Characterization of the Interface of Heat-pressed Glass–Ceramic Masses on Metal Support Cr–Co in Metal–Ceramic Prosthetic Restorations

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Abstract

Aim and objective: The aim and objective of this study is to evaluate the interface between heat-pressed glass–ceramic masses on a Cr–Co metal substrate using a scanning electron microscope and an X-ray energy dispersion spectrometer.

Materials and methods: A pressed porcelain–leucite-based ceramic (IPS InLine press-on-metal (PoM); Ivoclar Vivadent AG) was used. Three cylindrical metal specimens cast (diameter 5 mm, height 1.5 mm) in Co–Cr alloy and covered with pressed ceramic (height 1.5 mm), according to the instructions of the manufacturer. All the specimens were covered with conductive carbon and then examined with a scanning electron microscope. The interface areas were studied using projections from an ETD secondary emission detector and a reversing atomic SSD contrast beam at a magnification of 1200× and 2000×, with a voltage 25 kV acceleration and 110 mA climb current. The elemental analysis was done with genesis 3.5 software, without the use of templates. Surface mapping areas and linear line scan projections of elemental distributions during the interface were recorded.

Results: The distribution of specific elements in the ceramic coating concludes the existence of ion diffusion from one side of the interface to the other, which leads to an initial conclusion of the development of primary bonds with oxygen bridges. Also, in the interface, there are ledges of the mass of opaquer on the metal substrate, which results in the creation of a mechanical bond. Therefore, the adhesive mechanism must be due to both micromechanical retention and wetting phenomena and is similar to the conventional layering technique.

Conclusion: The PoM technique can be used as an alternative fabrication method for metal–ceramic restorations. Factors, such as material composition and properties, firing temperatures, cooling rates, operator's skill, porosities, and fabrication process, may affect the quality and strength of the bond between the core and the veneering materials.

Clinical significance: The PoM technique can be used as an alternative fabrication method for metal–ceramic restorations.

Keywords: Ceramic pressed to metal, Dental ceramics, Metal–ceramic restorations, PoM technique, Scanning microscopy.

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Introduction

Conventional porcelain fused to metal restorations are used extensively in dental prosthetics.[1,2] Even with recent advances in all-ceramic systems, metal–ceramic restorations continue to be common in fixed prosthodontics, because of their high mechanical strength and predictability.[1,3,4]

Metal alloys have been extensively used as cores of metal restorations ranging from single crowns to long-span fixed dental prostheses.[5,6] Traditionally, veneering ceramics are layered on metal core material to establish an optimum esthetic result. With this layering technique, porcelain powder is mixed with modeling liquid and the mixture is layered on the metal surface using a brush. The layer is usually overbuilt to compensate for consideration and firing shrinkage. This layering technique requires skill and multiple firings and applications.

An alternative technique is to press veneering ceramics to the core material, and a process for pressing ceramics to a metal core with the lost-wax technique and glass–ceramic ingots has been recently developed.[7-10] The commercial press-on-metal (PoM) technique, developed by Ivoclar Vivadent for dental laboratories, uses special equipment for hot pressing the remainder of the porcelain onto the alloy after the initial oxidation step and sintering of opaque porcelain.[11,12] With this pressing technique, first, a metal substructure is waxed and cast. After the casting has been opaqued, a complete contour anatomical waxing is performed on the casting, and subsequently, a sprue is attached to the wax, and the wax-core complex is invested. The wax is eliminated in an oven and ceramics ingots are heat-pressed into the mold and to the core. Thereby reproducing the anatomy created in the wax.

