

## Bond Strength of Two Ceramic Systems to Two different Bases Alloy Metals

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Received March 5, 2021.

Accepted for publication on April 21, 2021.

Published May 20, 2021.

### Abstract

**Background** The success and longevity of metal-ceramic restoration depend on metal-ceramic bond strength. A variety of ceramic and alloy systems are available nowadays.

**Objectives** the present study aims to evaluate and compare the bond strength of two ceramic systems to Ni-Cr and Co-Cr alloys. **Materials and Methods** A study sample of forty metal specimens was prepared. Twenty specimens were fabricated from Ni-Cr alloy, and twenty of Co-Cr alloy with dimensions (25× 3× 0.5 mm) according to ISO 9693. These are layered with two ceramic systems (Vita ceramic Vita VMK Master), and (IPS d.SIGN Ivoclar) with a dimension of (8×3×1.1 mm). Bond strength was evaluated by a three-point bending mechanical test. After testing the mode of failure was recorded. Scanning electron microscopy (SEM), and (EDS) analysis were performed. Results were analyzed using SPSS with descriptive statistics and a Student t-test.

**Results** The results revealed that the type of alloy used had a significant effect on bond strength ( $p < 0.01$ ). The Co-Cr alloy had significantly higher means compared to the Ni-Cr alloy. While the ceramic systems had a non-significant effect ( $p > 0.05$ ).

**Conclusions** The Co-Cr alloy showed significantly higher bond strength with both ceramic systems in comparison to Ni-Cr alloy. All groups tested had bond strength higher than that demanded by (ISO 9693) with a minimum of 25 Mpa.

**Keywords:** Alloy; ceramics; metal-ceramic bonds; metal-ceramic alloy.

### Introduction

Although there are great progress and growing use of all-ceramic restoration in fixed prosthodontics, metal-ceramic restoration is still considered an important part of dentistry. This is due to their excellent mechanical properties, durability, and fit of the metal with the superior aesthetic appearance of porcelain (Abrisham et al, 2017; Yoldan et al, 2020). In the

production of the metal framework of metal-ceramic restoration, the alloy should have adequate properties such as biocompatibility, strength, ease to manipulate, and reasonable cost (Al Amri and Hammad, 2012). Base metal alloys such as cobalt-chromium alloy (Co-Cr) and nickel-chromium (Ni-Cr) alloys have satisfactory properties such as high modulus of elasticity, low density, rigidity and low cost to be

used in the construction of metal-ceramic restoration as a good metallic choice instead of noble alloy (Sipahi and Özcan, 2012). Ni-Cr alloys were used in fixed prosthodontics because of their acceptable cost as compared to noble alloys. However, there is many health problems have been detected as a result of long-term use of these alloys. An allergic response is one of the problems faced by some patients, also the technician who was dealing with these materials during his work. Later, Co-Cr alloys were developed as a good option that is used for a patient who is sensitive to nickel (Al Jabbari, 2014; Singh et al, 2017). Controversy exists between studies about bond strength of base metal alloys and available ceramic systems (De Melo et al, 2005; Mirković, 2007). The Co-Cr alloy is characterized by good bond strength whether fabricated by casting or other production techniques (Lawaf et al, 2017; Dentalnega et al, 2019). The opaque layer is critical for the success of bond strength since it interacts with the oxide layer of the metal to form a primarily bonding mechanism (Huang et al, 2005). A chemical bond is attained through the oxide layer (Hamza et al, 2019). For this reason, more attention should be given to the selection of porcelain and metal combinations (Al Amri and Hammad, 2012). Mechanical interlocking that determined an engagement of ceramic into the undercut that formed on the metal surface while the compressive force was entirely dependent on the coefficient of thermal expansion between metal and ceramic and Vander walls, forces that depended on the molecular interaction of metal and ceramic charges (Sipahi and Özcan, 2012). Mechanical interlocking created on metal bonding surface by sandblasting with aluminum oxide and improve the wettability of opaque ceramic (Fernandes Neto et al, 2006; Külünk et al, 2011). However microstructural analysis of metal-ceramic interface reported the

presence of voids in ceramic layer even if the operators strictly followed manufacturer instructions (Massimi et al, 2011). These voids affect greatly the longevity of the restoration. Various testing methodologies were indicated to evaluate the metal-ceramic bond strength. The three-point bending test according to ISO 9693 is considered an ideal test for the evaluation of metal-ceramic bond strength because specimens are under traction, compression, and shear force at the same time (Korkmaz and Asar, 2009; Li et al, 2017). The design of the three-point bend test with a flat specimen more accurate than an anatomical tooth-contoured specimen with fewer experimental errors (Bae et al, 2015). Previous investigation of metal-ceramic restoration bond strength found no standard behavior exists among metal-ceramic systems (Korkmaz and Asar, 2009) and others recommend need to investigate bond strength of various metal alloy types and ceramic systems developed (Rathi et al, 2011). Therefore, the present study aims to evaluate and compare the bond strength of two ceramic systems (Vita ceramic Vita VMK Master) and (IPS d.SIGN Ivoclar) to Ni-Cr and Co-Cr alloys.

