

12-10-2022

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### Recommended Citation

Manne, Prakash and Ravuri, Kavya (2022) "Evaluation of porcelain to metal bond strength on use of recast nickel-chromium alloy," *Manipal Journal of Medical Sciences*: Vol. 7: Iss. 1, Article 10.

Available at: <https://impressions.manipal.edu/mjms/vol7/iss1/10>

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# Evaluation of porcelain to metal bond strength on use of recast nickel-chromium alloy

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## Abstract

**Purpose:** The objective is to assess how the bond between metal and ceramic is impacted by recasting the base metal alloy. **Materials and Methods:** A total of 78 metal-ceramic specimens were created using beryllium-free nickel-chromium alloy from Dentalloy International Pvt Ltd. There were 26 specimens in each of the three groups, and they were all prepared differently. The first group was cast from 100% fresh alloy and served as the control. The second group was made by mixing 50% used (once cast) alloy and 50% fresh alloy. The third group was cast using 100% reused (once-cast) alloy. All of the alloy specimens from the three groups were then bonded to porcelain using VMK 95 Metall Keramik from Vita Zahnfabrik, Bad Sackingen, Germany. After the bonding process, the samples were tested for their shear bond strength. To test the weight percentage of each element, ten fractured specimens from each group were subjected to microanalysis. The bond strength data was analyzed using one-way ANOVA followed by the Tukey multiple comparison test. **Results:** The study results support the use of once-cast alloys for metal-ceramic restorations. The bond strength of these specimens was found to be significantly higher at  $37.67 \pm 8.65$  MPa as compared to those made from fresh metal ( $25.43 \pm 7.34$  MPa). The SEM (scanning electron microscope) images of the fracture surface revealed a mixed mode of failure with visible porcelain fragments and the microstructure analysis of the fractured surfaces showed a remarkable decrease in the oxides of Ni, Cr, Mn, and Mo in the recast specimens. **Conclusion:** These findings strongly suggest that once-cast alloys can be safely used without compromising the strength of the porcelain-metal bond.

**Keywords:** Recasting, Base-metal alloy, oxide layer on recast metal, ceramic, electron probe microanalysis, metal-ceramic bond strength, nickel-chromium.

## Introduction

The key factor for successful metal-ceramic prostheses is the establishment of a long-lasting bond between the alloy and porcelain [1]. To create a strong chemical bond, it is crucial to form a stable oxide layer between the metal and porcelain [2]. The bond's nature, strength, and performance in the oral environment all impact the porcelain's ability to withstand fracture during the restoration's clinical use [3]. The reuse of previously cast metal is a

widespread practice employed by dental laboratories to reduce the unit cost of a fixed partial denture. However, this procedure can lead to a change in the metal oxide composition present at the surface of the dental alloy, which may have a crucial impact on the metal-ceramic bond [2]. Several studies published in the literature have assessed the effect of recasting on the metal-ceramic bond with high-noble and noble alloys [4, 5]. Nonetheless, additional information is required regarding the influence of recasting base metal alloys on the strength of the metal-ceramic bond. The primary objective of this study is to evaluate the influence of recasting on the bond strength between nickel-chromium alloy and dental ceramic.

## Materials and Methods

78 metal-ceramic specimens were prepared using a beryllium-free nickel-chromium alloy by Dentalloy

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Manuscript received: 24 June 2022

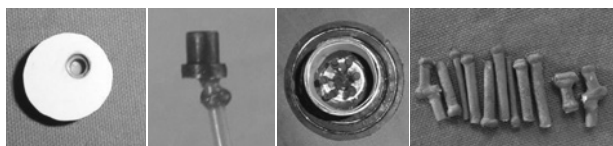
Revision accepted: 30 August 2022

**How to cite this article:** Manne P, Ravuri K. Evaluation of porcelain to metal bond strength on use of recast nickel-chromium alloy. *MJMS*. 2022; 7(1): 25-32