The PoM technique was developed to optimize working procedures and to increase the productivity and efficiency of dental laboratories.[11,13] It also exhibits many advantages in terms of

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esthetics, marginal fit, and intaglio accuracy.[11,15] More specifically, the firing shrinkage experienced with the layering technique is minimized, resulting in a better fit of the porcelains margins to the abutments.[7,8,15,16] Distortion of the metal may be reduced during veneering because of support from the investment.[7,17] Marginal adaptation was reported to be improved with the PoM technique with the metal–porcelain margin compared to conventional metal–ceramic restorations.[7,8,15,16-18]

The presence of a strong bond between porcelain and metal substructure determines the success of the metal–ceramic restoration. Many studies have been carried out on the bond strength of pressed ceramic to metal. Most of these studies concluded that the PoM technique is an acceptable alternative fabrication method for metal–ceramic restorations.[19]

According to the author's knowledge, there was no published research regarding the interface between the pressed ceramic layer and metal substructure. So, the aim of this study is to evaluate the interface between heat-pressed glass–ceramic masses on a Cr–Co metal substrate and the formation of the intermediate bonding layer.

The working hypothesis is that the phenomena that take place between metal and ceramic masses in the metal–ceramic bond do not occur between metal–heat-pressed glass–ceramic coating. The question should also be answered as to whether ionic diffusion phenomena develop during the interface similar to those of the metal–ceramic bond.

Material and Methods

Part of this research was carried out in the Department of Biomaterials, School of Dentistry, National and Kapodistrian University of Athens.

Pressed porcelain–leucite-based ceramic (IPS InLine PoM; Ivoclar Vivadent AG) was used. The PoM ingot was composed of a leucite-containing ceramic, the optical properties of which were optimized by small shares of further crystal phases. The chemical composition provided by the manufacturer is listed in Table 1.

Also, a Co–Cr alloy (IPS d.SIGN 20 Ivoclar Vivadent) for metal–ceramic restorations was selected. The chemical composition provided by the manufacturer is listed in Table 2.

Three cylindrical metal specimens of the above alloy cast using acrylic resin patterns (Duralay; Reliance Dental Co). The dimension of each specimen was 5 mm in diameter and 1.5 mm in height. All the casting procedures were carried out in accordance with the manufacturer's instructions and performed by a single operator. After the casting process, the specimens were bench cooled and carbide discs were used at low speed to remove sprues. Then they were cleaned by using a steam cleaner device and were dried at room temperature. Finally, the oxidation process was performed according to the instructions of the manufacturer. Proceeding to the process of veneering ceramic, the first and second firings were performed for each metal specimen by applying the respective opaque following the manufacturer's instruction.

Table 1: Ceramic composition of IPS InLine PoM ingot *(wt%)* according to the manufacturer[20]

SiO_2	50.0–65.0
Al_2O_3	8.0–20.0
Na_2O	4.0–12.0
K_2O	7.0–13.0
Other oxides, fluoride	0.0–6.0
Pigments	0.0–3.0

Wax patterns with the height of 1.5 mm were fabricated on the opaque surfaces of the metal specimens. The sprues with a diameter of 3 mm were attached to the top of the wax patterns and then the specimens were invested within the IPS Investment Ring using the IPS PressVEST Premium according to the manufacturer's instructions. Then, the lost-wax technique was performed and ceramic ingots (Ivoclar, Vivadent, Liechtenstein) were pressed into the mold in the furnace (Programat 3000, Ivoclar, Vivadent, Liechtenstein). After separation of the sprues and smoothing of the attachment points, the specimens were carefully sandblasted and cleaned with steam. Finally, they were dried thoroughly with oil-free air. The final test specimens had the dimensions shown in Figure 1.

