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Metal-Ceramic Interface Evaluation of a Gold-Infiltrated Alloy

Rupal Vasani, BDS, MS,[1] Isao Kawashima, PhD,[2] Gerald J. Ziebert, DDS, MS,[1] & David W. Berzins, PhD[3]

[1]Department of Prosthodontics, Marquette University School of Dentistry, Milwaukee, WI
[2]Department of Dental Biomaterials, Ohu University School of Dentistry, Koriyama, Japan
[3]Department of Dental Biomaterials, Marquette University School of Dentistry, Milwaukee, WI

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Correspondence
David W. Berzins, Marquette University School of Dentistry-General Dental Sciences, 1801 W. Wisconsin Ave., Milwaukee, WI 53233. E-mail: david.berzins@marquette.edu

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Abstract

Purpose: The success of metal-ceramic systems partially depends on the formation of a stable bond between metal and porcelain. The purpose of this study was to investigate the porcelain/metal interface and the mechanism of interfacial bonding in a gold-infiltrated alloy (Captek).

Materials and Methods: Captek specimens with feldspathic porcelain were evaluated by optical microscopy, scanning electron microscopy (SEM), electron probe microanalysis (EPMA), X-ray diffraction (XRD), and the Schwickerath crack initiation test for metal-ceramic bond compatibility. Specimens were processed with or without Capbond, a bonding agent. A traditional metal-ceramic alloy was also analyzed with microscopy for comparative purposes.

Results: Optical and scanning electron micrographs of Captek specimens processed with Capbond revealed close adaptation of porcelain to the surface of the metal with sporadic nodules extending from the Captek surface. In contrast, the specimens of Captek without Capbond showed a much flatter porcelain/metal interface. Comparatively, the porcelain/metal interface of the traditional metal-ceramic crown showed greater surface roughness than the Captek specimens. No metal oxides were observed at the porcelain/metal interface of the Captek specimens with XRD. During the Schwickerath test, the Captek specimens permanently deformed, not allowing for crack initiation at the porcelain/metal interface.

Conclusions: Microscopy and XRD analysis showed that micromechanical interlocking is the primary mechanism of porcelain adherence to Captek metal. The use of Capbond prior to porcelain application to Captek results in gold nodules on the surface to aid retention. Existing metal-ceramic bond compatibility standardized tests are not sufficient for evaluating Captek, primarily due to the flexibility of the material.

Introduced in 1956,[1] conventional metal-ceramic restorations remain the gold standard of fixed prosthodontics. Metal-ceramic restorations combine the esthetic qualities of porcelain with the strength, toughness, accuracy, and marginal adaptation of a metal substructure.[2-4] Although long-term clinical trials have shown traditional metal-ceramic systems to be very successful, a number of alternative systems have been developed in an attempt to improve the traditional metal-ceramic restoration. In an effort to eliminate the time-consuming casting process and also to produce a thinner metal core to optimize esthetics, various foil systems have been developed over the years. Some product examples have included Renaissance (or Ceplatec in Europe), Sunrise, Plati-deck, and Flexobond crowns;[5-8] however, due to poor short-term clinical data and mechanical properties, these systems fell out of favor. Captek (Precious Chemicals Company Inc., Altamonte Springs, FL) is the latest in the advancement of noncast metal substructure systems by employing capillary attraction to flow nearly pure gold into a high-noble metal 3D capillary network to produce a composite metal alloy substructure. The manufacturer claims that this system provides superior esthetics due to its inherent warm gold color and the ability to make thinner metal copings.

The longevity of the metal-ceramic system depends upon the formation of a stable bond between metal and porcelain that can withstand stresses common in the oral cavity. Thus, the metal-ceramic interface is critical in the functional and esthetic success of metal-ceramic prostheses. Conventional restorations invoke a combination of chemical, mechanical, van der Waals forces, and a slight coefficient of thermal expansion (CTE) mismatch contracting forces to fuse porcelain to metal.[9-11] Chemical bonding, believed to play the most important role in conventional metal-ceramic restorations, is dictated by an oxide layer formed on the metal substrate during firing, developing metallic, ionic, and covalent bonds with the oxides in the porcelain opaque layer.[12-14] The mechanical bond depends primarily on the surface roughness of the metal casting, whereas van der Waals forces are a minor contributor to the overall metal-ceramic bond strength.