International Pvt Ltd. The composition of the alloy included nickel (61%), chromium (25%), molybdenum (10.5%), silicon (1.5%), and carbon (<1%). For each of the three groups, 26 specimens were prepared. The first group of specimens was cast from 100% fresh alloy and served as the control (FM). The second group of castings was made by mixing 50% once-cast alloy with 50% fresh alloy (FRM) to determine whether the use of recast alloy significantly influenced the alloy-porcelain bond. The third group of castings was cast with 100% once-cast alloy (RM). The porcelain used in this study was VMK 95 Metall Keramik by Vita Zahnfabrik in Bad Sackingen, Germany. All alloy specimens of the three groups were bonded to porcelain. The bond strength between the nickel-chromium alloy and dental ceramic was evaluated using a circular interface shear bond test. This test was chosen for the present study due to its ability to create a uniform distribution of interfacial stresses [6, 7].

### A. Fabrication of the metal specimens [2,8,9]: (Fig 1)

The fabrication process for the metal specimens involved creating a metal die and making an impression using putty material. The metal die [8] was created by machining a cylinder with a diameter of 5 mm. The die measures 4 mm in diameter and 4 mm in height, with a 5 mm diameter cylindrical base that is 1 mm in height. An impression of the die was made using putty material (Affinis perfect impression, putty super soft; Coltene-Whaledent), and patterns for the specimens were created using inlay wax (Charminar dental products, Hyderabad). Please refer to Figure 1.



**Fig 1:** a) putty mould, b) wax pattern, c) investing of wax patterns, d) sprues prepared for recasting.

These patterns were sprued, invested, and cast. To reuse the alloy in the second and third group specimens, i.e., the sprues and buttons were

prepared by cutting them into smaller pieces (see Fig 1), sandblasted with 110  $\mu\text{m}$  aluminium oxide, and cleaned using an ultrasonic machine (Digital ultrasonic cleaner; CD 4820) before casting. Following the casting process and sprue separation, an abrasive treatment using aluminium oxide (110  $\mu\text{m}$ ) was performed for approximately 10 seconds at a distance of about 2 cm, under a pressure of 4 bars, and at a 45° angle. Subsequently, these specimens were cleaned in the ultrasonic cleaner for approximately 180 seconds.

### B. Procedure followed for ceramic application [8]:

All the castings underwent oxidation in a ceramic furnace (Multimat® Touch 2, Dentsply) before the opaque porcelain was applied. The circular surface with a diameter of 4 mm on the metal specimen received two layers of opaque porcelain and three layers of dentin body porcelain (VMK 95 Metall Keramik Vita Zahnfabrik, Bad Sackingen, Germany). The dentine body porcelain was incrementally built up to a height of 4 mm in two stages of 2 mm each. An additional application of these porcelain layers and another firing cycle were necessary for all the metal-ceramic specimens to account for shrinkage from previous firing cycles. Any excess porcelain was then trimmed off. The final test specimens had uniform dimensions, as illustrated in Figure 2.



**Fig 2.** Finished specimen.

### C. Simulating clinical conditions by thermocycling:

After eliminating specimens with visible defects, the samples for each group were subjected to

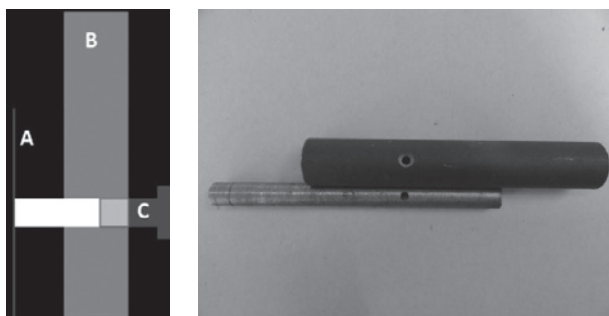
thermocycling between 10°C ( $\pm 5^\circ\text{C}$ ) and 55°C ( $\pm 1^\circ\text{C}$ ). The thermocycling process involved maintaining a temperature for 30 seconds at each end for a total of 5,000 cycles using a custom-fabricated thermocycling machine. The samples from each group were placed in color-coded bags and hung from the belt. The belt rotated continuously, transferring the bags containing the samples from the hot bath to the cold bath.

