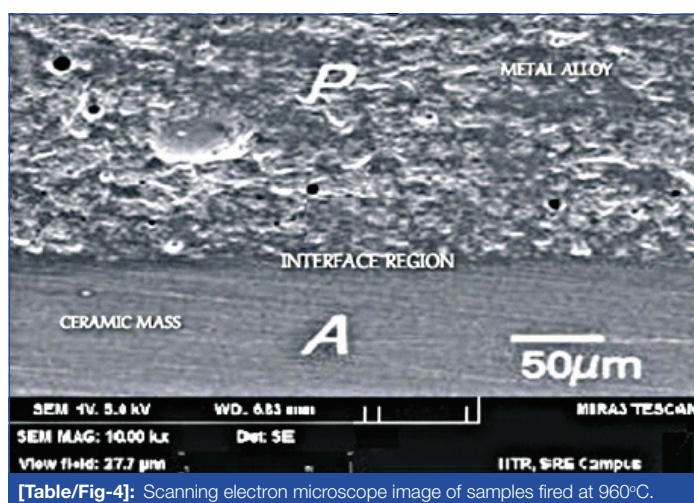
These test specimens were randomly divided equally into three groups using computer generated randomisation sequencing software. Specimens of each group (n=25) were fired to a specific temperature range i.e. 850°C, 900°C and 960°C [Table/Fig-2-4]. These temperature ranges were chosen as structural and chemical changes in porcelain are observed above this range [19].



[Table/Fig-2]: Scanning electron microscope image of samples fired at 850°C.



[Table/Fig-3]: Scanning electron microscope image of samples fired at 900°C.



[Table/Fig-4]: Scanning electron microscope image of samples fired at 960°C.

SEM and EDS (Hitachi S-4700 Scanning Electron Microscope, Japan) were performed on all specimens, at the junction of metal alloy and ceramic [10]. The magnification of the SEM was set at 2000x. The data was graphically recorded and analysed.

Bonding of the ceramic layer to the noble metal alloy was evaluated by SBS test as per the ANSI/ADA specification no.38. The noble metal alloy-ceramic strips of the three firing temperature groups were mounted, one at a time on a jig and placed in a universal testing machine (Instron, USA). Load was applied at the interface of metal alloy and porcelain at a cross head speed of 0.5 mm/min to separate the two layers [10].

STATISTICAL ANALYSIS

The values were recorded and statistically analysed with Statistical Package for the Social Sciences (SPSS) version 24.0, using one-way Analysis of Variance (ANOVA) and post-hoc Tukey HSD test.

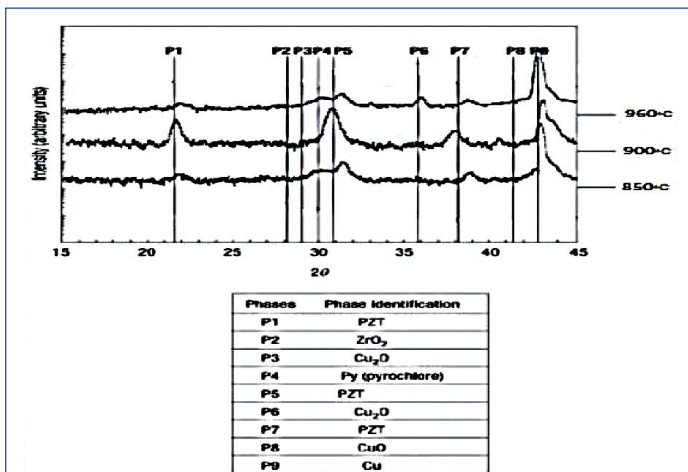
RESULTS

Scanning electron microscope: The scanning electron microscope photomicrographs were produced using a focused beam of electrons at a magnification of approximately 2 nm, after completing sample preparation and alignment, to obtain high quality images of the topography of ceramic noble metal alloy interface. Irregularities/ Coarseness (50 μm) was seen on the semi-precious alloy surface adjacent to ceramic layer at ceramic-noble metal alloy interface [Table/Fig-2-4]. These irregularities were probably produced during sandblasting of the alloy strips. However, some changes were seen with change in temperature.