## Materials and Methods

### Preparation of metal specimens

Forty rectangular metal specimens were prepared from Ni-Cr (Kera-NH, Germany) and Co-Cr (Keromit Np, Italy) alloys and divided into two groups. These specimens were made according to ISO 9693 with the dimensions of  $25 (\pm 1) \times 3 (\pm 0.1) \times 0.5 (\pm 0.05)$  mm. Then these specimens are covered by two different types of ceramic layer Vita VMK Master (Vita, Germany) and IPS d. SIGN (Ivoclar Vivadent, Germany), the dimension of the ceramic layer was of  $(8 \times 3 \times 1.1 \text{ mm})$  Wang et al, (2016) as shown in Figure (1). To fabricate the metal alloy specimens, casting wax strips with the dimensions of  $25 (\pm 1) \times 3 (\pm 0.1)$

× 0.5 (±0.05) mm were cut from green casting wax (Renfert, Germany). Wax patterns were invested in a phosphate-bonded investment (Bellavest SH, Bego, Germany). After the investment had been set, the ring was placed in a burnout furnace (Degussa, Germany). After removal from the furnace, all specimens were cast in a centrifugal casting machine (Degussa, Germany), divested, and then sandblasted for removal of residual investment material. Then specimens were ultrasonically cleaned for 5 min in distilled water using an ultrasonic cleaner unit according to the manufacturer's instructions (Degussa, Germany). After that, all specimens were subjected to an oxidation procedure. The oxidation cycle for Ni-Cr alloy specimens required heating at a rate of 980°C under vacuum, and holding temperature for 5 min. While the oxidation cycle for Co-Cr alloy specimens required heating at a rate of 960°C under vacuum and holding temperature for 5 min according to the manufacturer's instructions. Following the oxidation procedure, the oxide layer on each metal specimen would be formed. Then all-metal specimens were sandblasted with aluminum oxide (Al<sub>2</sub>O<sub>3</sub>) of 250 µm particle size at air pressure (3-4) bar and distance of 10 mm for 15 sec (Patel et al, 2015; Li et al, 2017). Lastly, the specimens were cleaned using an ultrasonic cleaner unit for 10 min in distilled water to remove debris and greasy material and to avoid metal alloy from contamination before porcelain application.

### Sample grouping

The forty metal alloy specimens were divided into four groups:

1. Group A: n=10, Co-Cr specimens layered with Vita ceramic (Vita VMK Master Vita, Germany).
2. Group B: n=10, Co-Cr specimens layered with d.SIGN ceramic (IPS d.SIGN Ivoclar Vivadent, Germany).

3. Group C: n=10, Ni-Cr specimens layered with Vita ceramic (Vita VMK Master Vita, Germany).

4-Group D: n=10, Ni-Cr specimens layered with d.SIGN ceramic (IPS d.SIGN Ivoclar Vivadent, Germany).

### Porcelain application

Before porcelain application, the metal specimens must be cleaned thoroughly by the steam cleaner. To build up ceramic in required dimensions and according to ISO 9693-1:2019 specifications, a hollow rectangular custom-made Teflon mold is to build up the ceramic of 8× 3× 1.3mm dimension (Schweitzer et al, 2005). Before porcelain application, the Teflon mold is lubricated with porcelain special liquid using a brush. The Vita ceramic groups opaque (powder and liquid) applied in two layers to metal alloy specimens using a brush and fired individually under vacuum in a ceramic furnace (Vita- T 40, Germany) at a temperature of 960°C and holding time of 1min. Then dentin porcelain was mixed, vibrated applied with a brush. Excess water was removed with a clean tissue and fired at 930°C and a holding time of 1 min in a vacuum according to the manufacturer's instructions. While the-d. SIGN ceramic groups opaque (paste type with special opaque liquid) applied in two layers to metal specimens using a brush and fired individually under vacuum in a ceramic furnace (Vita- T 40, Germany) at a temperature of 900°C and holding time of 1min, and second opaque layer at a temperature of 890°C and holding time of 1min. Then dentin porcelain was mixed, vibrated applied with a brush. Excess water was removed with a clean tissue and fired at 870°C and a holding time of 1 min in a vacuum according to the manufacturer's instructions.