Table 2: Alloy properties and composition *(wt%)* according to the manufacturer[20]

Composition (%)	*Co 60.2|Cr 30.1|Ga 3.9|Nb 3.2|Mo <1.0, Si <1.0, B <1.0, Fe <1.0, Al <1.0, Li <1.0*
ISO	22674:2016
ADA class	Base metal
Type/color	5/white
Density (g/cm^3)	7.8
Melting range (solidus/liquidus)	1145–1180°C
Elastic modulus (GPa)	234
Recommended metal ceramic	IPS Style, IPS InLine One, IPS InLine, IPS InLine PoM, IPS Classic, IPS d.SIGN

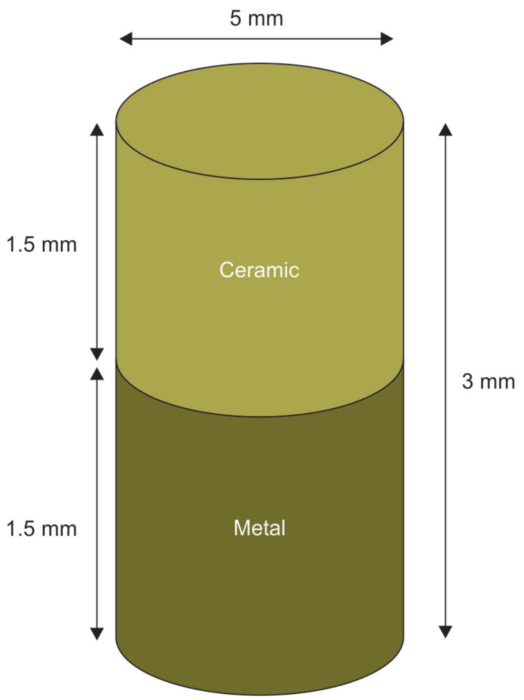


Fig. 1: Schematic illustration of the test specimen and respective dimensions

After their manufacture, the specimens were boxed in epoxy resin in such a way that the interface metal core-ceramic coating was toward the free surface.

The surfaces were smoothed with Si–C silicon–carbide discs with a grain size of 220–2200 grit with simultaneous water sprinkler to cool the material and then polished with 3.1 and 0.5 µm diamond in a metal grinding and polishing device. Finally, the specimens were cleaned in an ultrasonic bath with distilled water for 5 minutes, washed with water and ethyl alcohol solution, and allowed to dry in atmospheric conditions.

For the study under the electron microscope, all the specimens were covered with conductive carbon and then examined with a scanning electron microscope (Quanta 200,FEI, Hillsboro, Oregon, United States) which was connected to an X-ray energy dispersion spectrometer (EDS-Phoenix CDU, EdaxInt, Mawlaw, New Jersey, United States) equipped with ultra-thin Berylli window.

The interface areas were studied using projections from an Everhart-Thornley detector (ETD) secondary emission detector and a reversing atomic surface to surface distance (SSD) contrast beam at a magnification of 1200× and 2000×, with a voltage 25 kV acceleration and 110 mA climb current.

The elemental analysis was done with genesis 3.5 software, without the use of templates. Surface mapping areas and linear line scan projections of elemental distributions during the interface were recorded.

Results

The results of the elemental analysis of the metal-core and ceramic coating are summarized in Tables 3 and 4.

Figure 2 illustrates cross-sectional SEM image of the specimen. Energy dispersion spectrometer elemental mapping of this region is shown in Figures 3 and 4. On the interface, there are ledges of the mass of opaquer on the metal substrate. There are areas with a small contrast of atomic number (darker) within the mass of opaquer, which may correspond to grains of different phases, and areas that have a high contrast of atomic number (brighter) corresponding to the phase that meets the spaces between the grains. There is also a scattered phase that may correspond to glass (glass phase).

The profile of chemical composition (Fig. 5) shows that the intermediate layer is well defined. There is a sharp drop in the content of Co within the mass of the coating. This drop occurs directly, in the first 2–3 µm of the ceramic coating. Similarly, the content of Cr is reduced, although this is done at a greater depth within the mass of the coating, to an area of approximately 5 µm. In dark areas characterized as grains, an increased percentage of Si is observed scattered at a different distance from the interface, as there are three different peaks. In the lighter areas, there is an increase in the content of Zr.