#### D. Determining the Porcelain-Alloy Bond strength:

The shear bond strength between porcelain and the metal substructure was evaluated by a test method designed by de Melo *et al.* [8].

The testing of all bond-strength specimens was done in a specially designed steel fixture [8] for a tensile testing machine (Kamal Metal Industries, Ahmedabad) (Fig 3). The fixture containing a metal-ceramic specimen was placed on the tensile testing machine (Kamal Metal Industries, Ahmedabad) with a maximum load capacity of 500 kgf (kilogram force). The 100 kgf scale was utilized for this study. A crosshead speed of 1 mm/min was used to apply a tensile force on the steel fixture until fracture of the metal-ceramic test specimen occurred with shear loading. Using the interfacial area, the maximum load (in kgf) supported by the metal-porcelain interface was converted to MPa to obtain the shear bond strength value.

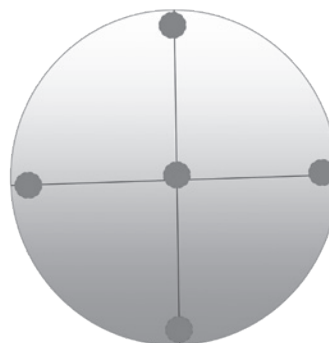
The following formula was used:  $N = F \text{ (kgf)} \times 9.8 \text{ m/s}$ .



**Fig 3:** Fixture fabricated for the tensile testing machine.

#### E. Microanalysis of the fractured surfaces

Five locations from 10 samples were analyzed (Fig 4). The EPMA (Electron Probe Micro Analyser) analysis was carried out with the Cameca SX 100 Electron Probe Micro Analyser, employing wavelength dispersive spectrometers (Petrology Division, Southern Region, Geological Survey of India, Hyderabad). Experimental conditions used were: Accelerating voltage- 15 kV, beam current- 20 nanoamperes, and beam diameter- 50 microns.



**Fig 4:** Representative areas of microprobe analysis for each sample.

#### F. Statistical analysis

The shear strength results were compared using statistical analysis with a one-way analysis of variance (ANOVA) and Tukey's multiple comparison test. Additionally, the weight percentage of each element's oxide was obtained through microprobe analysis and compared between the fresh alloy group (FM) and recast alloy group (RM) using the student's t-test.

#### Results

Tables 1 and 2 provide the means and standard deviations of the shear bond load, along with the results of the one-way ANOVA analysis and the Tukey multiple comparison tests. The Tukey multiple comparison test revealed that the bond strength between the ceramic tested and the alloy of group RM was significantly higher compared to that of the alloy ceramic combination of group FM ( $P=0.00$ ) and that of group FRM ( $P=0.01$ ). However, there was no significant difference in

the bond strength of alloy groups FM and FRM (P=0.24).

**Table 1:** Mean Comparison between FM, FRM, and RM by using the one-way ANOVA technique for the alloy – Dentalloy International.

| Source of Variation | Sum of squares | Mean sum of Squares | F     | P-value              |
|---------------------|----------------|---------------------|-------|----------------------|
| Between Groups      | 1542.65        | 771.33              | 11.08 | *0.00008 significant |
| Within Groups       | 3967.69        | 69.61               |       |                      |
| Total               | 5510.34        |                     |       |                      |

Statistical analysis: one-way ANOVA test; \*statistically significant (P<0.05)

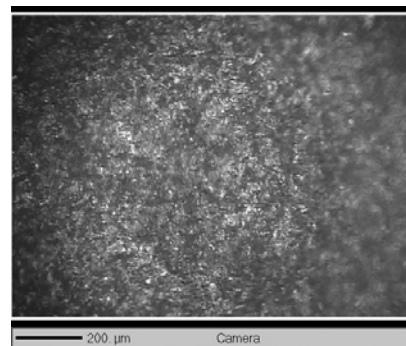
**Table 2:** Analysis of data by Tukey Multiple comparisons test for the alloy – Dentalloy International.