Contrary to this is the Captek system, as the manufacturer claims that there is no chemical bond at the metal-ceramic interface, rather only mechanical interlocking.[15,16] A review of the literature revealed only one study in a peer-reviewed journal (co-authored by a Captek developer) and a dissertation listed in the manufacturer's literature as having evaluated the bond strength and/or microstructural aspect of the porcelain/metal interface.[15-17] With this limited research on the Captek system and the advancement of similar non-lost-wax cast process technologies,[18] it is imperative to completely characterize the critical porcelain/metal interface of these nonconventional methodologies. More insight into the chemical and physical components at the metal-ceramic interface would aid in understanding the bonding mechanisms, predictability, and ultimately the success of the material, and also provide information for further evolutions of newer techniques in metal-ceramic restorations.

The purpose of the present study was to investigate the porcelain/metal interface and the mechanism of metal-ceramic bonding in Captek. This was accomplished using optical microcopy, scanning electron microscopy (SEM) with electron probe microanalysis (EPMA), X-ray diffraction (XRD), and the Schwickerath crack initiation test for metal-ceramic bond compatibility.

Materials and methods

Captek specimens were prepared following the manufacturer's instructions. First, the Captek P component was fired in a porcelain furnace (Centurion VPC, Ney, Yucaipa, CA) at 1075°C for 4 minutes in air. Next, Captek G was placed on Captek P and processed with the same firing cycle to form the Captek specimens. Since early anecdotal accounts had reported some incidence of porcelain/metal bond failures, an intermediate material known as Capbond was introduced by the manufacturer to be added to the Captek surface prior to porcelain application. Therefore, the use of Capbond was introduced as a variable in this study to better understand its effect on the porcelain/metal interface. For each test mentioned below, randomly selected specimens were processed with or without Capbond.

Optical microscopy and scanning electron microscopy

Optical microscopy and SEM were employed to examine the morphology of the porcelain/metal interface. Eight specimens of Captek ($10 \times 0.4 \times 3$ mm) were prepared, with four being processed using Capbond. Feldspathic porcelain (Omega 900, Vident, Brea, CA) was sequentially applied in several layers to all specimens for an overall porcelain dimension of approximately $6 \times 1 \times 3$ mm centered on top of the Captek specimens. Initial porcelain application consisted of two layers of opaque porcelain individually fired at 990°C, under vacuum, in a furnace. Subsequently, opaque dentin, dentin, and enamel porcelains were condensed and fired individually at temperatures of 970, 940, and 925°C, respectively, following the manufacturer's instructions. In addition to the specimens listed above, two full-coverage, premolar crowns consisting of Captek (with or without Capbond) and veneering feldspathic porcelain were prepared to examine clinically relevant shaped specimens. Also, two crowns of a conventional metal-ceramic alloy (Olympia, Heraeus Kulzer, Armonk, NY) with veneering porcelain were similarly analyzed for comparative purposes. These crowns were sectioned for microscopic analysis. All specimens were individually mounted in clear acrylic (Sampl-Kwick, Buehler, Lake Bluff, IL), ground with silicon carbide paper (Carbimet Discs, Buehler) using standard metallographic procedures, and polished with 1.0 μm aluminum oxide paste (Alpha Micropolish Alumina, Buehler). The specimens were examined with a metallurgical microscope (Olympus PME3, LECO Corporation, St. Joseph, MI), and images were captured with a digital image acquisition device (SPOT Insight™ 2MP Firewire Mono, Diagnostic Instruments Inc., Sterling Heights, MI) and software (SPOT Software 4.5, Diagnostic Instruments Inc.). Further analyses were carried out using a scanning electron microscope (X-650, Hitachi, Tokyo, Japan) equipped with energy dispersive X-ray spectroscopy (EDS) and wavelength dispersive X-ray spectroscopy (WDS).