Table 3: Elemental analysis and molecular composition of the metal core

			C norm.	*C atom.*	*C error*	*(1 sigma)*
El	*AN*	*Seriesunn.*	*[wt%]*	*[wt%]*	*[at%]*	*[wt%]*
Si	14	K-series	0.57	0.63	1.29	0.06
Cr	24	K-series	25.74	28.10	31.24	0.71
Fe	26	K-series	0.21	0.23	0.24	0.04
Co	27	K-series	55.79	60.90	59.75	1.45
Ni	28	K-series	3.28	3.58	3.53	0.12
Mo	42	L-series	6.01	6.56	3.95	0.25
Total			91.61	100.00	100.00	

Table 4: Elemental analysis and molecular composition of the ceramic coating (opaque)

			C norm.	*C atom.*	*C error*	*(1 sigma)*
El	*AN*	*Seriesunn.*	*[wt%]*	*[wt%]*	*[at%]*	*[wt%]*
O	8	K-series	31.20	36.77	60.92	4.27
Na	11	K-series	3.01	3.55	4.09	0.24
Al	13	K-series	5.10	6.02	5.91	0.28
Si	14	K-series	12.54	14.78	13.95	0.58
K	19	K-series	6.14	7.23	4.91	0.22
Ca	20	K-series	1.41	1.66	1.10	0.08
Cr	24	K-series	0.56	0.66	0.33	0.05
Fe	26	K-series	0.87	1.03	0.49	0.06
Zn	30	K-series	1.38	1.63	0.66	0.08
Zr	40	L-series	21.62	25.48	7.40	0.87
Ba	56	L-series	1.02	1.20	0.23	0.06
Total			84.85	100.00	100.00	

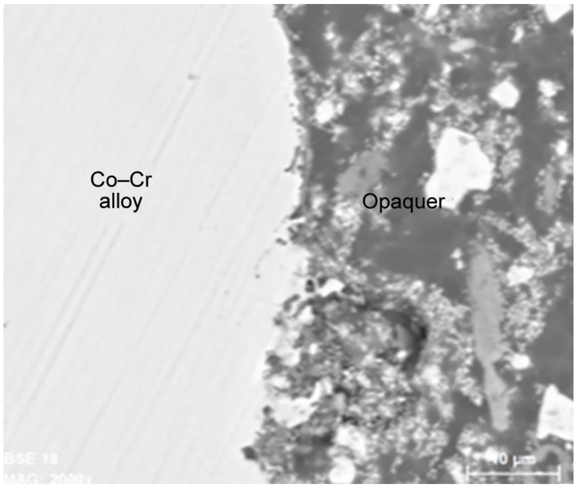
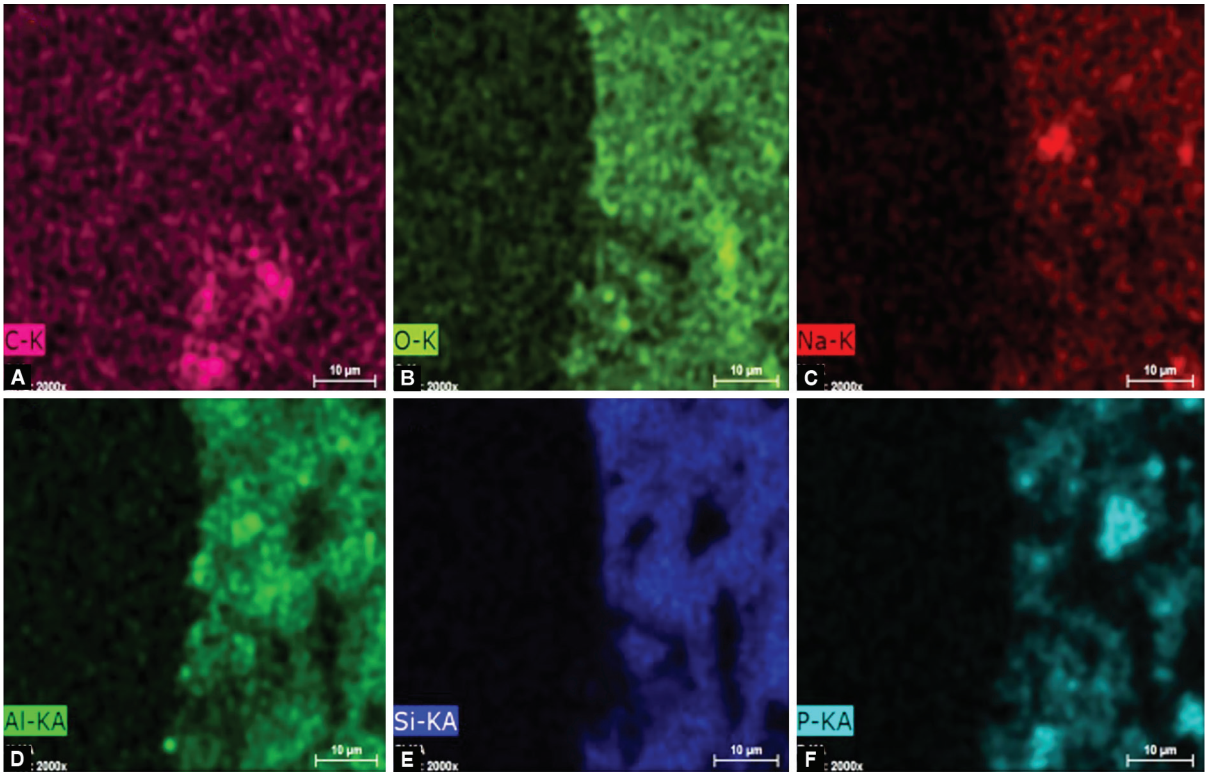


