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RESEARCH AND EDUCATION

Effects of the rare earth element lanthanum on the metal-ceramic bond strength of dental casting Co-Cr alloys

Yanan Zhou, BE,[a] Ning Li, PhD,[b] Hongmei Wang, DDS,[c] Jiazhen Yan, PhD,[d] Wenbo Liu, PhD,[e] and Sheng Xu, DDS[f]

ABSTRACT

Statement of problem. The metal-ceramic bond strength of dental casting Co-Cr alloys may be improved by the addition of the rare earth element lanthanum (La), but studies are lacking.

Purpose. The purpose of this in vitro study was to evaluate the effects of the rare earth element La on the metal-ceramic bond strength of dental casting Co-Cr alloys.

Material and methods. Four groups of specimens with different La content (0; 0.02 wt%; 0.04 wt%; 0.06 wt%) were prepared using conventional casting methods. The metal-ceramic bond strength was assessed by using the 3-point bend test. The microstructures were characterized by metallurgical microscopy, scanning electron microscopy, and X-ray diffraction analyses. The morphology and element distribution of metal-ceramic interfaces were evaluated by scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy. The results of bond strengths were statistically analyzed by the Tukey honest significant difference (HSD) test (α=.05).

Results. The specimens showed typical dendritic microstructures, few defects, and island-shaped intermetallic compounds rich in Mo and Cr, mainly consisting of α-Co phase of a face-centered cubic (FCC) structure and ε-Co phase of a close-packed hexagonal (HCP) structure. As the amount of La increased, the thickness of the native oxide layer and the diffusion layer at the interface increased, and the wettability between the oxide layer and the metal matrix improved. The mean ±standard deviation values of the metal-ceramic bond strengths were 28.11 ±4.53 MPa for group 0%, 33.13 ±5.65 MPa for group 0.02%, 37.48 ±7.86 MPa for group 0.04%, and 40.70 ±5.17 MPa for group 0.06%. The statistical analysis indicated that significant differences ($P<.05$) were observed among all groups tested, except for group 0.04% and 0.06% ($P>.05$). The debonded surfaces of Co-Cr specimens showed a mixed type of adhesive and cohesive fractures.

Conclusions. The microstructures, morphologies, and compositions of oxide films were influenced by the La content, and the La addition could significantly improve the metal-ceramic bond strength of dental casting Co-Cr alloys. (J Prosthet Dent 2019;121:848-57)

Metal-ceramic restorations have been extensively and successfully used because of their excellent mechanical properties and acceptable esthetics.[1-4] Current dental casting metal-ceramic alloys consist primarily of noble-metal alloys (Au-Pt) and base-metal alloys (Ni-Cr and Co-Cr).[5] The noble-metal alloys possess excellent corrosion resistance, high elongation, and good biocompatibility.[6,7] However, these alloys are expensive, and precious metal resources are rare.[8-11] Because of the low coefficient of linear expansion and adequate mechanical properties, Ni-Cr alloys are used as a substitute for noble-metal alloys, but the nickel ions (Ni^+) released may give rise to an allergic reaction and represent a carcinogenic risk.[12]

Dental Co-Cr alloys are regarded as the most suitable substitute for noble-metal alloys primarily due to their excellent mechanical properties, corrosion resistance, and biocompatibility. However, commonly available Co-Cr alloys exhibit a poor thermal matching with porcelain because of the martensite transformation that occurs during the porcelain firing process. This leads to a volume expansion and generates excessive residual stresses

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[a] Predoctoral student, School of Manufacturing Science and Engineering, Sichuan University, Chengdu, PR China.
[b] Professor, School of Manufacturing Science and Engineering, Sichuan University, Chengdu, PR China.
[c] Lecturer, Department of Stomatology, Guangxi University of Chinese Medicine, Nanning, PR China.
[d] Associate Professor, School of Manufacturing Science and Engineering, Sichuan University, Chengdu, PR China.
[e] Associate Professor, School of Manufacturing Science and Engineering, Sichuan University, Chengdu, PR China.
[f] Associate Professor, College of Stomatology, Guangxi Medical University, Nanning, PR China.

Clinical Implications

The rare earth element lanthanum can significantly improve the metal-ceramic bond strength of Co-Cr alloy, which may improve clinical performance.

that result in poor metal-ceramic bonding.[13] Consequently, porcelain fracture or debonding has been reported.[14] The bond strength between the metal and ceramic must be adequate to achieve the clinical longevity of metal-ceramic restorations.

Rare earth (RE) elements exhibit high chemical activity, and a small addition of RE elements can strongly alter the performance of an alloy.[15] RE elements play an important role in the material's metallurgy, including strengthening, melt purification, and grain refinement.[16,17] Theoretical research on the effects of RE elements on the properties of an alloy has been published. Pieraggi et al[18] reported that RE elements concentrate in the grain boundary due to their large radius. RE elements could play the role of pinning dislocations, hindering the outward diffusion of metal cations, and reducing the growth stress, thus increasing the thickness of the diffusion layer. Karaali et al[19] reported a double-layer structure in the oxide layer of Co-Cr porcelain alloy and that the outer layer was CoO and the inner layer was $CoCr_2O_4$ with spinel structure. However, information on the application of RE elements to improve the interface performance and metal-ceramic bond strength of dental casting alloys is lacking.

In this study, a small amount of the RE element lanthanum (La) was added to a Co-Cr dental casting alloy. La was selected because previous reports have experimentally determined that the addition of a small amount of La has no human toxic side effects.[20] Thus, the purpose of this in vitro study was to investigate the metal-ceramic bond strength and metallurgical and interfacial characterization of Co-Cr dental casting alloys with different La content and to determine whether this addition might provide a worthwhile approach to solving the problem of poor bond strength to Co-Cr alloys. The null hypothesis was that similar metal-ceramic bond strengths would be found among the groups with different La content.

