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Influence of thermal and mechanical cycling on the flexural strength of ceramics with titanium or gold alloy frameworks

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ABSTRACT

Objectives. The aim of this study was to evaluate the effect of thermal and mechanical cycling alone or in combination, on the flexural strength of ceramic and metallic frameworks cast in gold alloy or titanium.

Methods. Metallic frameworks (25 mm × 3 mm × 0.5 mm) ($N = 96$) cast in gold alloy or commercial pure titanium (Ti cp) were obtained using acrylic templates. They were airborne particle-abraded with 150 μm aluminum oxide at the central area of the frameworks (8 mm × 3 mm). Bonding agent and opaque were applied on the particle-abraded surfaces and the corresponding ceramic for each metal was fired onto them. The thickness of the ceramic layer was standardized by positioning the frameworks in a metallic template (height: 1 mm). The specimens from each ceramic–metal combination ($N = 96$, $n = 12$ per group) were randomly assigned into four experimental fatigue conditions, namely water storage at 37 °C for 24 h (control group), thermal cycling (3000 cycles, between 4 and 55 °C, dwell time: 10 s), mechanical cycling (20,000 cycles under 10 N load, immersion in distilled water at 37 °C) and, thermal and mechanical cycling. A flexural strength test was performed in a universal testing machine (crosshead speed: 1.5 mm/min). Data were statistically analyzed using two-way ANOVA and Tukey's test ($\alpha = 0.05$).

Results. The mean flexural strength values for the ceramic–gold alloy combination (55 ± 7.2 MPa) were significantly higher than those of the ceramic–Ti cp combination (32 ± 6.7 MPa) regardless of the fatigue conditions performed ($p < 0.05$). Mechanical and thermo-mechanical fatigue decreased the flexural strength results significantly for both ceramic–gold alloy (52 ± 6.6 and 53 ± 5.6 MPa, respectively) and ceramic–Ti cp combinations (29 ± 6.8 and 29 ± 6.8 MPa, respectively) compared to the control group (58 ± 7.8 and 39 ± 5.1 MPa, for gold and Ti cp, respectively) ($p < 0.05$) (Tukey's test). While ceramic–Ti cp combinations failed adhesively at the metal–opaque interface, gold alloy frameworks exhibited a residue of ceramic material on the surface in all experimental groups.

Significance. Mechanical and thermo-mechanical fatigue conditions decreased the flexural strength values for both ceramic–gold alloy and ceramic–Ti cp combinations with the results being significantly lower for the latter in all experimental conditions.

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1. Introduction

Ceramic-fused-to-metal (CFM) fixed-partial-dentures (FPDs) combine the high esthetic properties of ceramic materials and the mechanical strength of metallic frameworks. Thus, oral rehabilitation could be achieved within an esthetic and functional context, depending on the materials selected. Clinically, one of the main problems in CFM FPDs is often related to the adhesion between the different ceramic veneer and the metallic frameworks leading to fracture of the ceramics with or without metal exposure [1].

Commercial pure titanium (Ti cp) has been the material of choice in several disciplines in dentistry due to its biocompatibility, resistance to corrosion and mechanical properties similar to those of gold alloys [2]. Despite these favorable characteristics, Ti cp casting for prosthetic purposes has not been viable for many years since casting procedures led to the formation of an undesirable crust resulting in high reactivity and fragility [3]. This coating is called "α-case" and is formed by incorporation of the elements from the investment that may impair the adhesion between Ti cp and ceramics [4,5]. In order to allow the utilization of titanium, specific equipment was developed for casting, refractory investments were manufactured that were different from the conventional silica-phosphate investments, and ultra-low temperature ceramics were produced to be able to fire them on cast Ti cp [6]. Only then Ti cp was indicated for the fabrication of removable and FPDs [7].

Despite the recent technological advances, the fabrication of CFM FPDs using Ti cp as a framework is still considered more complex and sensitive when compared to gold alloys. Gold alloys are approved in dental applications owing to their chemical–mechanical properties, which also allow adequate adhesion of ceramics onto them [8]. However, the increased cost of alloys with high gold content limits their use in the clinical practice today and therefore they are being replaced by alternative alloys.