Fig. 2: SEM (Backscattered-electron (BSE) mode) micrograph of the specimen

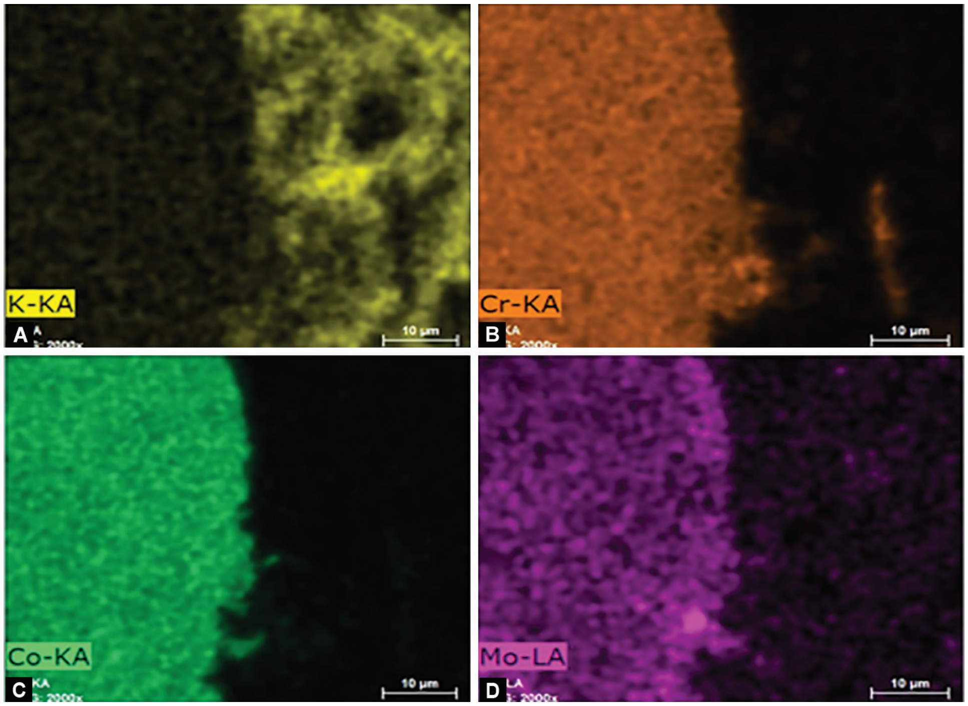
The distribution of the above elements in the ceramic coating shows the existence of ion diffusion from one side of the interface to the other, which leads to an initial conclusion of the development of primary bonds with oxygen bridges. Therefore, the adhesive mechanism must be due to both micromechanical retention and wetting phenomena and is similar to the conventional layering technique.