MATERIAL AND METHODS

Commercially available dental casting Co-Cr alloy ingots (CW-CC; Keningda Co Ltd) with a nominal composition (in wt%) of Co (62.0), Cr (25.0), Mo (5.0), W (7.0), and trace amounts of Si and the ceramic (VMK 95; VITA Zahnfabrik. H. Rauter GmbH & Co KG) with a nominal composition (in wt%) of SiO_2 (52.0), Al_2O_3 (15.0), K_2O (10.0), Na_2O (6.0), and trace amounts of TiO_2, ZrO_2, and SnO_2 were provided by the manufacturers. Specimens of all groups had the same chemical composition, except for La (0; 0.02 wt%; 0.04 wt%; 0.06 wt%). La additions beyond these values will cause excessive oxidation, causing the surface to darken in color, and become a health hazard; therefore, the maximum addition of La in this study was 0.06 wt%. Ten specimens (25×3×0.5 mm) for each group were fabricated by the lost wax-casting method (stipulated by ISO 9693-1).[21] In the casting process, the wax patterns were fabricated using a spray wax machine (ProJet DP3000; 3D Systems) and then invested (Bellavest SH; BEGO GmbH & Co KG) and cast using the Co-Cr alloy ingots using a high-frequency centrifugal casting machine (JG-554TP; Jing-Gong Medical, Co, Ltd). Specimens with incorrect size or with visible pores or cracks were discarded.

Acceptable specimens were ground on a handheld grinding machine (Marathon-3; SAE YANG Co Ltd) to make the surface smooth, ultrasonically cleaned for 5 minutes in ethanol, and then air-dried to ensure the removal of residual cleaner. Before porcelain application, the specimens were degassed and oxidized in a vacuum

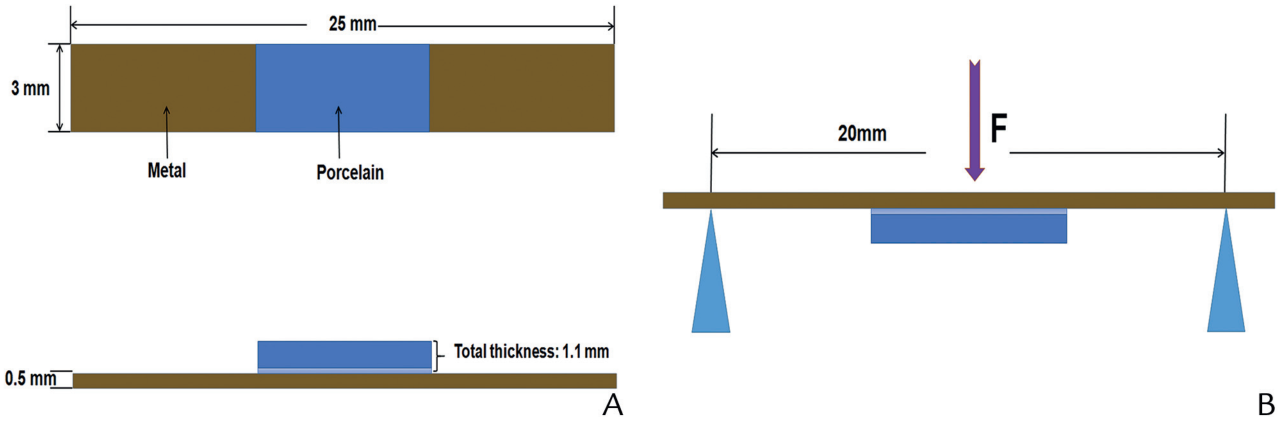


Figure 1. Schematics of porcelain specimens and 3-point bend test. A, Metal-ceramic specimen. B, 3-point bend test.

Table 1. Porcelain firing schedules

Program	Preheating Temperature, °C	Drying Time, min	Heating Rate (°C·min^{-1})	Firing Temperature, °C	Holding Time, min	Vacuum Degree, kPa
Degassing and oxidation	600	3	80	980	3	-95
Opaque	600	4	90	950	1	-95
Body porcelain	600	4	80	930	1	-95

Table 2. Metal-ceramic bond strengths of 4 groups (MPa)

Group	Number	Mean ±Standard Deviation	Minimum	Maximum
0%	6	28.11 ±4.53^{a}	25.01	32.90
0.02%	6	33.13 ±5.65^{b}	27.70	36.74
0.04%	6	37.48 ±7.86^{c}	33.63	44.33
0.06%	6	40.70 ±5.17^{c}	33.79	48.25

Groups with same superscript letter not statistically significantly different (P>.05).

porcelain furnace (JD; LIRRC Co, Ltd), and then airborne-particle abraded (1122; Xinou Co, Ltd) with 120-μm Al_2O_3 particles under an air pressure of 0.4 MPa for 30 seconds to reduce the oxide layer. For porcelain application, a thin layer of opaque porcelain and a layer of body porcelain were fired in succession to the central rectangular alloy area with the dimensions of 8.0×3 mm to achieve a total thickness of 1.1 mm (Fig. 1), and porcelain firing procedures were set in accordance with the recommendations of the manufacturer (Table 1).

The bond strengths of 6 specimens for each group were tested using a 3-point bend test on an electronic universal testing machine (RGM-4300; Reger Instruments Ltd) with a gauge length of 20 mm and a radius of curvature of the indenter of 1.0 mm according to the ISO 9693-1.[21] The specimens were placed on the apparatus with the ceramic positioned symmetrically opposite to a central load applied at a crosshead speed of 1.0 mm/min (Fig. 1). Metal-ceramic bond strengths were calculated from the expressions listed in ISO 9693-1.[21] Fracture types encountered in the 3-point bend test were assessed with optical microscopy.

Microstructural and interfacial characterizations were examined using a metallographic microscope (GX51; Olympus Co, Ltd), scanning electron microscopy (Inspect F; FEI) coupled with energy-dispersive X-ray spectroscopy (EDS), and X-ray diffractometer (XRD; Empyrean; Panalytical B.V.) techniques. The porcelain was removed from one specimen in each group, and the specimens were ground to 2000-grit with wet silicon carbide abrasive papers (NAMKYUNG ABRASIVE Co, Ltd), followed by polishing with diamond (3.5 μm and 1.5 μm) and alumina (0.05 μm) polishing pastes (Wuyi Hengyu Instrument Co, Ltd), and then a specimen in each group was etched for 10 minutes by immersing in a hydrochloric acid/hydrogen peroxide (80:20, v/v) solution at room temperature. Two specimens from each group were embedded in resin for interfacial characterization, with similar preparation to the metallographic experiments, except without etching. The morphology and elemental distribution of metal-porcelain interface were analyzed by scanning electron microscopy coupled with energy-