When dental restorations are cemented and exposed to the oral environment, several factors may limit their service life since dental materials may undergo physicochemical alterations. The incidence of repeated forces during chewing results in stress concentration, and thermal variations induce fatigue of the materials themselves and/or the interface between them. Thus, some authors have proposed several testing methodologies such as thermal or mechanical cycling procedures in order to simulate the oral conditions prior to mechanical testing [9–11].

Therefore, the objective of this study was to verify the effect of thermal and/or mechanical cycling for induction of fatigue and then to quantify the flexural strengths of ceramic–metallic framework combinations cast in gold alloy or Ti cp.

2. Material and methods

Two metal alloys indicated for the fabrication of CFM FPD frameworks, namely gold alloy (Olimpya-Jelenko, Heraeus Kulzer, Hanau, Germany) and Ti cp (Tritan, Dentaurum JP Winkelstroeter KG, Pforzheim, Germany), were used for the experiments.

2.1. Fabrication of metallic frameworks

Rectangular acrylic templates (26 mm × 3 mm × 0.5 mm) were used for the fabrication of the frameworks. Wax sprues (Horus, Herpo Produtos Dentários Ltd., São Paulo, Brazil) were attached perpendicular to one end of the template and were connected to a central wax rod of 5 mm diameter (Wax Wire for Casting Sprues, Dentaurum, Pforzheim, Germany). The assembly was mounted in a silicone ring and poured with investment material (Rematitan Ultra, Dentaurum JP Winkelstroeter KG, Pforzheim, Germany) mixed at a ratio of 100 g powder to 14 ml liquid. The investment material for gold alloy (Bellavest SH, Bego, Bremen, Germany) on the other hand, was mixed at a ratio of 100 g powder to 25 ml liquid, according to the manufacturer's instructions. After the investment material had set, the silicone ring and the sprue former were separated from the investment mold.

Metallic frameworks were cast in Ti cp ($n = 48$) in an electrical induction furnace (Rematitan Autocast, Dentaurum) under argon gas. Elimination of sprues and separation of metallic strips were performed with the aid of carbide discs at low speed. Gold alloy frameworks ($n = 48$) were cast in a vacuum-pressure casting machine. After removal from the investment material, the margins of the frameworks were trimmed to the dimensions of 25 mm × 3 mm × 0.5 mm. The surfaces of the specimens that would receive the ceramics were airborne particle-abraded with 150 μm aluminum oxide (Korox, Bego) at an angle of 45° for 10 s from a distance of approximately 2 cm, under 2 bar pressure. Frameworks were then ultrasonically cleaned in isopropyl alcohol (Vitassonic II, Vita, Bad Sackingen, Germany) for 5 min and allowed to dry at room temperature.

2.2. Application of ceramic layer

An ultra-low temperature ceramic system (Titankeramik, Vita Zahnfabrik, Bad Sackingen, Germany) adequate for utilization on Ti cp, and a ceramic system (Omega 900, Vita Zahnfabrik) that is indicated for gold alloys were applied on the air-abraded surfaces of the metallic frameworks.

An area of 8 mm × 3 mm was initially marked on the Ti cp frameworks with a graphite pencil. Then the bonder (Titankeramik, Paste Bonder, Vita) was applied in a thin layer with a brush. After firing, opaque was applied on the bonder by pulverization of powder (opaque ceramic) and liquid (Spray-On, Vita), homogenized in a container that was connected to a dispenser (Spray-On). The thickness of the ceramic layer corresponding to dentin ceramic was standardized by positioning the frameworks in a metallic template (height: 1 mm). After removal from the assembly, ceramic was fired. Due to shrinkage, a second layer was applied and finally the specimens were submitted to a final glaze firing.

The gold alloy frameworks were first subjected to degassing and after cooling the margins of an area of 8 mm × 3 mm were marked on the frameworks with a graphite pencil. The powder and liquid of the opaque were mixed with a brush and applied on the marked area. After firing and cooling the opaque, each framework was positioned in the metallic template for appli-

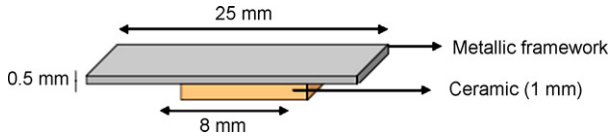

Fig. 1 – Final shape and the dimensions of the ceramic–alloy specimen.