Discussion

Metal–ceramic restorations have been used for several decades by clinicians to provide esthetic and masticatory function, as they combine esthetics with superior mechanical properties. However, these restorations have the potential for fracture of the ceramic veneer, which poses serious cosmetic and clinical



Figs 3A to F: Energy dispersion spectrometer elemental maps for various elements: (A) C, (B) O, (C) Na, (D) Al, (E) Si, (F) P



Figs 4A to D: Energy dispersion spectrometer elemental maps for various elements: (A) K, (B) Cr, (C) Co, (D) Mo

problems. The causes of such fractures are varied. Some of these are impact and fatigue load, occlusal forces, incompatible thermal expansion coefficients between the ceramic and metal substructure, use of metal with low-elastic modulus, seating force during trial insertion or cementation, improper design, microdefects within the material, and trauma.[20-24]

Recently, a new generation of ceramics has been introduced for veneering metallic cores. It was developed following the idea

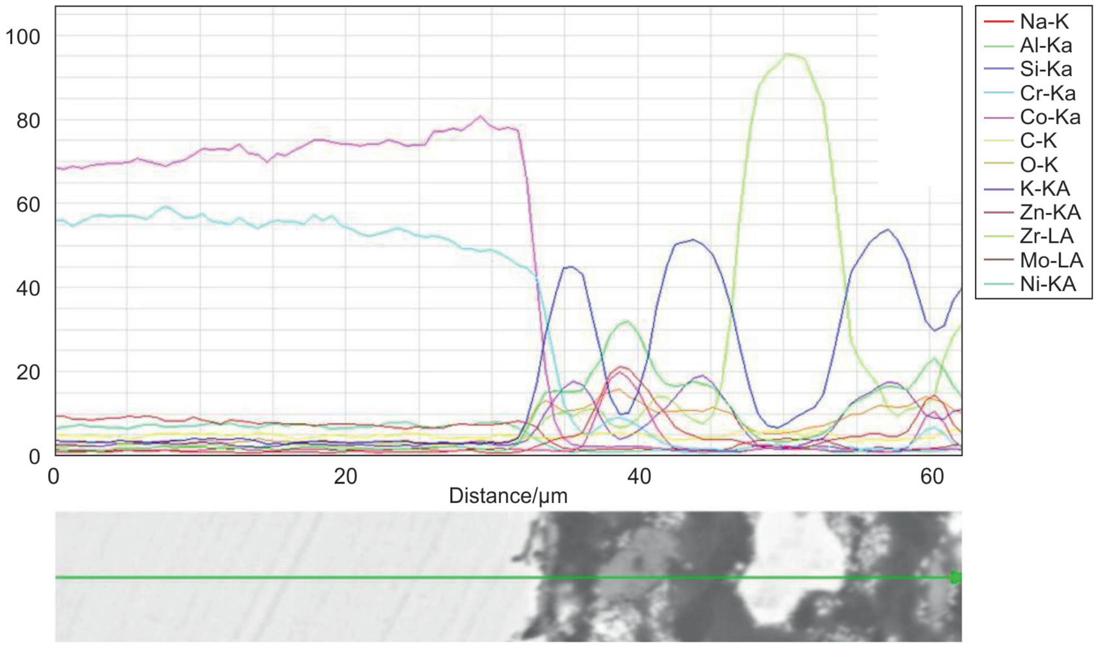


Fig. 5: Linear distribution of line scan elements on the core–overlay interface

of pressable all-ceramic systems. This research paper examined the characterization and qualitative analysis of the interface created during the thermocompression of glass–ceramic coating systems on metal substrates.