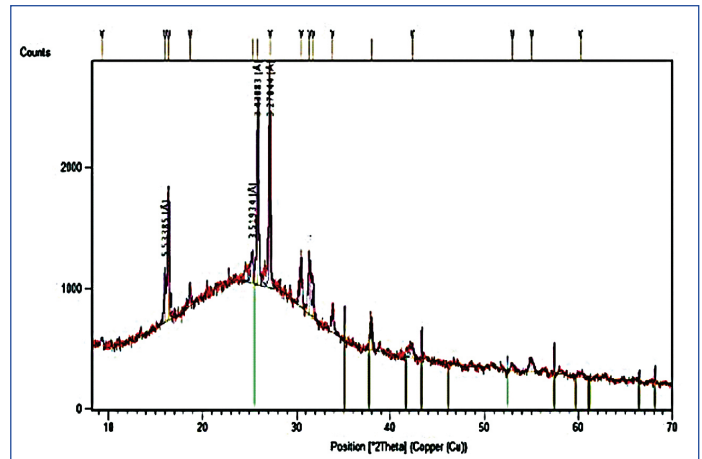
- At 800°C the interface was extremely corroded and rough with minimal porosities seen in the alloy half.
- At 900°C the interface had maximum contact between the two halves and more porosities were seen in the alloy half.
- At 960°C the porcelain metal interface was slightly more diffuse and porosities seen were equivalent to the 900°C section in the alloy half. It was hence evident that the interface with maximum integrity was seen at 900°C.

The higher numbers of porosities at elevated temperature may contribute to better mechanical interlocking between the ceramic metal interface.

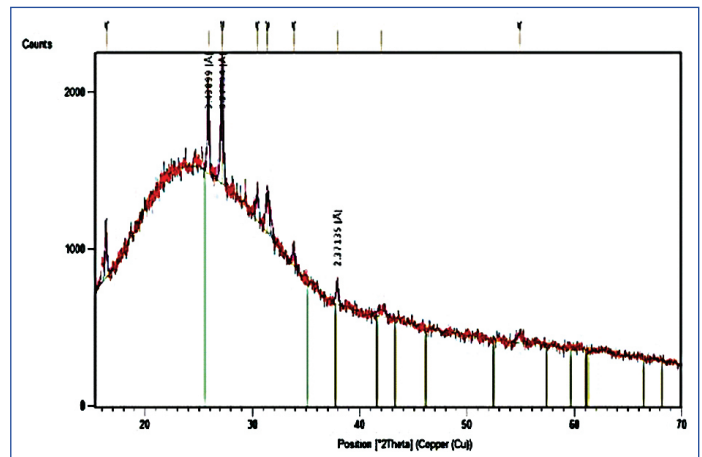
Energy diffraction spectroscopy: Energy diffraction spectroscopy revealed a wide interface between the ceramic layer and semiprecious alloy with nine peaks on the EDS graph (starting with peak P1 on the ceramic side to P9 on the noble metal alloy side). Inter-metallic compounds were formed at P1, P5 and P7, and a complete oxygen depletion zone was seen at P9 region. Further, EDS showed presence of intermetallic compound, lead zirconate titanate (PZT)(Pb[(Zr(x)Ti(1-x) O3) and a zone of partial oxygen depletion at the peak P1, whereas the interfacial complete oxygen depletion zone was formed at peak P9. [Table/Fig-5] shows the various peaks obtained at different firing temperatures (demarcated as peaks 1-9), with interpretation for ease of comprehension [Table/Fig-5-8].