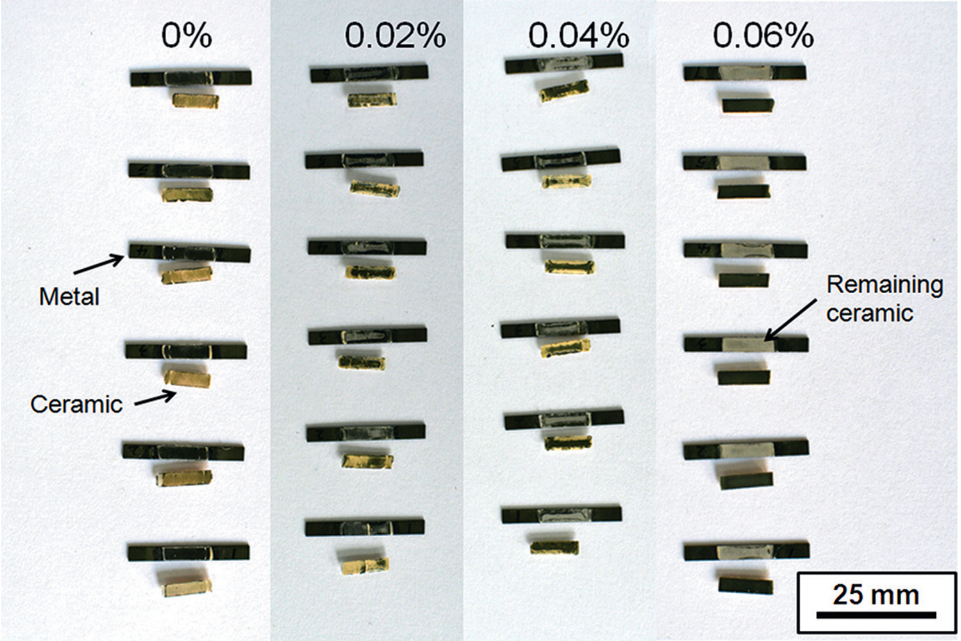


Figure 2. Optical micrographs of debonded surfaces of 4 groups after 3-point bend test.

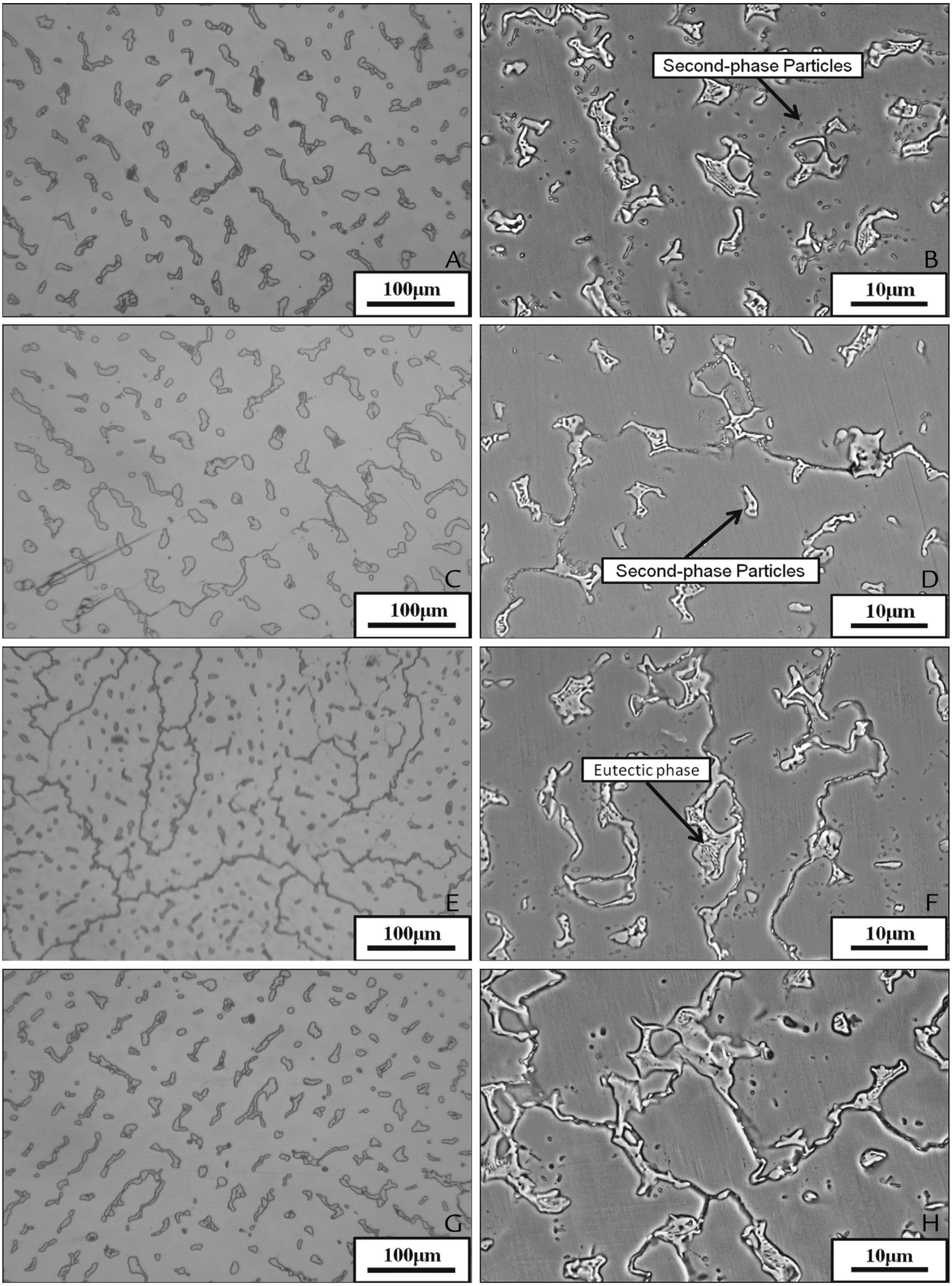


Figure 3. Metallographic images from polished section of 4 groups with different La content. A and B, Group 0%. C and D, Group 0.02%. E and F, Group 0.04%. G and H, Group 0.06% (original magnification ×100 and ×500). La, lanthanum.