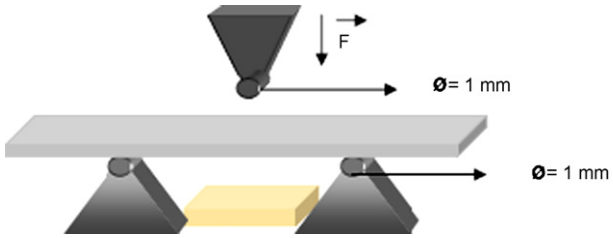

Fig. 2 – Schematic drawing of the rod with 1 mm diameter tip in relation to the pier that induced 50 N load for 20,000 times, with a frequency of 1 cycle/s.

cation of the ceramic layer that was then fired, corrected and glazed. The final shape and the dimensions of the specimens are illustrated in Fig. 1.

Twelve specimens for each ceramic–alloy combination were randomly divided into four subgroups and subjected to either thermal or mechanical cycling alone or a combination of the two or only stored in distilled water for 24 h at 37 °C (control group) prior to the flexural strength test.

2.3. *Thermal cycling*

Thermal cycling was performed for 3000 cycles between 4 and 55 °C in deionised water (Nova Etica, São Paulo, Brazil). The dwelling time at each temperature was 10 s and the transfer time from one bath to the other was 5 s.

2.4. *Mechanical cycling*

Mechanical cycling of the specimens was carried out in a mechanical cycling machine (custom made, Paulista State University, Dental School, UNESP, Sao Jose dos Campos, Brazil) that was developed to simulate the mechanical forces generated during the chewing cycle [12]. The device employed for this test was composed of two bases, at 2 cm distance from each other, on which cylinders (radius: 1.0 mm) were placed to allow positioning of specimens parallel to the ground and perpendicular to the axial load (Fig. 2). An upper rod with a 1 mm diameter tip was fixed on the pier that induced a 10 N load for 20,000 times at a frequency of 1 cycle/s. The testing device was placed on the machine base which contained a thermostat to allow testing in aqueous medium at a constant temperature of 37 °C. The formula according to the guidelines of ISO 9693 (1999) [13] was followed for the calculation of the data obtained from the flexural strength test.

The flexural tests were performed in a universal testing machine (Instron 4301, Instron Corp., Norwood, MA, USA) where the load was applied at a constant speed of 1.5 mm/min until fracture occurred. The load that led to initial separation of materials was obtained in kilogram force (kgf) and converted to Newtons (N) for the calculation of flexural strength according to the following equation where "P" was the maximum load upon fracture (N), "l" the distance between two supports (mm), "*b*" width and "*d*" thickness of the specimen (mm) by application of the following equation:

$$\text{flexural strength (MPa)} = \frac{3Pl}{2bd^2}$$

Specimens were analyzed under a stereomicroscope (Stemi 2000-C, Carl Zeiss, Gottingen, Germany) and the image was digitally recorded with a camera (Model MC 80DX, Nikon, Tokyo, Japan) connected to the microscope for the characterization of the metal surfaces and the failure modes. Some representative specimens were also observed under scanning electron microscopy (SEM) (JEOL-JSM-T330A, Jeol Ltd., Tokyo, Japan).

2.5. *Statistical analysis*

Statistical analysis was performed using SAS System for Windows, release 8.02/2001 (Cary, NC, USA). The means of each group were analyzed by two-way analysis of variance (ANOVA), with the flexural strength test as the dependent variable and the ceramic–metal combinations and fatigue conditions as the independent factors. *p* values less than 0.05 were considered to be statistically significant in all tests. Multiple comparisons were made by Tukey's adjustment test.

3. Results

The results of two-way analysis of variance (ANOVA) for the experimental conditions are presented in Table 1.