| Comparison Between Variables | Sample Size (N) | Mean ±Sd   | Mean Difference | Tukey Analysis P-Value |
|------------------------------|-----------------|------------|-----------------|------------------------|
| FM                           | 20              | 25.43±7.34 | 4.33±1.61       | 0.237 Not significant  |
| FRM                          | 20              | 29.76±8.95 |                 |                        |
| FM                           | 20              | 25.43±7.34 | 12.24±1.31      | 0.000 Significant      |
| RM                           | 20              | 37.67±8.65 |                 |                        |
| FRM                          | 20              | 29.76±8.95 | 7.91±0.30       | 0.000 Significant      |
| RM                           | 20              | 37.67±8.65 |                 |                        |

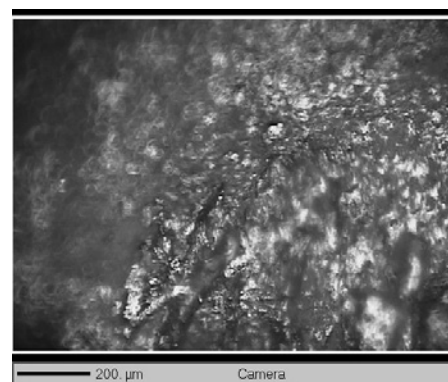
Statistical Analysis: Tukey Analysis

The mean bond strength of specimens that were made using metal as received from the manufacturer (FM) was 25.43±7.34 MPa, whereas that of the mixture of as received and recast alloy (FRM) was higher at 29.76±8.95 MPa. The mean bond strength value for recast metal-ceramic specimens (RM) was significantly greater at 37.67±8.65 MPa. All specimens in all groups demonstrated a mixed mode of failure (adhesive-between metal and ceramic; & cohesive-within ceramic), and all specimens had numerous porcelain fragments on the fractured surface. There was no significant difference in the silicon counts obtained from the fractured surface of

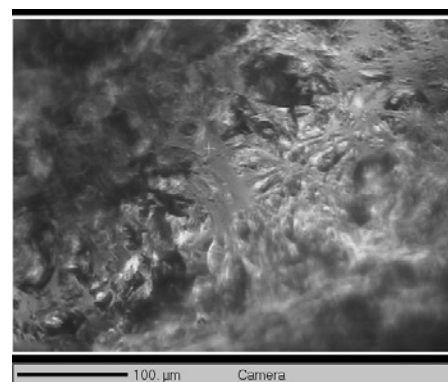
specimens from the fresh alloy group (Figs 5 and 6) and those of the recast alloy group.



**Fig 5:** Image of the fractured specimen of Group FM (reflected light).



**Fig 6:** Image of the fractured specimen of Group RM (reflected light).



**Fig 7:** Image of a ceramic portion of the fractured specimen (reflected light).

However, there was a significant decrease in the oxides of the elements present in the alloy, such as oxides of nickel, chromium, molybdenum, and manganese (Table 3).

**Table 3:** Analysis of weight percentage of oxides by student's t-test.

| Clinical Parameter             | Dent int FM st    | Dent int RM st    | Difference      | t value | P-Value  |
|--------------------------------|-------------------|-------------------|-----------------|---------|----------|
|                                | Mean $\pm$ SD     | Mean $\pm$ SD     | Mean $\pm$ SD   |         |          |
| Na <sub>2</sub> O              | 3.50 $\pm$ 1.68   | 3.56 $\pm$ 1.73   | 0.06 $\pm$ 0.05 | -0.162  | 0.871 NS |
| TiO <sub>2</sub>               | 2.74 $\pm$ 2.31   | 3.43 $\pm$ 2.92   | 0.69 $\pm$ 0.61 | -1.307  | 0.194 NS |
| MnO                            | 0.05 $\pm$ 0.04   | 1.12 $\pm$ 1.36   | 1.07 $\pm$ 1.32 | -5.530  | 0.000 S  |
| K <sub>2</sub> O               | 3.71 $\pm$ 2.29   | 4.18 $\pm$ 2.39   | 0.47 $\pm$ 0.10 | -0.998  | 0.321 NS |
| Al <sub>2</sub> O <sub>3</sub> | 9.47 $\pm$ 2.93   | 7.89 $\pm$ 3.28   | 1.58 $\pm$ 0.35 | 2.535   | 0.013 S  |
| SiO <sub>2</sub>               | 27.25 $\pm$ 14.25 | 30.19 $\pm$ 12.81 | 2.94 $\pm$ 1.44 | -1.082  | 0.282 NS |
| NiO                            | 19.72 $\pm$ 14.39 | 10.89 $\pm$ 12.72 | 8.83 $\pm$ 1.67 | 3.250   | 0.002 S  |
| Cr <sub>2</sub> O <sub>3</sub> | 18.06 $\pm$ 12.04 | 9.00 $\pm$ 8.20   | 9.06 $\pm$ 3.84 | 4.397   | 0.000 S  |
| MoO <sub>3</sub>               | 4.11 $\pm$ 3.32   | 2.32 $\pm$ 3.03   | 1.79 $\pm$ 0.29 | 2.813   | 0.006 S  |
| SnO                            | 4.47 $\pm$ 2.53   | 3.43 $\pm$ 2.73   | 1.04 $\pm$ 0.20 | 1.979   | 0.051 NS |