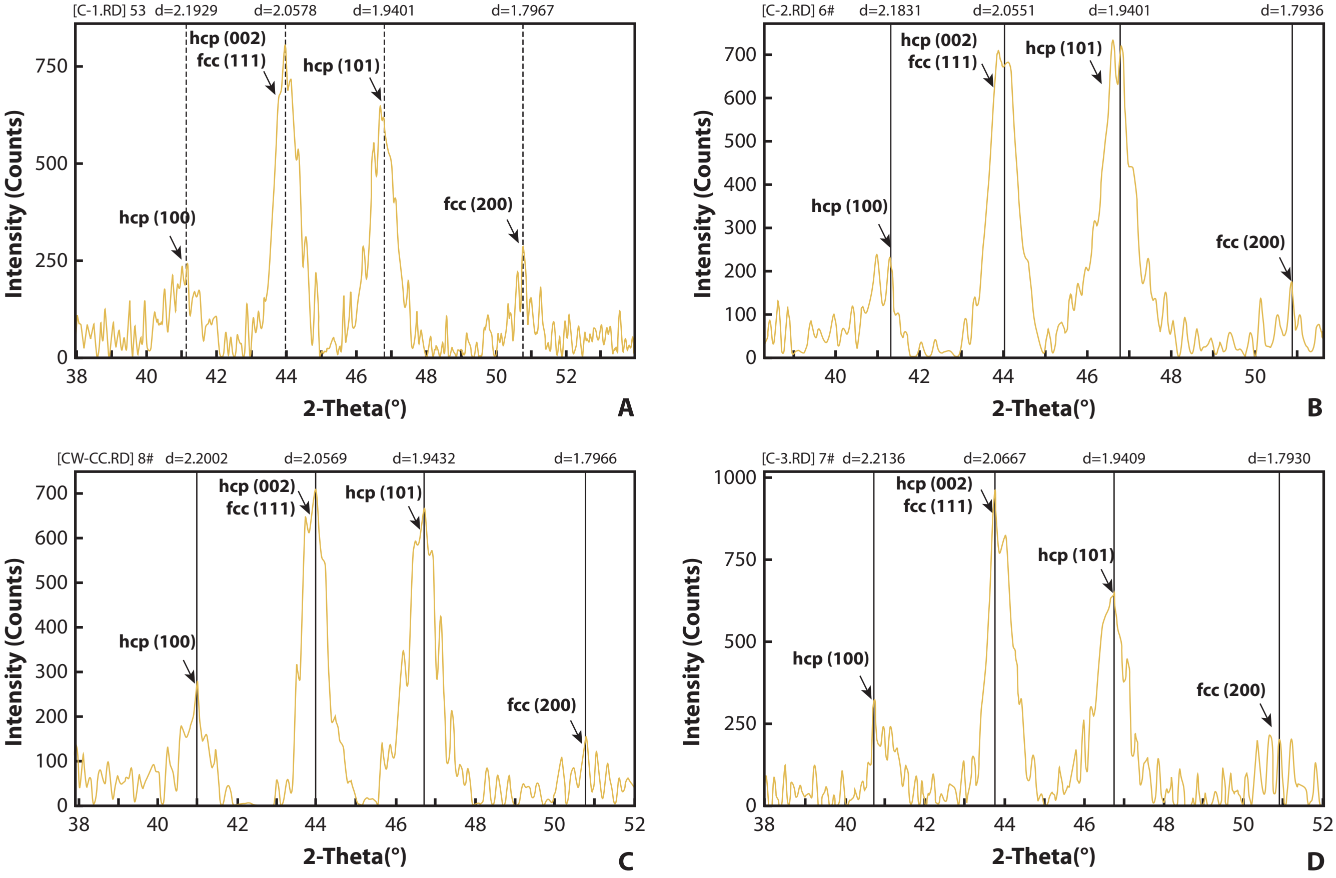


Figure 4. XRD spectra of 4 groups of Co-Cr alloy with different La content. A, Group 0%. B, Group 0.02%. C, Group 0.04%. D, Group 0.06%. La, lanthanum; XRD, X-ray diffractometer.

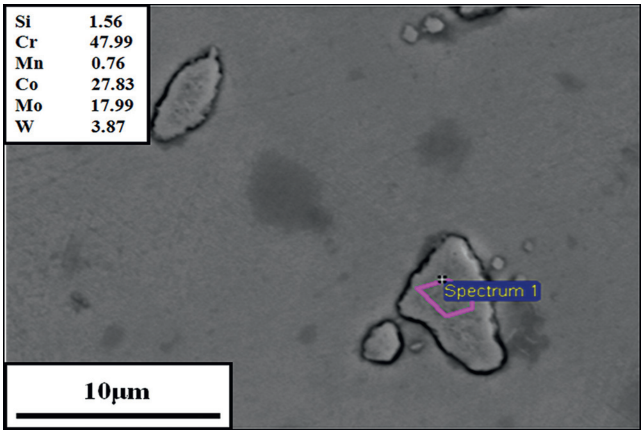
dispersive X-ray spectroscopy. XRD measurements were used for identification and quantification of phases presented in the Co-Cr alloys, and the XRD patterns were obtained using Cu-K$_{\alpha 1}$ radiation (wavelength: 0.15418 nm) and $\theta/2\theta$ scans between 38 and 54 degrees at a scan rate of 3 degrees per minute.

Differences among the four groups in bond strength were assessed by the Tukey honest significant difference (HSD) test using a statistical software program (IBM SPSS Statistics, v19.0; IBM Corp) (α=.05).[22]

RESULTS

Table 2 shows that as the addition of La increased, the bond strength of Co-Cr alloys improved, and significant differences were found (P<.05) among all groups except for groups 0.04% and 0.06%.

The optical micrographs indicated that the failure type of the 4 groups was a mixture of adhesive and cohesive fractures (Fig. 2). The debonded surface of the group 0% had only a small amount of remaining ceramic, almost an adhesive fracture. The debonded surfaces of groups 0.02%, 0.04%, and 0.06% contained obvious remaining ceramic; moreover, as the addition of La increased, the amount of remaining ceramic increased, especially the failure type of group 0.06% being almost a cohesive fracture.



Si	1.56
Cr	47.99
Mn	0.76
Co	27.83
Mo	17.99
W	3.87

Figure 5. Composition of second-phase particles of Co-Cr alloy (original magnification ×1500).