The mean flexural strength values for the ceramic–gold alloy combination (54.7 ± 7.2 MPa) were significantly higher than those of the ceramic–Ti cp combination (32.2 ± 6.7 MPa)

Table 1 – Results of two-way analysis of variance for the experimental conditions (*$p < 0.05$)

Source of variation	Sum of squares	Degrees of freedom	Mean ratio square	Probability	
				F	*p*
Metal	10093.50	1	10093.50	248.30	0.0001*
Cycling	763.90	3	254.60	6.26	0.001*
Metal × cycle	71.80	3	23.90	0.59	0.624
Error	2926.80	72	40.60		
Total	13856	79			

Table 2 – The mean (S.D. = standard deviations) flexural strength values (MPa) for ceramic–gold alloy and ceramic–Ti cp combinations after 24 h water storage (control), thermal (T), mechanical (M) and thermo-mechanical (T + M) cycling

Metal type	Experimental condition	Bond strength (MPa)*
Au	Control	58.011[a]
	T	56.356[a]
	M	52.483[a]
	T + M	51.980[a]
Ti	Control	38.525[b]
	T	31.884[b,c]
	M	29.730[b,c]
	T + M	28.831[c]

* The same superscripted letters indicate no significant differences (Tukey's test, $\alpha = 0.05$).

regardless of the fatigue conditions performed ($p < 0.05$) (Table 2).

Mechanical and thermo-mechanical fatigue conditions decreased the flexural strength results significantly for both ceramic–gold alloy (52 ± 6.6 and 52.5 ± 5.6 MPa, respectively) and ceramic–Ti cp combinations (29.7 ± 6 and 28.8 ± 7 MPa, respectively) compared to the control group (58 ± 7.8 and 38.5 ± 5.1 MPa, respectively) where the tests were performed after 24 h water storage at 37 °C ($p < 0.05$) (Tukey's test). The interaction effect between the two fatigue variables was not statistically significant when the ceramic–metal combinations and fatigue conditions were considered individually ($p = 0.624$).

SEM analysis at 1000× magnification, complementary to the flexural strength tests, showed adhesive detachment of the ceramic from the Ti cp frameworks with the most superficial metallic layer seeming to have been removed from the surface yielding an irregular surface with acute angles (Fig. 3a). In stereomicroscope images at 50× magnification, ceramic remnants were also not noticed (Fig. 3b). On the other hand, gold alloy frameworks exhibited a residue of ceramic material on the surface exclusively in all experimental groups (Fig. 4a). On stereomicroscope images (50×), clearer regions confirmed the presence of ceramic remnants on the surface (Fig. 4b).

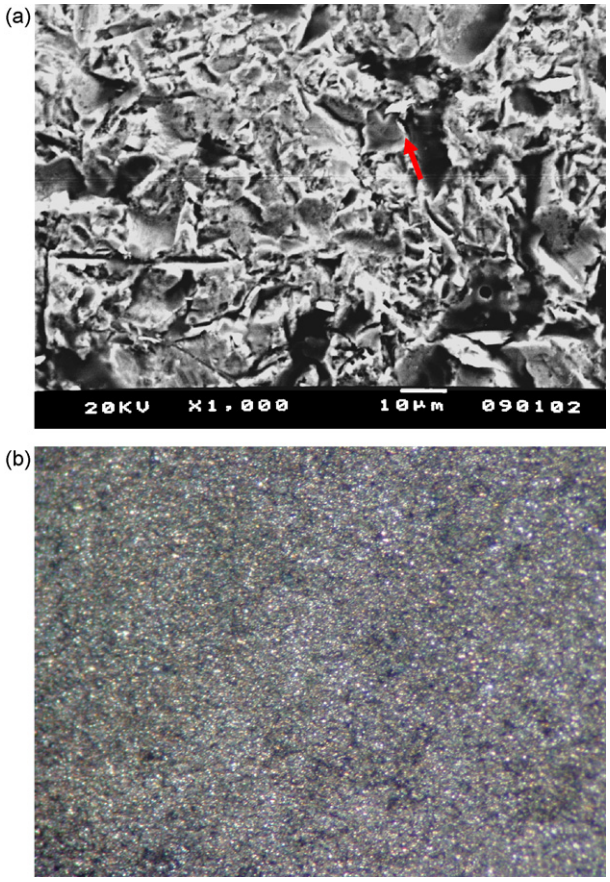

Fig. 3 – (a) Representative SEM micrographs of the ceramic–Ti cp surface after flexural strength testing (1000× magnification). Note the irregular surface with acute angles. (b) Stereomicroscope images (50×) also showing no ceramic remnants on the Ti cp surface.