### Three points bending test

The prepared metal-ceramic specimens

were subjected to a three-point bending test by using a universal testing machine (Testometric 3-points test, Holland). Each specimen was positioned on specially fabricated metal supports (20 mm of distance) with the porcelain facing downward opposite to the applied load. The force was applied at the midpoint of the metal specimen at a crosshead speed of 1.5 mm/min according to ISO standard 9693 and recorded up to failure (Hammad and Talic, 1996; Wang et al, 2016). In this test, the force causing interfacial crack initiation at one of the two ends of the ceramic layer is measured and recorded digitally with a personal computer using software provided by the testing machine manufacture and then used in calculating the bond strength (Özüpek and Ünlü, 2012).

### Calculation of bond strength

The debonding strength/crack initiation for the materials that loaded in three-point flexure test configuration the following calculation formula was used  $\tau_b = k F_{fail}$  Where F was the fracture force or failure load (in Newtons) which measured for specimens failing by a debonding crack occurring at one end of the ceramic layer during the three-point bending test. The coefficient k is a constant that can be calculated numerically based on the flow chart in ISO 9693 standards with units of mm<sup>-2</sup>. The value of k depends on the modulus of elasticity and the thickness of specimens (Wang et al, 2016). The elastic modulus of the base metal alloy was obtained from the curve in ISO 9693 (Co-Cr alloy 300.16 GPa; and Ni-Cr 214.3 GPa).

### Mode of failure

Fractured specimens were observed under the stereo-microscope (MEIJI, Japan) at a magnification of 20X to categorize the type of failure after a three-point bending test.

Bond failure classified into three types, ad-

hesive, cohesive, and mixed-mode:

1. Adhesive mode: the failure occurs between metal and ceramic, there was no ceramic particle on the metal surface.
2. Cohesive mode: entirely within porcelain and there is a great fragment of the ceramic covered metal surface after the debonding.
3. Mixed-mode: a combination of adhesive and cohesive failure. When small remnants of ceramic were found in the metal (Akova et al, 2008).

### Scanning electron microscope (SEM) and elemental composition analysis

SEM analysis was applied for one specimen from each group after the failure of the ceramic veneer (Belkhode et al, 2019). The images were also observed under (Inspect S50, Netherlands) at 500X magnification to study the surface characteristic. This device provides energy-dispersive X-ray spectroscopy (EDS) which provides more details and explains the chemical element found at the metal-ceramic interface.

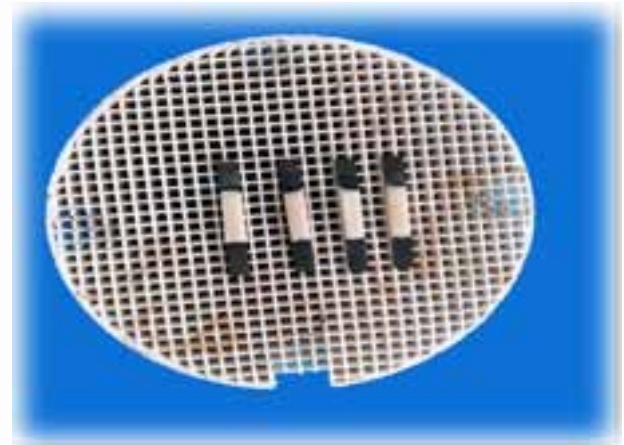
### Statistical analysis

Descriptive statistical analysis includes the mean, minimum, maximum, and standard deviations were calculated for bond strength in (MPa) for all tested groups. The student's t-test was used between tested groups to evaluate the effect of type of alloy and ceramic system on bond strength of metal-ceramic specimens. A p-value of  $\leq 0.05$  was considered statistically significant.