The Co-Cr dental casting alloys exhibited a typical dendritic microstructure (Fig. 3). The XRD analysis confirmed they consisted mainly of the α-Co solid

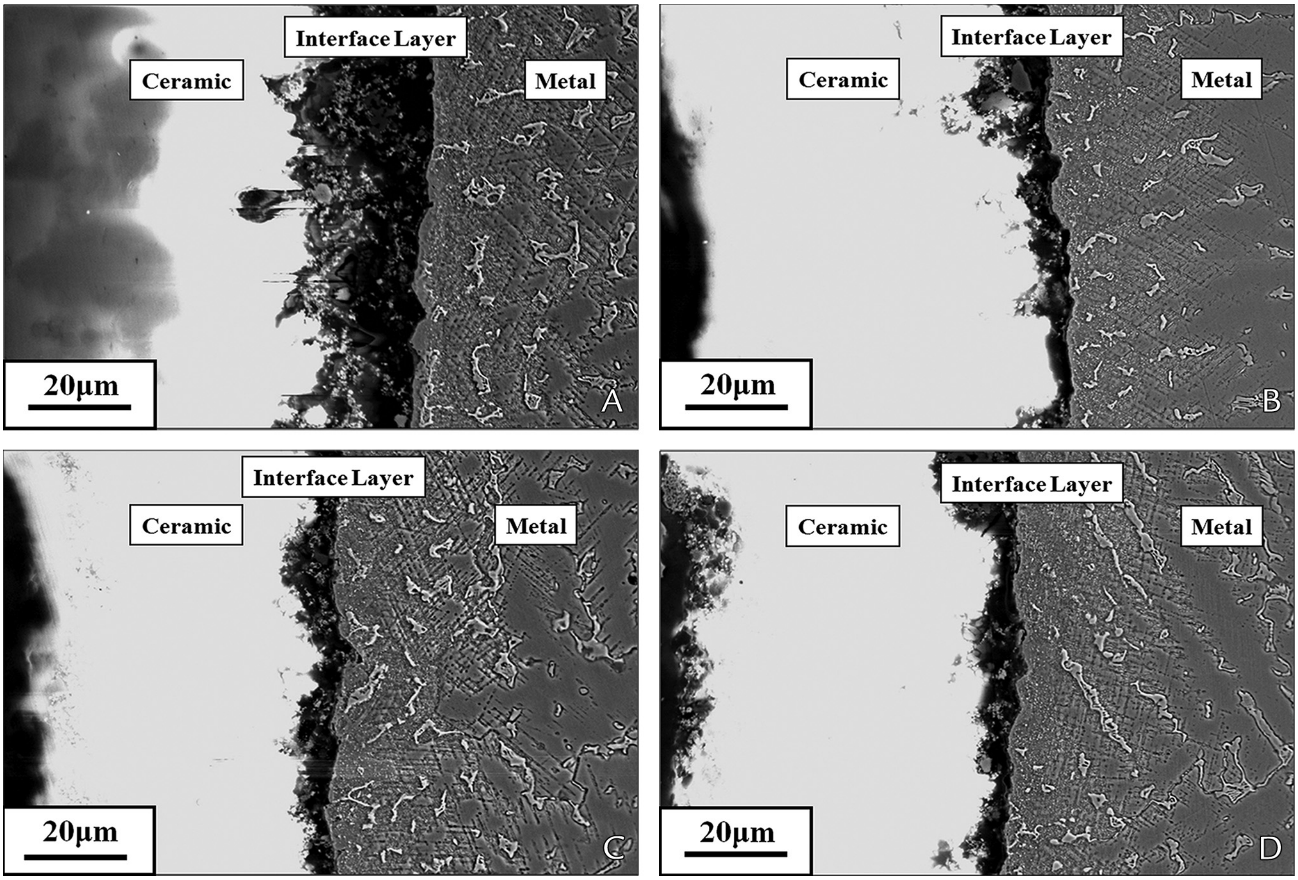
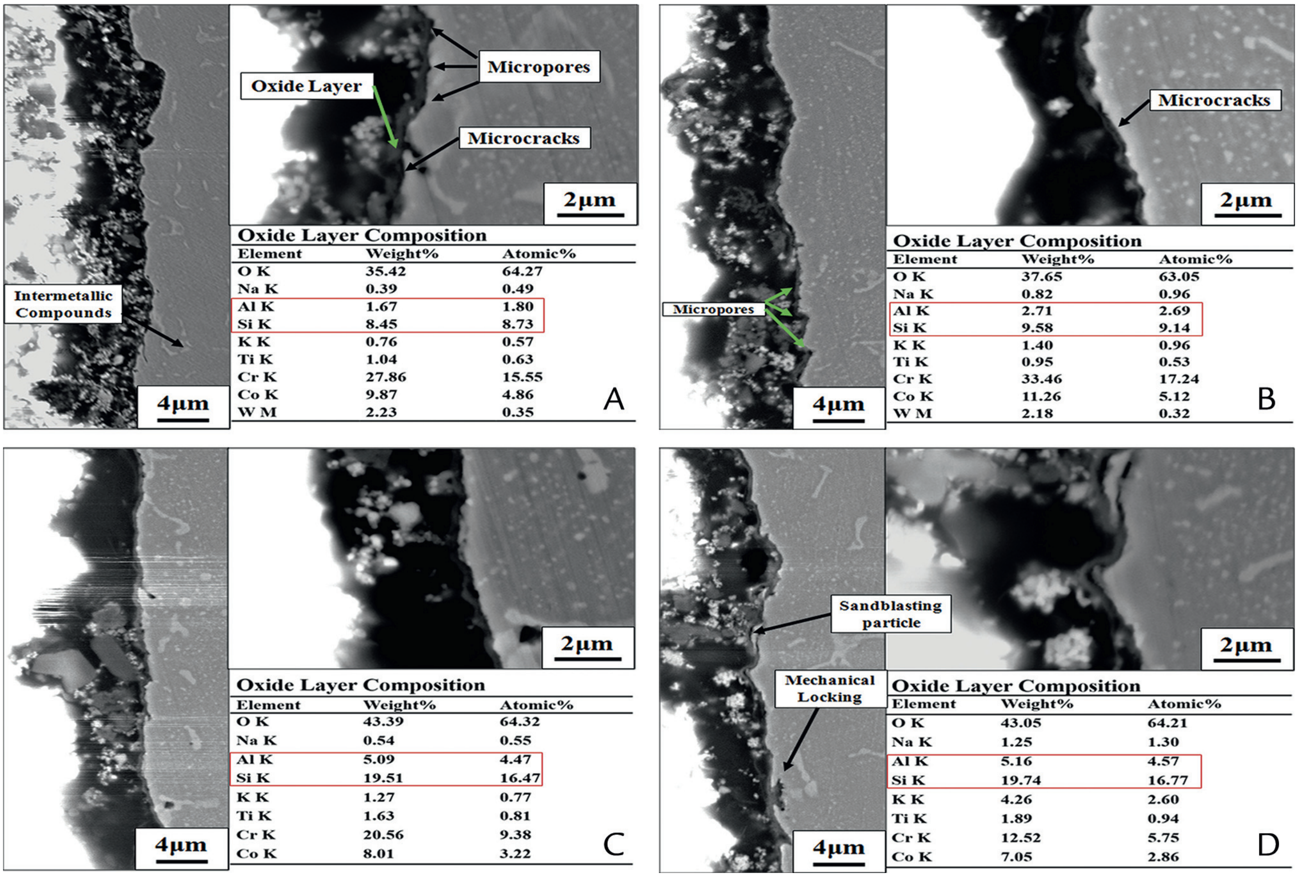