The evaluation of surface morphology and microstructure as revealed by the use of optical-electronic microscopy and microanalysis of X-rays constitutes a reliable method of studying and characterizing surfaces and interfaces. From the above-mentioned control techniques, additional information is provided on the topography of the area as well as the elemental and molecular composition of the materials and is an important source of information for the qualitative and quantitative assessments of the area of the interface.[4,9-11]

Electronic scanning microscopy significantly upgraded the ability to study the microstructure of ceramic materials, although the absence of significant differences in the mean atomic weights of the elements that make up both the glass and crystalline phases in some of them, makes it particularly difficult to distinguish the phases clearly and clearly recognize of the boundaries between them.[9-11]

Microanalysis of X-rays is also a valuable technique for the study of microdistributions of different phases and has been used in the analysis of interphases, e.g., porcelain–metal and bone–implant. But even such a specialized technique is sometimes not sensitive enough to detect small chemical variations that exist between the largely similar neighboring structures. The choice of visual medium-resolution is particularly crucial because the use of high magnifications in surface scanning analyses offers a clearer and more detailed distribution of the different phases but is involved by the surface characteristics and morphology of the interface. For this reason, the assessment of ion penetration should be carried out in a fixed scanning area and in addition to the images of the digital reconstruction of the emission spectrums.[11,25]

A metal–ceramic restoration is a material composition that is based on a durable bond between an alloy material and a ceramic material. The interface between metal and ceramic is formed by the opaquer, which, if carefully applied and fired, produces a sound bond between the two materials. Good wettability of the alloy surface by the opaquer, which is very viscous at high temperatures, is a prerequisite for an optimal bond.[24, 25]

In general, the area of the interface brings together the conditions for the coexistence of a multitude of imperfections since it consists of the contact and the union of two different basic materials. The difference in thermal expansion factors and the modulus of elasticity of the two phases (core-coating) leads to the development and accumulation of stresses along with the interface, significantly affecting the strength of restoration. In addition, the thickness ratio and geometry of the core-coating materials shape the quality of their interface, affect the possible development of a bond between them and modify the clinical behavior of restoration accordingly.[25,26]

It is well known that the most important of the numerous adhesive mechanisms for a durable and strong metal–ceramic bond are (a) adhesion by mechanical bond, (b) adhesion by chemical bond, and (c) adhesion by intermolecular forces (van der Waals forces).[10,11,19]

According to the results of this research study, we can assume that the above three adhesive mechanisms are present between the metal-core and pressable ceramic.

The study of SEM images of the specimen has shown that in the interface, there are projections of the mass of opaquer on the metal substrate, which results in the creation of a mechanical bond.

The ceramic bonds mechanically to the metal surface by filling depressions and/or enclosing protruding structures and anchor points, which are present on the surface after metal conditioning. In addition to this mechanical bond, the ceramic demonstrates a certain compressive strain, since its coefficient of thermal expansion is lower than that of the alloy. The type of metal tested in this study was considered thermally compatible with the pressable ceramic, as it is recommended for the IPS InLine PoM ceramic, according to the manufacturer (Table 2).[19]

Analysis of the results has shown that ion diffusion phenomena are observed on the metal–heat-pressured ceramic coating surface and a Si-rich transition zone, which probably plays a crucial role in