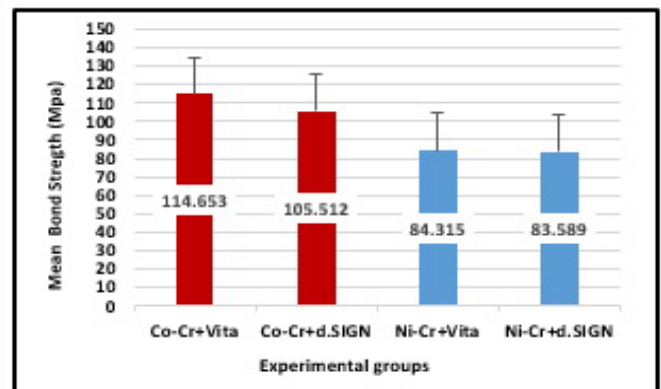
### Results

In figure (2) the highest mean value of bond strength in group A of (Co-Cr+Vita ceramic), followed by group B of (Co-Cr+d.SIGN ceramic), then group C of (Ni-Cr+Vita ceramic), and the lowest mean value of bond strength in group D of (Ni-Cr+d.SIGN ceramic). The student's t-test was used to evaluate the significance of differ-

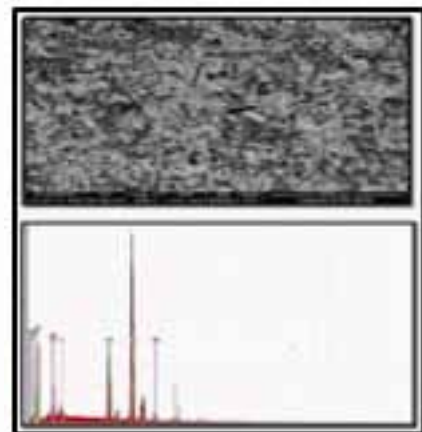
ence among the studied groups as listed in the table (1). It showed that there are no significant differences ( $p > 0.05$ ) noticed between Co-Cr+Vita and Co-Cr+d.SIGN ceramic and that between Ni-Cr+Vita and Ni-Cr+d.SIGN ceramic. However, highly significant differences ( $P < 0.01$ ) were noticed between Co-Cr+Vita and Ni-Cr+Vita ceramic and that of Co-Cr+ d.SIGN and Ni-Cr+ d.SIGN ceramic. For the mode of failure, Table (2) shows the frequency of failure mode. The Co-Cr+Vita and Co-Cr+d.SIGN ceramic showed 60% cohesive and mixed failure. However, Ni-Cr+Vita ceramic revealed 50% cohesive failure, and Ni-Cr+ d.SIGN ceramic 40% cohesive, and mixed failure. The SEM images showed different surface morphologies for specimens after the ceramic failure to metal. There is a white area that represented the adherent opaque ceramic on a metal surface (light due to its low electrical conductivity) and a black area represented the metal alloy (dark color due to its high conductivity) (Han et al, 2018). SEM images for groups Co-Cr+Vita and Co-Cr+d.SIGN ceramic as in figures (3) and (4) showed whiter and homogenous areas of opaque ceramic. While groups Ni-Cr+Vita and Ni-Cr+ d.SIGN ceramic as in figures (5) and (6) showed multiple areas of a dark spot of exposed metal with a white area of opaque porcelain. The dark areas of metal appeared smaller in size and the grayish areas represented mixed opaque and oxide layer (Wang et al, 2016). All specimens viewed with SEM showed cohesive failure through the body ceramic and an area of cohesive/adhesive failure appeared in the opaque /oxide layer. The EDS analysis revealed elemental penetration in the interfacial layer across the metal-ceramic interface. The EDS as in figures (3, 4, 5, and 6) illustrated the major elements of Co-Cr alloys, Ni-Cr alloy, and ceramic of Vita and d.SIGN as in table (3).



**Figure (1): Metal-ceramic specimen design for three-point bending test.**



**Figure (2): Bar chart showing the bond strength of the studied groups in Mpa.**



**Figure (3): SEM and EDS of Co-Cr+Vita Ceramic.**

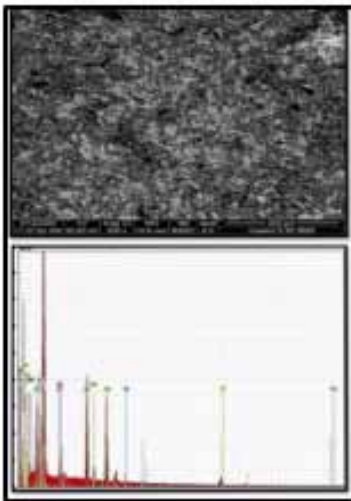


Figure (4): SEM and EDS of Co-Cr+d.SIGN ceramic.