Figure 6. Scanning electron micrographs of metal-ceramic interfaces of etched specimens. A, Group 0%. B, Group 0.02%. C, Group 0.04%. D, Group 0.06% (original magnification ×1000).

solutions of a face-centered cubic (FCC) structure and ε-Co solid solutions of a close-packed hexagonal (HCP) structure (Fig. 4), and these results were consistent with those of the study by Jabbari et al.[10] The higher magnification images (Fig. 3B, D, F, H) demonstrated the existence of numerous island-shaped second-phase particles and a small amount of eutectic-phase particles at the interdendritic regions. An EDS analysis performed on the second-phase particles showed that the main composition was 48Cr-28Co-18Mo-4W-Si-Mn (Fig. 5). To estimate the volume fractions of the FCC-Co phases in the 4 groups, the integrated intensities of the $(200)_{fcc}$ and $(101)_{hcp}$ peaks were used. The quantitative determination, performed by using the method of Sage and Gillaud (Revue De Métallurgie 1950; 49:139-45), resulted in the α-Co–phase volume fractions of 39.69%, 34.60%, 34.01%, and 38.98% for the 4 groups. The differences were not statistically significant ($P>.05$). Consequently, the addition of a small amount of La had no obvious effect on the microstructures or phase compositions of the Co-Cr alloys.

The morphology of the metal-ceramic interface of etched specimens showed numerous pits on the metal surface from the airborne-particle abrasion that increased the effective areas of the metal-ceramic interface and promoted mechanical bonding (Fig. 6). A large amount of isothermal martensites (ε-Co solid solution) occurred within a distance of 50 μm from the metal-ceramic interface. From the Co-Cr binary system, it was found that the FCC phase possessed a lower energy (G) at high temperatures, whereas the thermodynamically stable phase was expected to be HCP ε-Co. Martensitic phase transformation ($\alpha \rightarrow \varepsilon$) was rather sluggish under normal cooling conditions primarily due to limited chemical driving forces available at the room temperature. However, the porcelain firing process provided chemical driving forces for the element diffusion, which also satisfied the conditions of martensite transformation at the interface.

The morphology and composition of native oxide films showed that the composition was mainly composed of the oxides of Co, Cr, Al, and Si (Fig. 7). Furthermore, the content of Al and Si in the native oxide layer increased with the increase of La. As is commonly observed, there was almost no oxide layer in the group 0%, and the oxide layer was thin and discontinuous. Many micropores and microcracks were evident in the interface between the native oxide layer and metal



Oxide Layer Composition (A)

Element	Weight%	Atomic%
O K	35.42	64.27
Na K	0.39	0.49
Al K	1.67	1.80
Si K	8.45	8.73
K K	0.76	0.57
Ti K	1.04	0.63
Cr K	27.86	15.55
Co K	9.87	4.86
W M	2.23	0.35

Oxide Layer Composition (B)

Element	Weight%	Atomic%
O K	37.65	63.05
Na K	0.82	0.96
Al K	2.71	2.69
Si K	9.58	9.14
K K	1.40	0.96
Ti K	0.95	0.53
Cr K	33.46	17.24
Co K	11.26	5.12
W M	2.18	0.32

Oxide Layer Composition (C)

Element	Weight%	Atomic%
O K	43.39	64.32
Na K	0.54	0.55
Al K	5.09	4.47
Si K	19.51	16.47
K K	1.27	0.77
Ti K	1.63	0.81
Cr K	20.56	9.38
Co K	8.01	3.22

Oxide Layer Composition (D)

Element	Weight%	Atomic%
O K	43.05	64.21
Na K	1.25	1.30
Al K	5.16	4.57
Si K	19.74	16.77
K K	4.26	2.60
Ti K	1.89	0.94
Cr K	12.52	5.75
Co K	7.05	2.86

Figure 7. Morphology and composition of oxide layers of 4 groups. A, Group 0%. B, Group 0.02%. C, Group 0.04%. D, Group 0.06% (original magnification: left, ×2000; right, ×10 000).

matrix, which indicated that the wettability of the oxide layer was poor (Fig. 7A). The oxide layer of group 0.02% was so thin that the native oxide layer could not be fully spread at the metal surface (Fig. 7B). In contrast, the oxide layers of groups 0.04% and 0.06% (about 0.5 µm) had a clear outline and few defects, and the oxide layers could be fully spread to the small pits of the metal surface due to better wettability (Fig. 7C, D). In addition, the oxide layer of group 0.06% grew to the metal substrate to provide mechanical locking, increasing the mechanical bonding (Fig. 7D).

The results of the EDS analysis, measured across the metal-ceramic interface, determined that the thickness of the diffusion layer was 2 µm for group 0% (Fig. 8A), 5 µm for group 0.02% (Fig. 8B), 5.6 µm for group 0.04% (Fig. 8C), and 7 µm for goup 0.06% (Fig. 8D). As the increase of La, the thickness of the diffusion layer increased, which demonstrated that the result was analogous to that of the native oxide layer.

DISCUSSION

In this study, the metal-ceramic bond strengths of Co-Cr dental casting alloys with different La content were investigated and compared with each other. The findings of this study showed that La could significantly improve the metal-ceramic bond strength, and thus, the null hypothesis that there would be similar metal-ceramic bond strength among the groups with different La content was rejected.