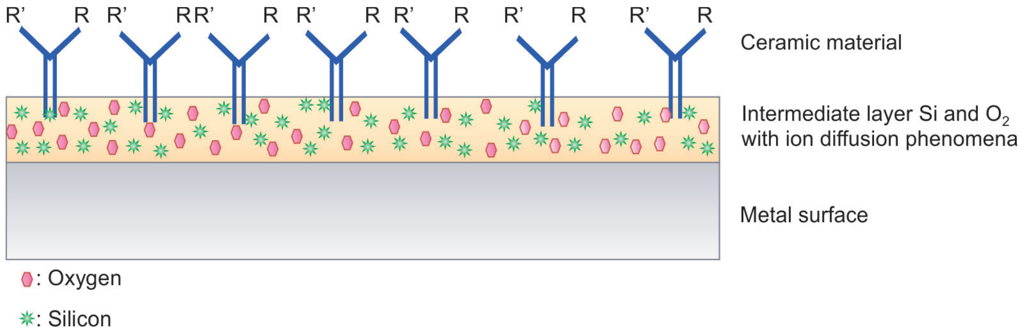


Fig. 6: Model of the chemical bond

the creation of a primary chemical bond with O_2 bridges between the two materials (Fig. 6).

The chemical bond is initiated by oxygen atoms which were present in both the metal surface and the ceramic and thus link the two materials. The IPS InLine PoM ingots are made of a glass–ceramic material containing leucite and based on synthetic glass raw materials, which contain small quantities of opalescent glass–ceramic and translucent components. Both in their pressed and unpressed conditions, the ingots demonstrate anisotropic structure, which is responsible for their homogeneous distribution of the leucite crystals and the high strength.[2,14,19,26]

Of course, a variety of factors, such as material composition and properties, firing temperatures, cooling rates, operator's skill, porosities, and fabrication process, may affect the quality and strength of the bond between the core and the veneering materials.

Laboratory observations confirmed that the PoM technique requires much less time than the conventional layering technique for preparing dental porcelain. However, the PoM technique has some limitations that are emphasized by the manufacturer: the technique is contraindicated for alloys that contain more than 10% silver (Ag), and the pressing should be done on dental alloys in the coefficient of thermal expansion (CTE) range of $13.8–14.5 \times 10^{-6}$/K (25–500°C).[19]

The clinical success of the PoM technique has been documented in numerous *in vivo* and *in vitro* studies.

Khmaj et al.[27] evaluate the bond strength of four categories of noble alloys using two techniques of porcelain application. They concluded that for both conventional layering and PoM techniques, all four noble alloys had a mean metal-to ceramic bond strength that substantially exceeded the 25 MPa minimum in the ISO Standard 9693.

Farzin et al.[2] compared the bond strength of ceramics that were pressed or layered to a Ni–Cr alloy and showed that it is possible to improve metal–porcelain bond strength significantly by applying an overpressure during porcelain firing. Similar results were found in many other researches.[18]

Fahmy and Salah[18]review a PoM ceramic vs a conventional veneering system regarding marginal gaps, fracture resistance, microhardness, and surface roughness. They showed superior marginal adaptation, decreased microhardness, and similar load to failure and roughness values of the PoM ceramic system. Moreover, considerable ease and speed of fabrication of this system were evident. Similar results were found in the research carried out by Schweitzer et al.,[1] Holden et al.,[28] and by Goldin et al.[29]

Also, Lee[30] describes the fabrication of metal–ceramic restoration with CAD-CAM technology using the PoM technique. He concluded that this technique has many advantages such as efficiency, simplicity, and less human error. Similar results were found in the research carried out by Bayramoglu et al.[31]

CONCLUSION

According the results of this study, the following could be concluded:

- The PoM technique can be used as an alternative fabrication method for metal–ceramic restorations.
- The adhesive mechanism that occurs must be due to both micromechanical retention and wetting phenomena and is similar to the conventional layering technique.
- Factors, such as material composition and properties, firing temperatures, cooling rates, operator's skill, porosities, and fabrication process, may affect the quality and strength of the bond between the core and the veneering materials.

CLINICAL SIGNIFICANCE

The PoM technique can be used as an alternative fabrication method for metal–ceramic restorations. It was developed following the idea of pressable all-ceramic systems.

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