X-ray diffraction

X-ray diffraction, an analytical technique able to identify and characterize the structure of crystalline materials, is useful for studying dental alloy oxidation and porcelain/metal interfaces.[19,20] Four specimens of Captek (2 with and without Capbond) were prepared as described above; however, to achieve a minimum thickness of porcelain, only a thin layer of opaque porcelain was applied to the specimens. This is required because the depth of penetration of the X-ray beam is typically 50 μm or less, and the specimens were analyzed in two arrangements with the X-ray beam either incident upon the porcelain surface or along the cross-section of the porcelain/metal interface. Additionally, porcelain without the Captek substrate was also examined. The specimens were analyzed with a micro-XRD unit (RINT-2500, Rigaku Corporation, Tokyo, Japan) using Cu Kα radiation with a Ni filter. Scans were conducted at 2°/min over a 2θ range of 30 to 120°, and XRD pattern peaks were indexed with ICDD-2000 files (International Center for Diffraction Data, Swarthmore, PA).

Schwickerath crack initiation test for metal-ceramic bond compatibility

The Schwickerath crack initiation test described in ISO 9693 for metal-ceramic dental restorative systems was followed to test for the debonding/crack initiation strength of the porcelain/metal interface. Four rectangular Captek specimens ($25 \times 0.45 \times 3$ mm^3) with an applied area of porcelain ($8 \times 1 \times 3$ mm^3) were prepared according to ISO 9693 and as described above (Fig 1). The specimens were subjected to a three-point bending test at crosshead speed of 1.5 mm/min using a universal testing machine (Instron Corporation, Norwood, MA) with the porcelain side opposite to the applied load. Additionally, four rectangular Captek specimens without porcelain were tested to determine the elastic modulus of Captek.

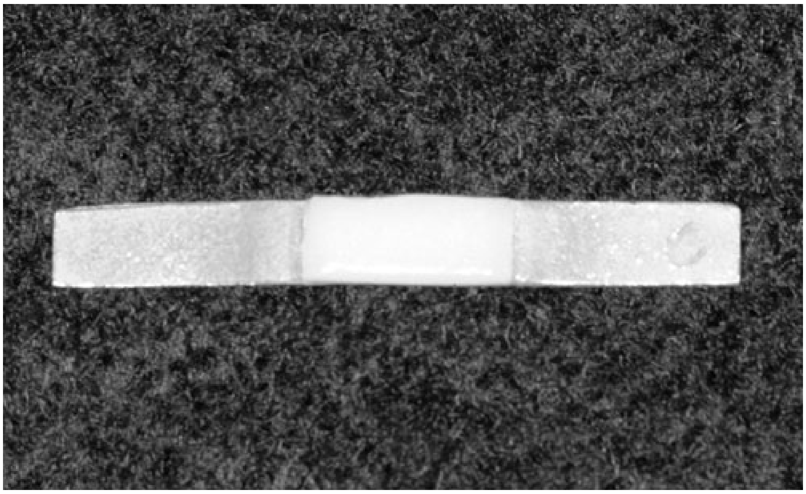

Figure 1 Captek specimen used for bond compatibility testing. Rectangular specimens for microscopy and XRD analyses were of similar design but different dimensions.

Results

Scanning electron and optical micrographs of Captek specimens processed with Capbond revealed close adaptation of porcelain to the surface of the metal with sporadic nodules extending from the Captek surface (Figs 2 and 3). In contrast, the specimens of Captek without Capbond showed a much flatter porcelain/metal interface. Comparatively, the porcelain/metal interface of the traditional metal-ceramic crown showed greater surface roughness than the Captek specimens but lacked the