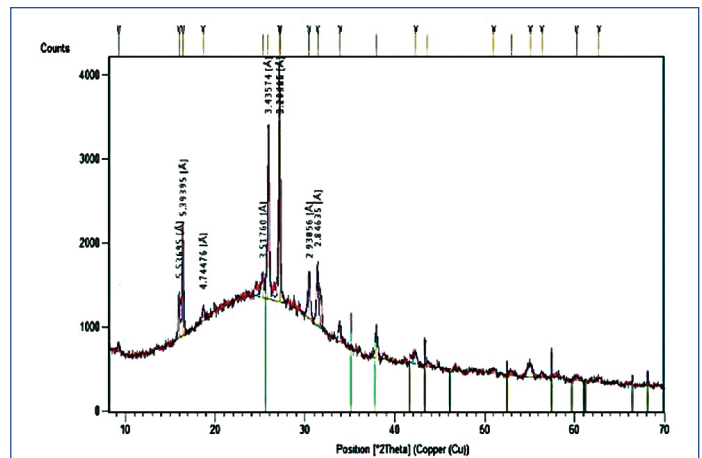
[Table/Fig-5]: EDS image of samples at 850°C firing temperature.



[Table/Fig-6]: EDS image of samples at 900°C firing temperature.



[Table/Fig-7]: EDS image of samples at 960°C firing temperature.



[Table/Fig-8]: EDS image showing various peaks obtained at different firing temperatures.

The SBS values of porcelain to semi-precious alloy at firing temperatures of 850°C, 900°C and 960°C was found to be 16.31 MPa, 24.33 MPa and 19.41 MPa respectively [Table/Fig-9].

Temperature	Shear bond strength	F value	p-value (ANOVA)
850°C	16.31±0.78	2385.00	<0.05
900°C	24.33±0.74		
960°C	19.41±0.89		
Comparison between temperatures (°C)		Shear bond strength of porcelain (Post-hoc tukey test)	
850 vs. 900		p-value <0.05	
850 vs. 960			
900 vs. 960			

[Table/Fig-9]: Interface Shear Bond Strength (SBS) in MPa. (Mean±SD, n=75) at different firing temperatures.

*using one-way ANOVA and post-hoc Tukey HSD test

There was statistically significant effect of temperature on SBS ($F=2385.00$, p -value <0.05). Further, post-hoc Tukey test showed significantly (p -value <0.05) higher SBS at 900°C , compared to both 850°C and 960°C .

Therefore, maximum SBS was seen at 900°C (of the three firing temperatures used in this experiment). It was also observed that the ceramic layer never completely separated but fractured under shear load, leaving a thin layer adhered to the alloy surface. PZT compounds are piezoelectric intermetallic compounds which are significantly insoluble and exhibits quenched disorder in the B-cation arrangement, creating a “disorder gap” between small, density-functional theory (DFT)-accessible supercells and the structural molecular framework. The long-range coulomb interactions of the charged ions in the system enable long-range structural correlations.

DISCUSSION

Success of metal-ceramic restorations depends mainly on the bonding between the metal alloy and the porcelain layer. This bond should remain stable under various mechanical and chemical stresses. Scanning electron microscopy and energy diffraction X-ray spectroscopy studies have provided new insight into the bonding mechanisms between metal alloys and ceramic. Elemental level transformations induced by heat energy at the base metal alloy/porcelain interface have been studied earlier [10]. Metal-ceramic interface region was seen by SEM images which clearly showed the changes taking place there. Roughness was noted at the alloy surface which enhanced the wetting of the metal surface by the porcelain. The porcelain mass interlocked itself into these irregularities.

Extensive studies performed by Bomr MP and Pask JA proved that the porcelain dissolved from the interface region leaving behind a layer of metal oxide saturated porcelain in thermodynamic equilibrium [12,14,21]. This metal oxide layer in turn was in thermodynamic equilibrium with the metal alloy thus forming a continuous electronic structure. In this study, it was found that the metal oxide layer was formed just adjacent to the middle part of the interface region. Intermingled zones of oxygen depletion were formed in the interface region at all three firing temperatures, which probably initiated the formation of inter-metallic compounds rather than oxides as was evident from the EDS graphs.

Metal oxides, chemically, have a terminal oxygen atom which can be easily removed whereas in case of intermetallic compounds, more energy is required to break the bond between the two. Intermetallic bond is many times stronger than van der Waal's forces due to their long branched microstructure [22]. The ceramic-noble metal alloy interface with nine different peak regions was also seen to be much wider compared to the ceramic-base metal alloy interface with three different regions [10].