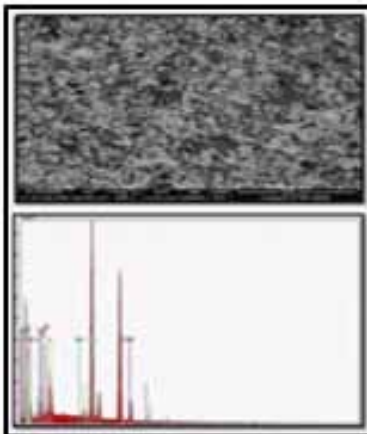


Figure (5): SEM and EDS of Ni-Cr+Vita Ceramic.

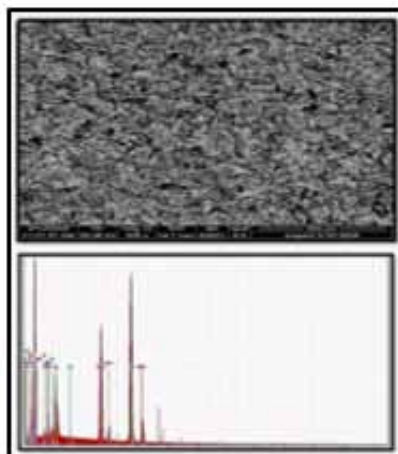


Figure (6): SEM and EDS of Ni-Cr+d.SIGN ceramic.

Table (1): Student's t- test between studied groups.

Studied groups	T	p-value	C.S
Co-Cr+Vita Vs Co-Cr+d.SIGN ceramic	2.165	.059	$P>0.05$ (NS)
Ni-Cr+Vita Vs Ni-Cr+d.SIGN ceramic	0.152	.883	$P>0.05$ (NS)
Co-Cr+Vita Vs Ni-Cr+Vita	6.070	.000	$P<0.01$ (S)
Co-Cr+d.SIGN ceramic Vs Ni- Cr+d.SIGN ceramic	3.048	.010	$P<0.01$ (S)

Table (2): Study mode of failure.

Groups	Adhesive failure		Cohesive Failure		Mixed Failure	
	NO	%	NO	%	NO	%
Co-Cr+Vita	4	40	4	40	2	20
Co-Cr+d.SIGN	4	40	5	50	1	10
Ni-Cr+Vita	5	50	5	50	0	0
Ni- Cr+d. SIGN	6	60	3	30	1	10

Table (3): Normalized weight percentage of each element on metal/ceramic interface.

Groups	Co- Cr+Vita	Co- Cr+d.SIGN	Ni- Cr+Vita	Ni- Cr+d.SI GN
Cr wt. %	14.54	12.83	27.22	20.10%
Co wt. %	65.27	15.96		
W wt. %	6.83	1.63		
C wt. %	4.24	7.05	3.87	8.03
Ni wt. %			30.27	46.11%
O wt. %	2.51	27.65	22.56	7.13
S wt. %	1.72		3.72	6.46
Si wt. %	1.33	5.04	2.94	2.11
Zr wt. %		16.37		
Al wt. %		4.49	4.09	0.69
P wt. %		3.40	0.64	0.96
In wt. %		1.93		
K wt. %		1.39		0.23
Na wt. %		1.15		0.87
Mn wt. %		0.38		0.47
Ba wt. %			2.37	
Ta wt. %			2.13	6.77

## Discussion

Variable numbers of metal alloys and ceramic systems are commercially available. It's important to predict the success of the compatibility of various metal alloys and ceramic systems. The clinical durability of metal-ceramic restorations depends first of all on the quality of bond strength between metal alloy and ceramic veneer (Korkmaz and Asar, 2009). Various tests had been designed by scientists to evaluate metal-ceramic bond strengths. The three-point bending test that was used in the present study was suggested by (Lenz et al, 1995). This test provides simulation to the clinical condition because the specimens are under compression, shear bond strength, and traction at the same time. The easy fabrication of the specimens was another cause for using the three-point bending test in measuring the bond strength of metal-ceramic restoration, the specimens were easy to fabricate and valued (Korkmaz and Asar, 2009). The nickel-chromium and cobalt-chromium base metal alloys were used in this study in fabrication metal specimens as an alternative to the precious metal alloys since they are more biocompatible and have good properties such as corrosion resistance, low in density, and cost (Sipahi and Özcan, 2012). Another advantage of base metal alloy is their high modulus of elasticity, which allows the production of very rigid metal construction that resists delamination and provides good support to the applied ceramic (Mirković, 2007). There are mainly four factors necessary in forming metal-ceramic bond strength which included chemical bonding, mechanical interlocking, Vander Waals forces, and compressive forces which depend on the coefficient of thermal expansion (Schweitzer et al, 2005). The chemical bonding that formed from the chemical interaction of ions and diffusion within the metal-ceramic interface by forming