The metal-ceramic bonding mechanism has been attributed to chemical bonding, mechanical bonding, compressive bonding, and Van der Waals force, in which the chemical bonding plays a major role.[8,23-25] Chemical bonding, as the predominant factor, depends primarily on the chemical reaction at the metal-ceramic interface, and as a result, the nature and morphology of the oxide layer on the surface play a crucial role in chemical bonding. In this study, the effect of La is mainly embodied in improving chemical bonding force. Mechanical bonding depends mainly on the surface roughness and wettability, which is intimately related to airborne-particle abrasion process and porcelain technology. Compressive bonding is the result of the microstructure change produced by the thermal effects, more precisely, which is caused by the difference in the coefficient of thermal expansion between the metal and ceramic. The Van der Waals force is inevitable and

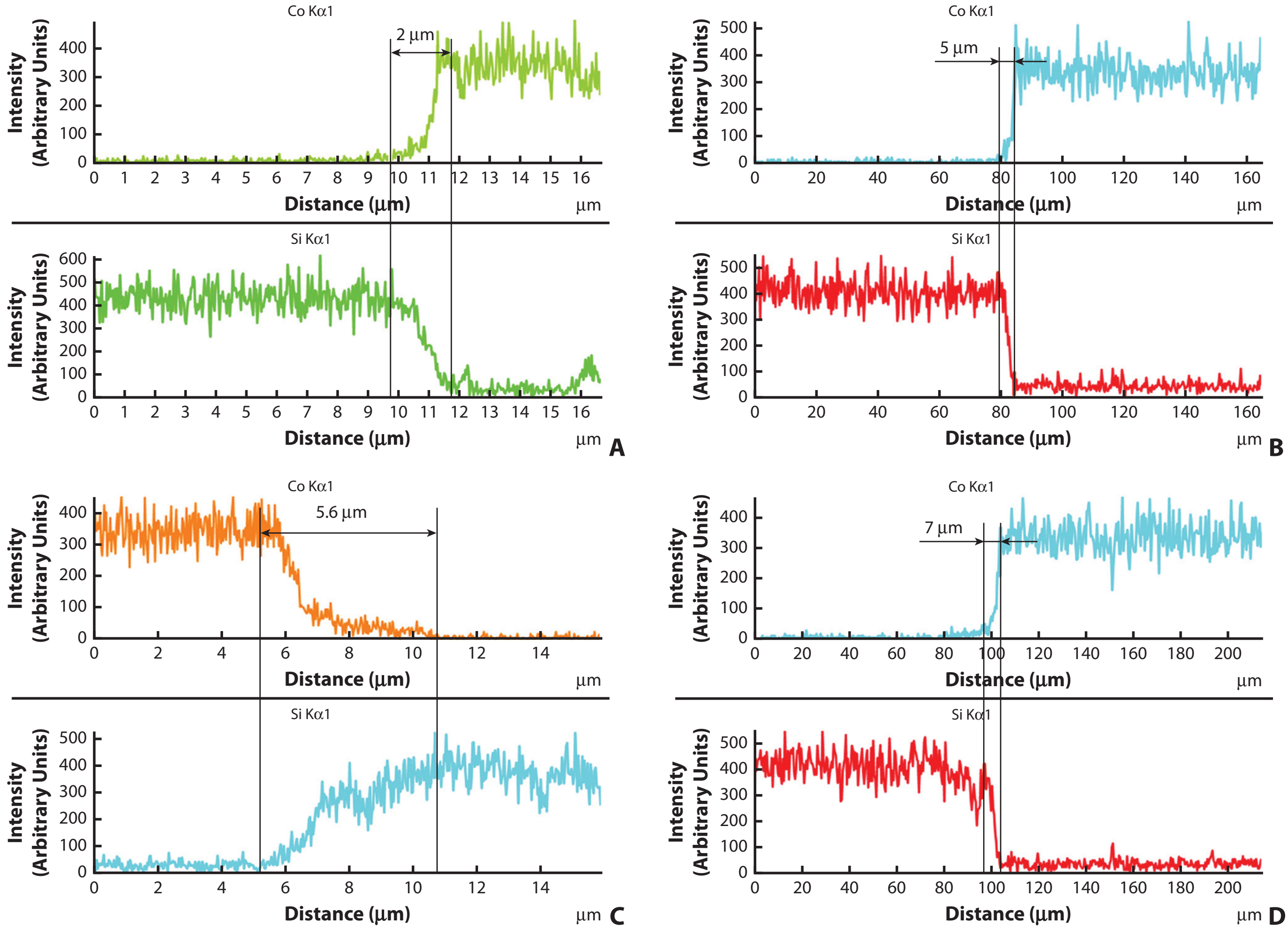


Figure 8. Energy-dispersive X-ray spectroscopy line analysis of metal-ceramic interfaces. A, Group 0%. B, Group 0.02%. C, Group 0.04%. D, Group 0.06%.

difficult to control in the material study but only accounts for a little part of the influencing factors.

The RE La element, as a chemically active element, could refine grains, reduce the solute segregation, and improve the microstructure. La could react with most of the elements in the alloy to form compounds with high melting point, high hardness, and high dispersibility, and it has a larger atomic radius;[26] thus, the strengthening effects of La on the alloy include solution strengthening, second-phase strengthening, and grain-refining strengthening. These strengthening effects improve the mechanical properties of the Co-Cr alloy; furthermore, the improvement of the microstructures and properties of the metal affects the native oxide layer.[27] The homogeneity of the microstructures made the composition of the oxide layer uniform and had a better continuity, improving the corrosion resistance of the Co-Cr alloy. Importantly, La oxides exist in the native oxide layer and changed the nature of the oxide layer, which is the most significant factor affecting the bond performance. In addition, La oxides reduced the interface energy of the oxide layer, thereby improving the wettability between the metal and native oxide layer.[28]

La is prone to react with Cr_2O_3 and Al_2O_3 and changed the penetrability of the oxide layer, making it easier for atoms in metals and ceramics to pass through the oxide layer and to diffuse and react to form a chemical bonding. Simultaneously, the La addition increased the resistance to high-temperature oxidation,[29] and the La oxide existed at the interface and was deeply rooted in the metal matrix, improving the mechanical bonding between the oxide layer and metal matrix. In addition, the oxide layer was formed by the outward diffusion of metal ions and inward diffusion of oxygen ions. After the diffusion of metal cations, vacancies could be left in the metal matrix, and the accumulation of vacancies would give rise to defects between the metal and the oxide layer, which reduced the bond strength; however, the addition of La element could reduce the outward diffusion of metal cations.