4. Discussion

Although it is often difficult to predict the metal types, clinical findings indicated that CFM FPD failures are frequently experienced in the form of ceramic fractures with metal exposures [1,14]. In this study, the ceramic–gold alloy combination presented statistically higher mean values than those of ceramic–Ti cp combinations. This finding is in agreement with several other studies [15–17] where better outcomes were also observed with the use of noble alloys compared to other alloys. The mean for the ceramic–Ti cp group, despite being lower compared to the other group, was superior to the minimum of 25 MPa suggested by ISO [13]. Several factors involved in the complex manufacturing process for casting and investing of Ti cp that require improvement, could be the reasons for lower results in this group. Some equipment was developed that allowed casting of Ti cp in the absence of oxygen. Despite the utilization of argon gas instead of oxygen, Miyakawa et al. [3] identified the presence of four layers after Ti cp casting and the authors advocated removal of the two first layers before application of the ceramic layer. In this study, elimination of the superficial layer was performed by the use of a carbide bur at low speed in order to eliminate the α-case layer. However, this procedure is operator driven by the dental technicians in the laboratories and it can never be certain that the α-case layer is completely removed.

The composition of the investment material and casting procedures also influence the formation of the α-case layer. For this reason, some authors advocated the use of Mg-based [18] and others suggested Zr-modified Mg-based investment materials [19]. Addition of these elements increases the thermal expansion coefficient of the investment material and limits its interfacial reactivity with Ti cp at high temperatures. In this study, the investment material used was in compliance with the manufacturer's recommendations. The pressure at which liquid titanium is injected into the investment [18], and the temperature at which this procedure is performed, could also influence the consequential adhesion of the ceramics to Ti cp [20]. Within this context, ceramics were developed to make

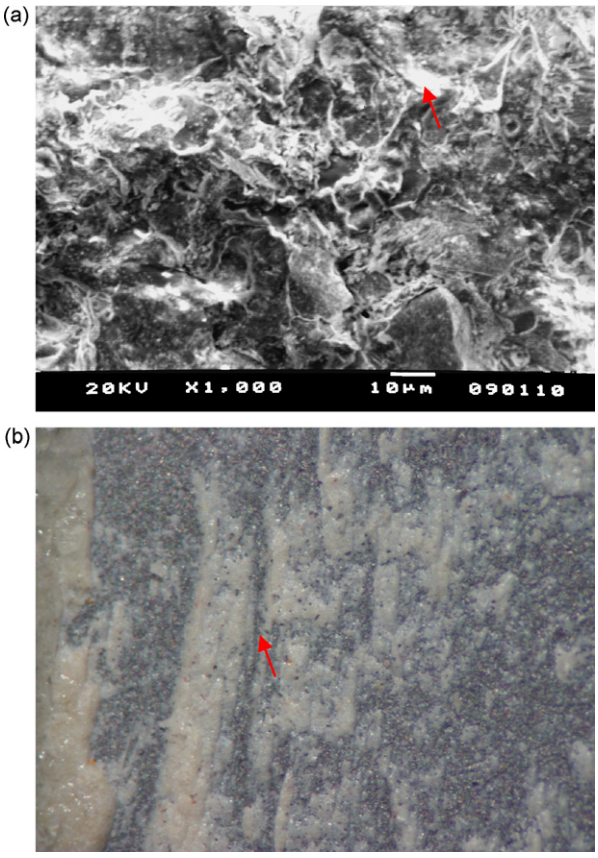

Fig. 4 – (a) Representative SEM micrographs of the ceramic–gold alloy surface after flexural strength testing (1000× magnification) with the arrow indicating ceramic residues, (b) Stereomicroscope images (50×) confirmed the presence of remnants of ceramic on the surface.

their firing possible on Ti cp at temperatures below 800 °C in order to prevent the excessive formation of oxides on the metallic framework surface [7].

Mechanical tests such as shear strength test, three- or four-point bending test and biaxial flexural test, could be applied to compare or quantify the bond strength between the ceramic and the metal. The test selected for this study was the flexural strength test as recommended by ISO guidelines for testing metal–ceramic combinations [13]. Fischer [21] considered the flexural strength test as a sensitive method for the measurement of the bond strength between the ceramics and metals due to the complexity of casting acrylic patterns without the presence of residues, internal and external defects and especially without dimensional alterations. The thickness (0.5 mm) desired for the test specimens was difficult to achieve. During framework preparation for the experiments, a higher failure rate in the casting process occurred with Ti cp than with the gold alloy, most probably due to its low specific weight (4.5 g/cm^2).