Statistical Analysis: Student's t-test. Statistically significant if  $P < 0.05$ ; (Statistically significant results that are in bold).

## Discussion

To prevent the depletion of natural resources and for economic reasons, metal alloys from the casting surplus are often reused by melting and recasting - either partially or completely [10]. The failure of porcelain on a porcelain-metal restoration is a significant and frequently encountered issue in clinical practice, which is both expensive and time-consuming to repair [11]. There have been several proposed methods to measure the adhesion between metal and ceramic, but none of them are completely error-free [12]. When testing the ceramic-metal bond, it is crucial to use a reliable test design that minimizes experimental variables and residual stresses [6]. No particular test was totally free of inherent error due to the complexity of the ceramic-metal bond. After evaluating multiple test designs and research instruments, the circular-planar interface shear test by de Melo [8] was chosen for its exceptional reliability and accuracy. Unlike other bond tests, this test has the least residual stresses at the metal-ceramic interface due to its minimal experimental variables. Additionally, this test is not as critical as pull-through tests or bend tests, making it a more practical choice. It is worth noting that stress concentration effects present themselves as a significant factor in all bond tests, except the parallel shear test [13].

Most of the bond experiments mentioned in the literature have shown the presence of stress

concentration near the load application site, which can be a problem. However, in the current study, this problem may be less significant since the specimen was fully supported by the test apparatus [8]. The device created for this test was designed so that the force on the plunger was directed precisely at the metal-ceramic interface, very similar to the intraoral incident forces on the metal-ceramic crown [9].

It is important to consider thermal stresses when dealing with the metal-ceramic interface due to the varying coefficients of thermal expansion between metal and porcelain. To avoid residual stresses in metal-ceramic restorations, the current practice is to design the metal with a slightly greater CTE (coefficient of thermal expansion) than the porcelain - usually around  $0.5^{\circ}\text{C}$  [6,11]. In this study, Dentalloy International alloy was used with a CTE of  $14.1 \times 10^{-6}$ , which is slightly higher than the CTE of Vita VMK 95 porcelain ( $13.7 \times 10^{-6}$ ).

Studies have shown that thermal cycling can cause repetitive stress on the metal-ceramic interface, resulting in a deterioration of the bond between the two materials [2]. In this study, temperature variations of  $55 \pm 1^{\circ}\text{C}$  and  $10 \pm 5^{\circ}\text{C}$  were used with a mean dwell time of 30 seconds in each temperature bath, as recommended by Gale and Darvell [14].

The samples were subjected to 5,000 cycles, which is equivalent to approximately six months of clinical

service. The samples were subjected to 5,000 cycles to simulate approximately six months of clinical service and replicate the range of temperatures dental restorations are exposed to in the mouth, caused by hot and cold foods.