The effect of La on the bond properties is presented in Figure 9. After the porcelain specimens are heated in the

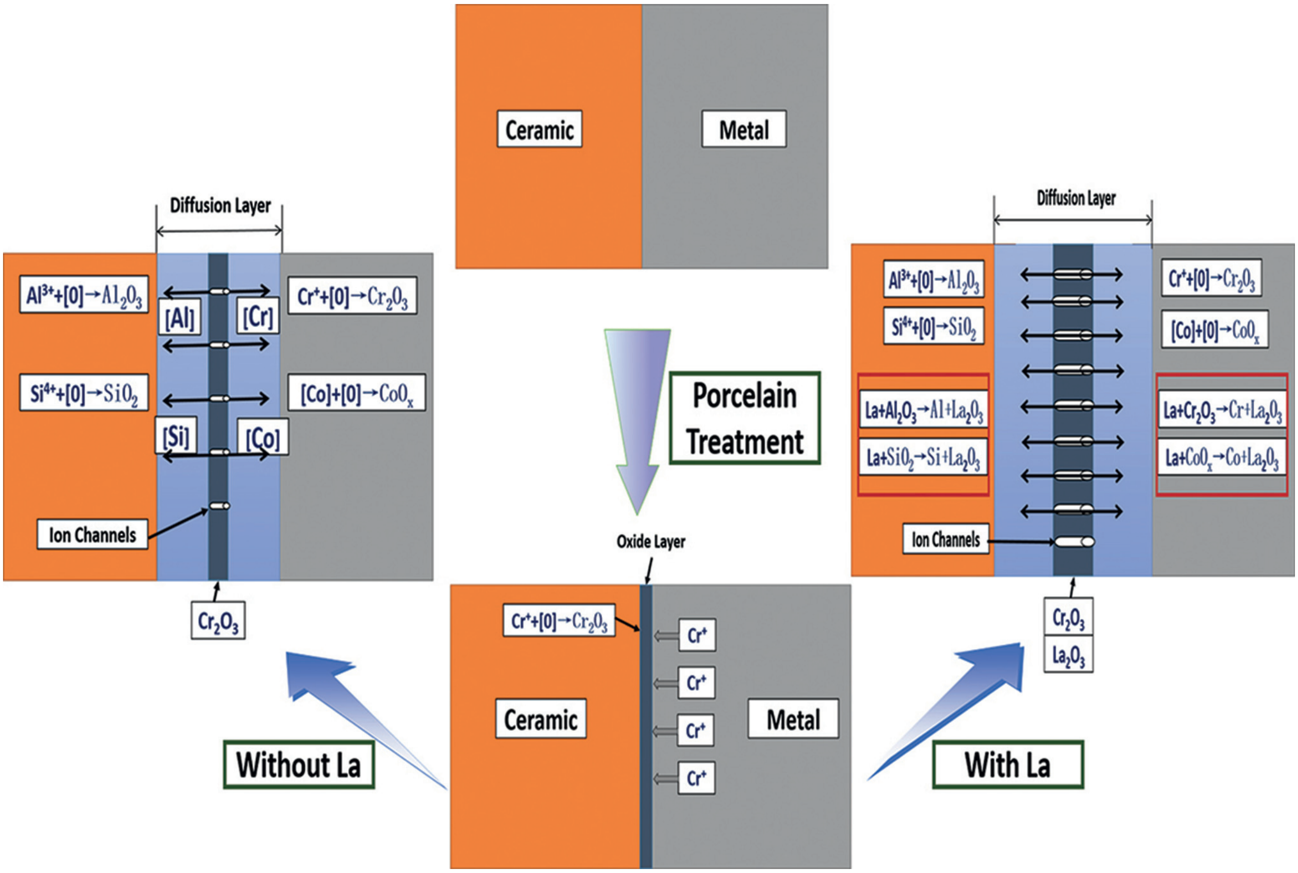


Figure 9. Mechanism of effect of La on native oxide layer. La, lanthanum.

porcelain furnace, ceramic and metal atoms began to diffuse as the temperature increased. Metal ions in the metal matrix diffused to the metal-ceramic interface and are combined with [O] in the ceramic to produce chemical reaction (1) to generate M_xO_y. Compared with Co, Si, Mo, W, and Ti, Cr had a lower standard free energy of the reaction with O; therefore, the main composition of the oxide layer was Cr_2O_3. In the absence of La, the thickness of the chemical reaction diffusion layer is limited because the diffusion of ceramic and metal atoms depends mainly on the ion channels in the oxide layer, and the Cr_2O_3 oxide layer is compact. After adding La into the Co-Cr alloy, La has strong chemical affinity to [O] and reacts with oxides due to its excellent activity (reaction (2)), especially in high temperature during the porcelain firing. Therefore, the La addition causes the main compositions of the oxide layer to become La_2O_3 and Cr_2O_3. The properties of the La_2O_3 are different from those of the Cr_2O_3, and the most prominent point is that the number of ion channels is larger than that in the other oxide layers. The EDS analysis (Fig. 7) validates this interpretation. Consequently, the addition of La would greatly increase the thickness of the reaction diffusion layer and oxide layer and increase the chemical bond force. Under certain conditions, due to the improvement of chemical reactions, La oxides may penetrate into the metal matrix and further improve the mechanical bond force.

$$(M)^{n+}+[O]\rightarrow M_xO_y \tag{1}$$

$$La+M_xO_y\rightarrow La_2O_3+M \tag{2}$$

Overall, the improvement of the metal-ceramic bond strength and the study of La in Co-Cr alloy have a great value for application. The solution of the bond strength problem will improve metal-ceramic restorations with Co-Cr alloy. Nevertheless, additional studies are required to investigate other properties including mechanical properties, corrosion resistance, and biocompatibility.

CONCLUSIONS

Based on the findings of this in vitro study, the following conclusions were drawn:

1. The addition of the La improved the metal-ceramic bond strength, and the debonded surfaces of Co-Cr specimens displayed a mixture of adhesive and cohesive fractures.

2. There was no significant difference in the microstructure and phase content of the 4 groups. With the increase of La, the thickness of the oxide layer and diffusion layer and the wettability between the oxide layer and the metal matrix increased.

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Corresponding author:
Dr Ning Li
School of Manufacturing Science and Engineering
Sichuan University
No. 24 South Rd, Yihuan Road
Wuhou District
Chengdu, Sichuan 610065
PR CHINA
Email: lining@scu.edu.cn

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