The lead, zirconium and titanium ions from the metal alloy come in contact with oxygen (from the ceramic) when porcelain is fired at an increasing temperature gradient in the firing chamber. These metal ions form an intermetallic compound known as Lead zirconate titanate (PZT) in the presence of oxygen [10]. In case of noble metal alloys this compound was formed away from mid interface zone between porcelain and noble metal alloy (peak 1).

The PZT compounds are piezoelectric intermetallic compounds which are significantly insoluble and exhibits quenched disorder in the B-cation arrangement, creating a “disorder gap” between small, density-functional theory (DFT)-accessible supercells and the structural molecular framework. The long-range coulomb interactions of the charged ions in the system enable long-range structural correlations. A variety of Zr/Ti arrangements and composition have been researched upon to bridge the “disorder gap” [23]. However, complete elimination of this gap was not possible. The other regions of the ceramic-alloy interface (peaks 2-8) were found to have metallic oxides, whereas the region around

peak 9 (adjacent to semiprecious alloy) had an oxygen depletion zone, which led to the presence of unreacted copper ions.

As per the EDS graph of the noble metal alloys and dental porcelain, PZT inter-metallic compound was formed at P1, P5 and P7 of interfacial zone. Whereas, in the EDS graph of base-metal alloy and dental porcelain, intermetallic compounds (Ni_3Al and CrSi_2) were formed right at the interface (Region II) [10]. Ni_3Al and CrSi_2 are true intermetallic compounds with higher intermetallic bond strength whereas PZT compounds contain oxygen and are piezoelectric in nature. The oxygen depletion zone was not found at the mid-interface of noble metal alloy and dental porcelain, instead it was found at P9 region (which was away from the mid-interface) and was different from an earlier study between base metal alloy/porcelain in which it was formed at the mid-interface [10]. In the earlier study also, it was shown that at 900°C fewer oxides were released, hence a more homogenous mass is formed leading to better strength of porcelain [18,19]. Due to this pattern, the interfacial bonding of ceramic and the noble metal alloy was greatly reduced compared to the pattern of formation of oxygen depletion zone and nature of intermetallic compounds formed in case of nickel-chromium alloys [10]. This was evident from the interpretation of the intensity peaks of all the three groups (firing temperature) obtained on the energy dispersion graph. The current study proved the existence of the interfacial diffusion zone through SEM and the composition of this zone through EDS. On analysing the interpretation of the experiments, it could be stated that intermetallic compounds were formed away from the junction of the porcelain and metal layer, predominantly an interaction of the metal ions and oxygen.

The SBS values further substantiated the results interpreted from the EDS graphs. A rough estimate of the relative weight percentages of the elements could be interpreted depending on the intensity of the peaks. Higher the peak more would be the relative weight percentage of the element [23,24]. Presence of oxygen in the firing chamber would annul the formation of metallic compounds at the interface and lead to failure of the PFM restoration [10].

The SBS testing showed that the selected ceramic-noble alloy bonding (16.31 MPa, 24.33 MPa and 19.41 MPa at 850°C , 900°C and 960°C , respectively) was found to be weaker than ceramic-base metal alloy (20.41 MPa, 35.19 MPa and 30.35 MPa at 700°C , 900°C and 960°C respectively) tested earlier [10]. The average SBS was maximum at 900°C followed by 960°C and 850°C . The specimens fired at 850°C displayed weak porcelain-metal bonding because the porcelain particles did not form a homogenous mass at 850°C , hence this group had the least SBS values. At 960°C , the components of porcelain probably started disintegrating, leading to decreased SBS [25-27]. The SBS values could be correlated with the EDS graphs obtained. The lack of formation of intermetallic compounds in the interface region justified the decreased SBS of the specimens of that group and nature of fracture which took place within the porcelain mass instead of forming right at the interface. At 900°C and 960°C , the EDS graph trends were almost similar but a slight difference was observed in SBS values [10,19].

Previous studies have shown that when fired at 960°C the components of the porcelain mass became over desiccated and friable, which explains the slight decrease in the SBS of specimens fired at 960°C , compared to the specimens fired at 900°C [10,19]. It has been observed that porcelain firing did not cause chemical impurities formation on the surface of Cobalt-chromium alloy and sintered metal exhibit significantly higher corrosion resistance than the casted one due to its homogeneity of structure and chemical composition. Increased thickness and the heterogeneity of the oxide layers on alloy surface might be responsible for decrease in corrosion resistance [28]. A comparison of the findings from present and previous studies from the last 10 years have been made [Table/Fig-10] [1,10,19,28].