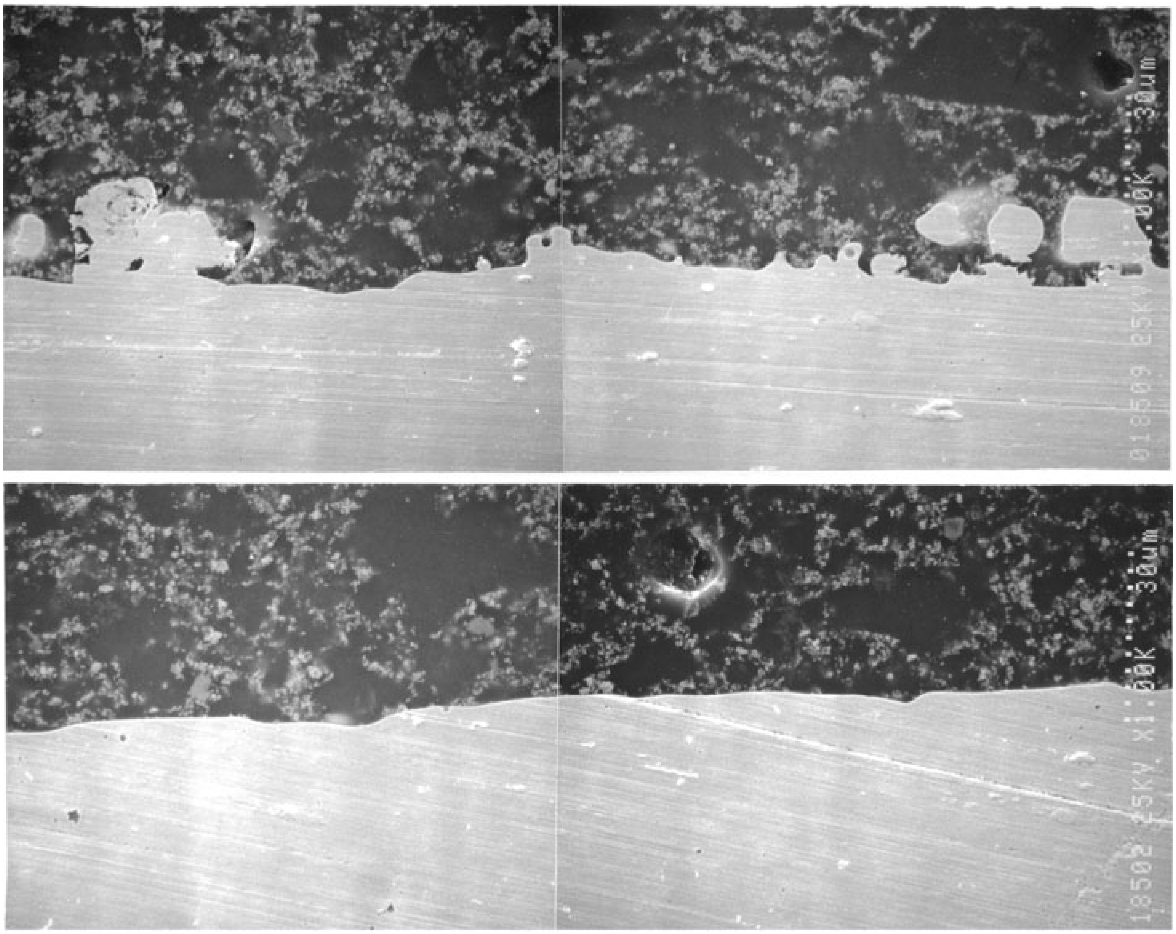

Figure 2 SEM micrographs of Captek processed with Capbond (top) and Captek without Capbond (bottom).