Microscopic observations at low magnification (50×) showed residual ceramic and the opaque layer in the ceramic–gold alloy group. On the other hand, when Ti cp surfaces were evaluated, the frameworks were found to be free of ceramic remnants, presenting a grayish shade. Kimura et al. [4] stated that the formation of a metallic oxide layer on Ti cp does not adhere to the substrate and thus fracture is regarded as adhesive between the metal or metallic oxide layer and the ceramic. Even though aggressive testing conditions were not considered, the absence or small amounts of residual ceramic on Ti cp have also been monitored in previous studies [11,22,23].

Most in vitro experiments are performed using static mechanical tests in dental research that do not address the aggressive oral environment. It is known that the oral environment is able to induce physicochemical alterations to the dental materials. Temperature alterations provide conditions for the occurrence of degradation in the aqueous environment [24] and also encourage mechanical fatigue of materials themselves or their interfaces, triggered by the repeated incidence of chewing loads [25,26]. Water storage of ceramic materials for instance, decreases their mechanical properties [9]. The reduction may be related to the solubility of different oxides and this process may be higher in ceramics designed for use with Ti cp because of the presence of alkaline metallic oxides [24]. Previous studies investigating the adhesion between metal and laboratory resin or metal and ceramics disclosed reduced adhesion after thermal cycling [2,11,27–29]. The exposure of CFM FPDs to temperature alterations during immersion in water induces repeated tensions that weaken the adhesion of materials due to the mismatch between the thermal expansion coefficient of the restoration components. All groups in the present study, submitted to thermal cycling only, presented reduced resistance to fracture, yet without statistically significant differences compared to the other group of the same material that was not submitted to thermal cycling. The study of Tróia Júnior et al. [23] investigated the same thermal variation and metallic substrate (Ti cp) and also did not find any influence of thermal cycling on the adhesion of ceramic to this metal. Probably, this factor would be more significant in more drastic conditions than those employed in the study of Shimoe et al. [30] who performed extended thermal cycling (100,000 cycles) and observed a 30% reduction in mean bond strength. This study concentrated more on the effect of mechanical cycling alone and the combined effect of thermal and mechanical cycling, thus extended thermal cycling was not performed.

According to Scherrer et al. [25], all materials and their combinations should be submitted to fatigue conditions before mechanical testing is performed. Induction of mechanical fatigue or computer models, are helpful means in the estimation of predictability of restorations in order to avoid catastrophic failures in vivo [26]. Some factors inherent in mechanical cycling may also influence the outcomes, namely the nature of the material, number of cycles, liquid medium present, load and frequency applied. Mechanical cycling in this study was performed in the presence of water at a constant temperature of 37 °C and frequency of 1.3 Hz to simulate the chewing cycle as closely as possible [31].

Encouraged by the favorable biological and mechanical properties of commercially available Ti cp, some investigators have been conducting clinical and laboratory research. Over a 5-year period, a clinical follow-up study by Lovgren et al. [32] for ceramic-veneered titanium restorations with the Procera (Ti cp) system proved favorable regarding its perfor-

mance in the oral environment. Ti cp may alter the utilization of other metals in dentistry since it is a material with absolute biological compatibility and a wide spectrum of employment in prosthetic dentistry. However, only an improved technical process will increase the longevity of Ti cp based CFM FPDs.

5. Conclusions

Based on the results of this study, the following conclusions can be drawn:

1. The mean flexural strength values for the ceramic–gold alloy combination were significantly higher than those of the ceramic–Ti cp combination regardless of the fatigue conditions performed.
2. Mechanical cycling for 20,000 times and thermal cycling for 3000 times followed by mechanical fatigue conditions reduced the flexural strength results significantly for both ceramic–gold alloy and ceramic–Ti cp combinations compared to the control group where the tests were performed only after 24 h water storage at 37 °C.
3. Microscopic analysis of the specimens after flexural strength test showed complete adhesive detachment of the ceramic from the Ti cp frameworks exclusively, indicating the weakest interface of the assembly was located between the Ti cp framework and its oxide layer.

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