Author's name and year	Place of study	Sample size	Materials used	Parameters compared	Conclusion
Present study (2021)	Lucknow, India	75	Porcelain-noble-metal alloys	Shear bond strength at different firing temperatures (850°C, 900°C, 960°C)	The mean value of shear bond strength was 16.31 MPa at 850°C, 24.33 MPa at 900°C and 19.41 MPa at 960°C. Irregularities/Coarseness (50 µm) was seen on the semi-precious alloy surface adjacent to ceramic layer at ceramic-noble metal alloy interface in SEM images. EDS showed presence of intermetallic compound, lead zirconate titanate (PZT) (Pb[(Zr(x)Ti(1-x)O ₃) and a zone of partial oxygen depletion at the peak P1, whereas the interfacial complete oxygen depletion zone was formed at peak P9.
Rylska D et al., 2021 [28]	Poland	Two cobalt-chromium alloys, Soft Metal LHK (milling in soft material and sintering) and MoguCera C	Cobalt-chromium alloys	Corrosion resistance	The sintered metal exhibited significantly higher corrosion resistance than the casted one due to its homogeneity of structure and chemical composition. The sintered metal seems to be a favourable framework material for porcelain fused to metal crowns.
Tripathi A et al., 2018 [10]	Lucknow, India	75	Porcelain fused to Nickel-chromium alloy	Shear bond strength at different firing temperatures (700°C, 900°C, 960°C)	Mean shear bond strength was 20.41 MPa at 700°C, 35.19 MPa at 900°C 30.35 MPa at 960°C. In SEM images porcelain and metal alloy interface showed roughness of the metal alloy surface adjacent to the ceramic layer. An inter-metallic compound and oxygen depletion zone was seen by EDS.
Tripathi A et al., 2016 [19]	Lucknow, India	40 specimens in 6 groups	Porcelain	Compressive strength and abrasion resistance at different firing temperatures (660°C, 760°C, 860°C, 900°C, 960°C, 990°C)	The specimens fired at 900°C exhibited superior strength and abrasion resistance. SEM analysis depicted a homogeneous mass of dental porcelain, implying that firing was complete at 900°C.
Teng J et al., 2012 [1]	China	30	Zirconia core specimen (Test group) and Metal-ceramic (control group)	Shear bond strength of a zirconia core ceramic. Three different surface conditioning methods were evaluated: polishing with up to 1200 grit silicon carbide paper under water cooling, airborne-particle abrasion with 110 µm alumina particles, and modification with zirconia powder coating before sintering	The mean shear bond strength values in MPa were 47.02 (6.4) for modified zirconia, 36.66 (8.6) for polished zirconia, 39.14 (6.5) for airborne-particle-abraded zirconia, and 46.12 (7.1) for the metal ceramic group (control group).

[Table/Fig-10]: Comparison of studies from last 10 years with the present study [1, 10, 19, 28].

Limitation(s)

The limitation of this study lay in difficulty in generalisation of the study findings to all noble alloy systems.

CONCLUSION(S)

During veneering of ceramic over noble metal alloy heat treatment leads to formation of intermetallic compound, lead zirconate titanate which is probably the prime factor in bonding of ceramic to noble metal alloy. The mean SBS showed an upward trend till 900°C and decreased thereafter. The bonding between porcelain-noble metal alloys was weaker as compared to porcelain-base metal alloys.

Future studies could focus on the estimation of number of voids created during the firing cycle and evaluation of corrosion resistance of metals at different firing temperatures which could also be a possible reason for metal-ceramic debonding. Attempts can be made to generate higher metal-ceramic bond strength to avoid prosthesis failure. A detailed study can be conducted to evaluate the composition of oxide layer and its bond strength. This study also opens future avenues for evaluating bond strength using other techniques as well and generating a material of desired bond strength.

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