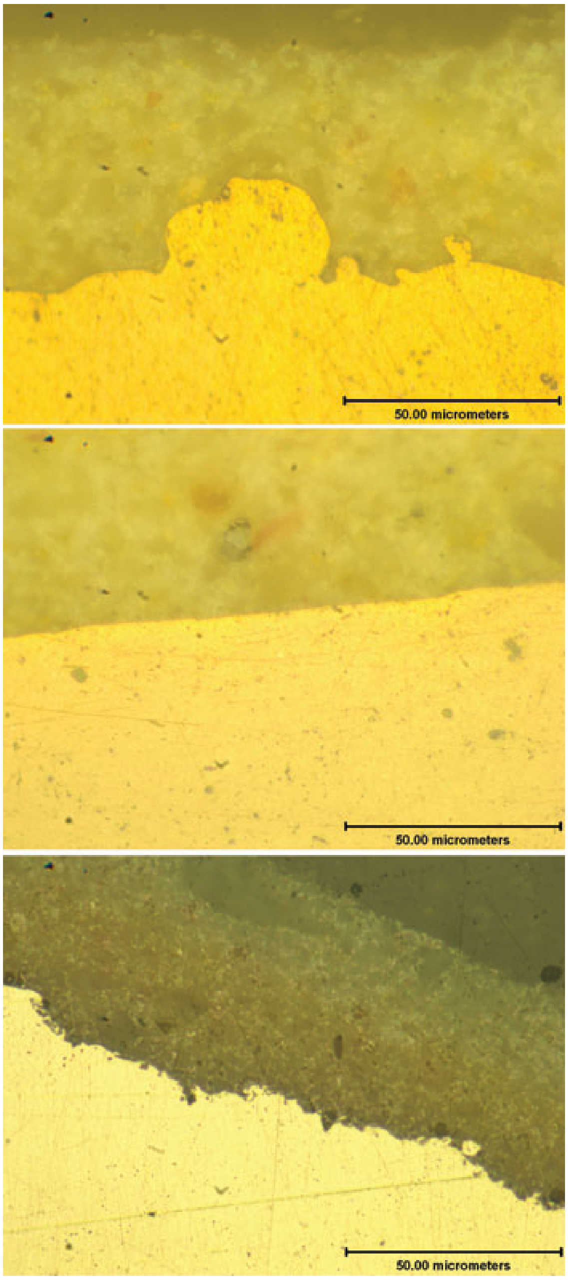

Figure 3 Optical micrographs of Captek processed with Capbond (top), Captek without Capbond (middle), and a traditional metal-ceramic (bottom) system.

larger nodules of the Capbond-processed specimens. EDS analysis coupled to the SEM showed the Captek metal (with and without Capbond) to be composed primarily of gold with minor amounts of palladium and silver. A micrograph with elemental maps showing the spatial distribution of gold and silicon constructed with the EPMA for a Captek specimen processed with Capbond is shown in Figure 4. Areas of penetration of gold into the silicon arising from the porcelain may be observed, further illustrating the 3D micromechanical profile of the porcelain/metal interface when Capbond is used. This feature was not observed on similar elemental maps for the Captek specimens without Capbond (not shown).

The X-ray diffraction analysis of the porcelain/Captek metal interface showed peaks consistent with a gold face-centered cubic (FCC) solid solution (Fig 5). Compared to the ICDD-2000 standard file for gold, the peaks were slightly shifted, reflecting the solid solution additions of silver and palladium. This shift, however, was less pronounced as the micro-XRD analysis progressed from the bulk metal to the porcelain/metal interface, illustrating that the surface of Captek is enriched in gold compared to the inner structure. No qualitative difference was found between the Captek specimens with and without Capbond, and no crystalline metal oxides were observed at the porcelain/metal interface.

During the Schwickerath crack initiation test for metal-ceramic bond compatibility, the Captek specimens permanently deformed, not allowing for crack initiation at the porcelain/metal interface. The elastic modulus of the Captek specimens without porcelain was determined to be 39 GPa (SD = 12).