covalent and metallic and ionic bonds between metal and ceramic accompanied by the oxidation of the metal alloys (Sipahi and Özcan, 2012; Wang et al, 2016). Mechanical interlocking in the present study was created with sandblasting of all-metal specimens with Al<sub>2</sub>O<sub>3</sub> with large particle size (250 µm) recommended by Patel et al, (2015) were reported that better bond strength can be achieved when the metal surface sandblasted with a large particle size as (250µm). The results revealed that all specimens of the studied groups had higher bond strength than the minimum value of bond strength of 25 MPa as required by the ISO 9693. Nevertheless, it revealed significantly higher bond strength ( $p < 0.01$ ) in Co-Cr alloy groups (Co-Cr+Vita and Co-Cr+ d.SIGN ceramic) compared to Ni-Cr alloy groups (Ni-Cr+Vita and Ni-Cr+ d.SIGN ceramic) in figure (2), and table(1). For many reasons for these findings, the EDS analysis of Ni-Cr groups revealed increased percentages of Cr content (27.22, and 20.10) groups C and D respectively compared to the Co-Cr alloy groups table (3). This means thicker oxide layer formation in Ni-Cr alloy groups increases the risk of metal-ceramic bond failure (Huang et al, 2005). Another important causative factor high modulus of elasticity of these Co-Cr alloy which leads to an increase in the rigidity and resistance to the delamination, also, reduce the possibility of bending under load, thus leading to stronger bond strength and providing good support to the ceramic (Han et al, 2018). The finding of this study may in agreement with the study by Mirković, (2007). Another reason for this significant difference between Ni-Cr and Co-Cr base metal alloy groups were the different chemical composition of these two alloys which causes consequently variation in the mechanical properties of the alloys and the bond strength values (Yoldan et al, 2020). These results conflicted with De

Melo et al, (2005), who concluded that no significant differences in the shear bond strength of Ni-Cr and Co-Cr alloy were studied. This may be due to differences in the testing procedure and preparation of specimens, the former study may not perform proper oxidation of the metal and sandblasting with aluminum oxide size of 100 $\mu$ m (De Melo et al, 2005). The ceramic systems investigation have a different form of opaque porcelain, the Vita ceramic used powder and liquid, while the d.SIGN from Ivoclar provides the opaquer in paste form. The present study showed non-significant differences as shown in table (1) between two types of ceramic systems regarding bond strength ( $p > 0.05$ ). This may disagree with Al Amri and Hammad due to different types of metal alloy used in their study, tests, and particle size of aluminum oxide for sandblasting the metal specimens (Al Amri and Hammad, 2012). Mode of failure of metal-ceramic specimens in the present study demonstrated 60% of cohesive and mixed failure in Co-Cr groups that associated with significantly higher bond strength. However, Ni-Cr groups revealed 50% and 40% cohesive and mixed failure. These findings could be in agreement with Al Amri and Hammad who suggested that cohesive failures might be an indication of strong metal-ceramic bond strength (Al Amri and Hammad, 2012). However, other researchers found no direct relationship between bond strength and failure types (Wang et al, 2016) concerning the experimental procedure and investigated materials.

### Conclusions

According to the three-points bending test of the evaluated experimental groups, a higher bond strength value was indicated than the minimum value required by ISO 9693. Also, the Co-Cr alloy with two ceramic systems has higher bond strength than that of Ni-Cr alloy. Furthermore, the mode

of failure in Ni-Cr was more frequently adhesive, while is more cohesive in Co-Cr alloy which is illustrated by the higher bond strength of Co-Cr alloy. Therefore, and according to these study findings, the Co-Cr alloy could be recommended for long-span metal-ceramic restoration than Ni-Cr alloy.

### Acknowledgments

The authors would like to thank Mr. Saad Gaeed Jbara and Mr. Ahmed Basim Naji Al-Daboo at «Saad Dental Lab» for their technical assistance during specimen manufacturing.

### Conflict of interest

We are the author's (Samah Khaleel Radhi and Suha Fadhil Dulaimi) state that the manuscript for this paper is original, and it has not been published previously and it's part of MSc. dissertation and is not under consideration for publication elsewhere, and that the final version has been seen and approved by all authors.

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