The alloy analyzed in this research exhibited a notable increase in the strength of the bond between metal and ceramic when recast. The findings from this study are in line with conclusions by Moffa *et al.*, [7], which indicate that recasting the alloy does not have a negative impact on the bond strength of porcelain to base metal alloys. Recasting has a substantial effect on the microstructure and physical characteristics of noble alloys [4]. However, this does not apply to base metal alloys. Hesby [15], as well as Palaskar *et al.*, [16,17], noted no significant differences in the physical properties of recast base metal alloy.

According to McLean [18] and most manufacturers of base metal alloys for metal-ceramic restorations, at least 50% new metal (for high gold alloy) should be included in copings. However, there is currently no experimental justification for this 50% rule for base metal alloys. Palaskar *et al.*, [16] argued that if the once-cast alloy is completely cleaned and deoxidized, fresh alloy need not be added in any proportion. In the current study, there was no significant change in bond strength when 50% fresh alloy was added to once cast alloy. However, the outcome of the current study differs from the study of Ucar *et al.*, [2], who observed a decrease in the bond strength of 50% fresh alloy mixed with once-cast nickel-chromium alloy when compared to the bond strength of ceramic with fresh alloy.

The current standards, ANSI/ADA specification number 38 and ISO standard 9693, for evaluation of the metal-ceramic bond employ a three-point bending test. The minimum bond strength that is deemed acceptable with this test is 25 MPa. Ucar *et al.*, showed an agreement in the test results of the 3-point bending test and shear bond strength test [2]. The bond strength values observed in this study for all the metal-ceramic specimen groups prepared exceeded the minimum recommended value of 25

MPa. These findings indicate that recasting the base metal alloy does not negatively impact the bond strength between metal and ceramic in dental restorations.

Al-Hiyasat and Darmani [19] found that alloys with higher levels of chromium (i.e., 16-25%) and molybdenum had lower metal dissolution rates, which could potentially decrease the cytotoxicity of the alloys. Since the tested alloy in this study has a high concentration of Chromium at 25% (Dentalloy International), it is possible to reuse it at least once.

Many researchers believe that the bond between ceramics and metals is a direct result of oxide formation at the interface of the two materials. There is a zone of approximately 10-25  $\mu\text{m}$  width at the porcelain-metal interface that has a different composition than the bulk of the metal and appears lighter in colour. According to Baran *et al.*, [20], this non-stained appearance is due to the depletion of some alloy elements. Although it is widely accepted that the presence of an oxide on the alloy surface is necessary for bonding to occur, a thick oxide layer that cannot be completely dissolved by porcelain may serve as a weak point in the transition zone between porcelain and metal [20].

McLean demonstrated that nickel and chromium oxide decreased the thermal coefficient of expansion of porcelain and suggested that this may induce stresses that could cause failure of non-precious ceramic-metal restorations [12]. Péraire *et al.*, [10] found that recasting of nickel-based alloy showed a slight decrease in chromium concentration. The results of this study showed a significant decrease in the concentration of the oxides of nickel, chromium, manganese, and molybdenum. This decrease in the oxide layer, inducing a lesser amount of stress on the metal-ceramic interface, could have been the reason for the increased bond strength of the metal-ceramic specimens when the alloy was recast.

McLean's study showed that the thermal coefficient of expansion of porcelain decreased when nickel and chromium oxide were present, potentially leading to stresses in non-precious ceramic-metal

restorations [12]. Peraire et al., [10] also observed a slight decrease in chromium concentration upon recasting nickel-based alloy. The current research findings indicated a significant reduction in the oxides of nickel, chromium, manganese, and molybdenum. This reduction in the oxide layer may have contributed to lower stress levels at the metal-ceramic interface, possibly explaining the enhanced bond strength observed when recasting the alloy.

## Conclusion

The present study reveals that nickel-chromium alloys can be safely reused without any detrimental effects on their bond with ceramic, thereby reducing the cost of dental prostheses and promoting sustainable practices. The findings suggest that base metal alloys can be reused at least once without requiring the addition of fresh alloy. However, it is imperative to strictly adhere to cleaning procedures to ensure the removal of surface contaminants from the sprues and buttons before reusing the alloys. The implementation of such measures can significantly reduce waste and enhance the operational efficiency of dental laboratories.

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