Discussion

Microscopy and X-ray diffraction demonstrated that the primary retentive mechanism of porcelain on Captek metal is micromechanical and not chemical in nature. Microscopy revealed close adaptation of porcelain to the surface of the Captek metal with evidence that mechanical interlocking is formed mainly after the application of Capbond, as the specimens without Capbond showed a much flatter porcelain/metal interface. This may explain why Capbond was introduced to the fabrication process following anecdotal accounts of porcelain debonding. Initially, the precise details of Capbond were proprietary, although the current manufacturer's literature states that Capbond contains 25-μm gold particles,[21] which is consistent with the results of this study. The concept of using a gold-based agent as a coating or bonding agent with the aim of improving esthetics, strength, or bonding has been shown previously in both conventional metal-ceramic and foil crown systems.[22-24] It is interesting to note that the conventional metal-ceramic interface, where mechanical retention is typically thought to be secondary to chemical retention, appeared to actually be rougher than the Captek surface, although it lacked the nodular surface of the Capbond-processed Captek.

EDS analysis showed a lack of base metals in the Captek metal, whereas traditional metal-ceramic alloys contain oxide-forming base metals, such as tin, indium, gallium, or copper. Not surprisingly, no crystalline metal oxides were observed at the porcelain/metal interface of the Captek specimens when analyzed with micro-XRD. X-ray diffraction patterns of conventional metal-ceramic restorations have shown the presence of an oxide layer at the interface.[19,20] Thus, the porcelain/Captek bond is derived mainly by the microretentive surface architecture of the alloy. A previous noncast metal-ceramic restoration similarly showed a lack of base metals, oxide formation, and evidence of chemical bonding.[5] Retention of porcelain on the metal foil was highly localized mechanical retention and resulted from the penetration of porcelain into a porous alloy network and intergranular grooves. Micromechanical retention has also been ascribed as the primary mechanism of porcelain

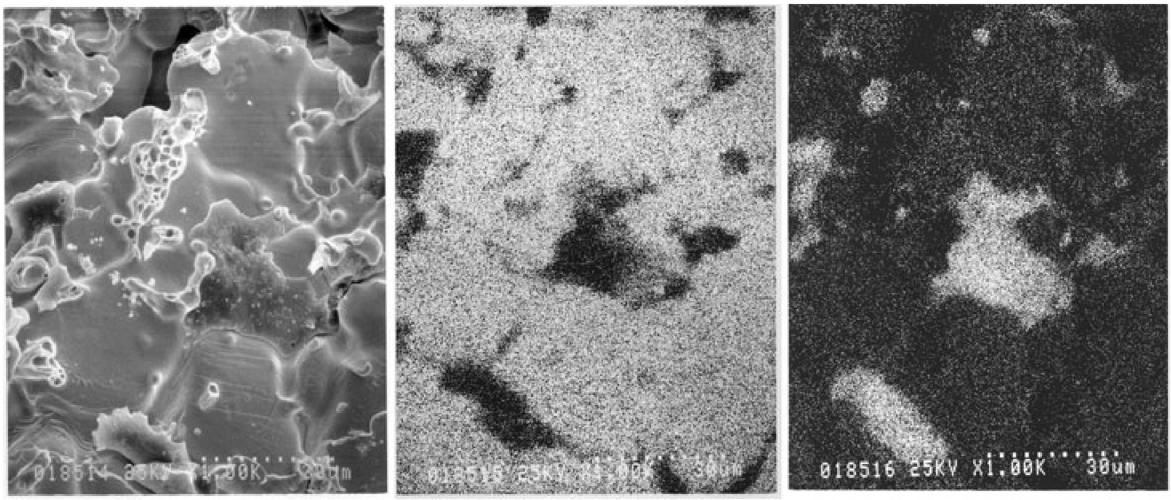

Figure 4 Micrograph (left) showing EPMA elemental map for silicon (middle) and gold (right). Light areas indicate the positive presence of the element.

bonding to palladium-silver alloys where Pd-Ag nodules form on the surface as a result of a Nabarro-Herring creep mechanism induced by the internal oxidation of base metals.[25] The Pd-Ag nodules in that study were smaller than those observed on the Captek specimens processed with Capbond. It should be mentioned that the effect of slight coefficient of thermal expansion mismatch between the porcelain and metal on retention was not studied; however, given the lack of recommendation from the manufacturer as to the selection of porcelain based upon CTE, it is presumed that this bonding mechanism plays little role in Captek restorations. While the results here are not surprising given the reported composition of Captek, the mechanism of bonding and characterization of the porcelain/metal interface had not been established by independent research and was only speculatively described. The question then arises if the level of micromechanical interlocking in Captek sufficiently bonds porcelain to the metal.

An experimental design that measures the absolute bond strength of metal-ceramic restorations appears singularly elusive.[26,27] Several tests encompassing shear, tension, flexure, and torsion stresses have been designed,[28] but the ability to compare different measured bond strengths is problematic. Thus, in 1996, the International Organization for Standardization (ISO) adopted a standardized test known as the Schwickerath crack initiation test for metal-ceramic bond compatibility. The Captek specimens were not suitable to be examined with this method because they permanently deform rather than having porcelain debond, illustrating Captek is much more flexible compared to other metal-ceramic systems that have been evaluated with this method. Further development of test methods to evaluate the porcelain/metal bond in this system and perhaps future nonconventional metal-ceramic restorations is needed. Very little information is found on the bond strength of porcelain to Captek, apart from reports arising from a doctoral research

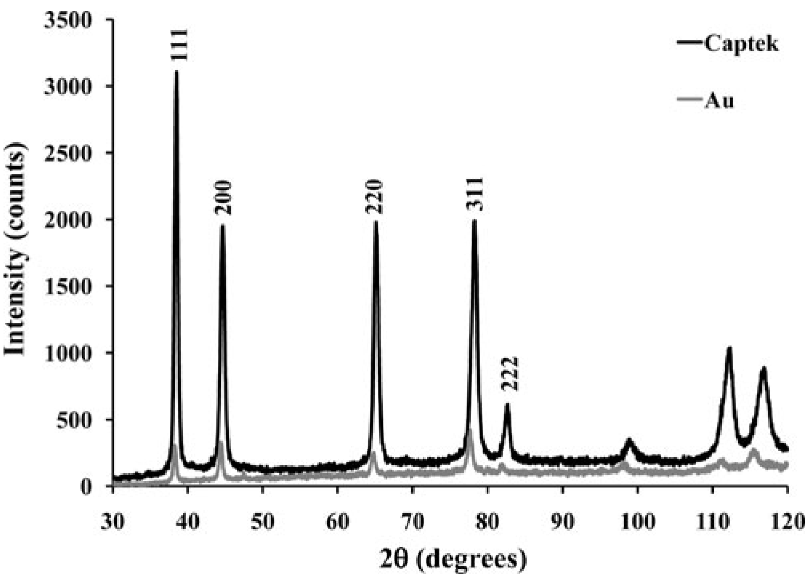

Figure 5 XRD patterns of the porcelain/Captek metal interface and gold from the ICDD file (bottom).

project where the shear bond strengths of Captek were found to be at least equal to those of conventional metal-ceramic systems;[17] however, these results have not appeared in the peer-reviewed literature.

The elastic modulus of Captek was determined to be 39 MPa, which is less than that of Type I gold alloys used for inlays[29] and also that of 99% pure gold.[30] A possible explanation for this might be incomplete infiltration of gold (Captek G) into the porous network rendered after the Captek P material is fired, resulting in an internal microstructure with voids or pores. As discussed previously, a porous network has been noted in other noncast metal-ceramic restorations,[5] although it was not reported in the study on Captek mentioned earlier.[15] Past research has shown substantial decreases in the elastic modulus and strength with increased porosity in a powder metallurgy-based alloy.[31] Whether this is a factor with Captek requires further research. Nevertheless, given its apparent flexibility, the scenario of using Captek for fixed partial dentures, as is currently practiced, should be approached cautiously.

Conclusion

Microscopy and XRD analysis showed that micromechanical interlocking is the primary mechanism of porcelain adherence to Captek metal. The use of Capbond prior to porcelain application to Captek results in gold nodules on the surface to aid retention. Existing metal-ceramic bond compatibility standardized tests are not sufficient for evaluating Captek, primarily due to the flexibility of the material.

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