

## EFFECT OF OXIDATION AND PORCELAIN FIRING TEMPERATURES ON TENSILE BOND STRENGTH OF PORCELAIN TO NI-CR-MO BASE METAL SURFACES IN DENTISTRY

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### ABSTRACT

**Purpose:** The main objective of this experimental study was to investigate the following hypothesis. 1. Ceramic base metal surfaces subjected to a particular oxidation and porcelain firing temperature cycle produce the highest tensile bond strength to porcelain. 2. To determine the interfacial adhesion of porcelain to ceramic base metal surfaces the tensile bond strength test is appropriate. **Materials and Methods:** The specimens having a disk of 10mm diameter, height 4mm and an extended rod of gauge diameter 3mm and gauge length 25mm, were cast from commercially known Ni-Cr-Mo base metal alloy i.e. Wirocer (BEGO, Germany) and divided into three groups of 10 specimens each. Before obtaining the specimens wax patterns were prepared using a mould of standard dimensions and invested in a metallic ring using phosphate bond investment material. The investment ring was placed inside a furnace to burn out the wax. The ready moulds having cavity exactly as per the design was used for casting the specimens. Tensile test specimens were cast using centrifugal induction casting machine (Degussa, Germany). All specimens were recovered after deinvesting and sand blasting using Al<sub>2</sub>O<sub>3</sub> abrasive and ultrasonic cleaning. Each group of specimens was subjected to a standardized oxidation and porcelain firing temperature cycle and fused together using porcelain material. The specimens were loaded to fracture under tensile testing machine (Hounsfield H5K-S-0195). The tensile bond strength data was statistically analyzed and fractured surfaces were observed under SEM (scanning electron microscope). **Results:** Group II specimens subjected to cycle II, oxidation and porcelain firing temperatures showed statically higher mean tensile bond strength values. The reason for this was due to good interlocking of the atoms of the oxides in the oxidation layer with the metallic atoms of the base metal and also with the porcelain. The temperatures selected as per cycle I and cycle III showed variation in the formation of oxides, which affecting the tensile bond strength values. Failures of the specimens found occurred in the porcelain region and adhesion zone. **Conclusion:** Tensile bond strength is appropriate for the analysis of the porcelain to the metal ceramic surfaces. The oxides formed on the ceramic surface during oxidation and porcelain firing temperatures used in this study as per different cycles, found affecting greatly the bond strength values. It was suggested to use temperatures according to cycle II for better tensile bond strength results. **Acknowledgements:** This work was partially carried out at Bapuji Dental College and Hospital, and the author thank the Principal for providing materials and ceramic laboratory facilities.

**Keywords:** Oxidation layer, bond strength, firing temperatures

### INTRODUCTION

Metal-ceramic restorations are prepared under clinical conditions using low fusing porcelains. A good understanding of the bonding mechanism is essential for successful metal-ceramic restorations. Although various theories and concepts have been proposed for the actual

mechanism of bonding, it still remains elusive. As according to Maeda, the maximal occlusal force in anterior intraoral quadrants ranged from 255 to 372 N for an individual tooth. Although this range of forces on a well-designed metal-ceramic restoration is insufficient to cause subclinical fractures of anterior teeth,

occasional chipping and fracture of porcelain at the porcelain-metal interface or through the porcelain in common. Failures in restorative dentistry can be attributed to improper selection of metal-ceramic materials, oxidation temperature, and porcelain firing temperatures.<sup>1</sup> In this experiment, a standardized metal-ceramic metal alloy Wirocer, Bego Germany make was used. Three different oxidation and porcelain temperature cycles were opted for all the groups of alloys. Of the three different oxidation/porcelain firing temperature cycles used in this study one produced a high of 70 MPa tensile bond strength.

## MATERIALS AND METHODS

The materials used in this experimental study are presented in table 3. Thirty specimens were fabricated using standardized Nickel-Chromium base metal alloy (Wirocer, BEGO, Germany, Ni 66%, Cr 22%, Mo 9%, Si 1.6%, Fe 0.5%, Ce 0.4%) for ceramic restorations and divided into three groups of 10 specimens each. Each specimen has a cylindrical disk of diameter = 10mm, height 4 mm and another headed rod of gauge diameter 3mm as shown in figure 1.

Wax patterns were prepared from two molds of standard dimensions. A phosphate bond graphite free casting investment (Whip- mix) was used and a two-stage burn out procedure was used with a peak temperature of 850<sup>0</sup>C (using KAVO type 5635 furnace). The alloy was heated to its casting temperature of 1420<sup>0</sup>C in a ceramic crucible (silicast, make) and cast using high frequency centrifugal induction casting machine (Degussa, Degutron model1088, Germany make). After devasting, the specimens were sand blasted using 60µm Al<sub>2</sub>O<sub>3</sub> and recovered after ultrasonic cleaning in distilled water for 10 minutes and dried. The surface for porcelain bonding was abraded to stimulate the alloy surface preparation used under clinical conditions

The manufactures instructions for the initial oxidation treatment for the alloy recommend an oxidation temperature up to 980<sup>0</sup>C with out stating the lower range of temperatures. So, in this experimental study three different oxidation/porcelain firing temperature cycles were employed. All the specimens were divided into three groups of 10 specimens each. Each group of specimens was subjected to particular oxidation / porcelain firing temperature cycle as indicated in table 1.

Commercially popular porcelain VITA VMK 95 that is compatible with the alloy was selected. Two layers of opaque porcelain was applied using the spatulation method of condensation on the tail end of each sample and had a total thickness of approximately 0.2 mm. Then two layers of body porcelain i.e. dentin that had an approximate thickness of 1-1.25 mm was added over the opaque porcelain. The porcelain firing was done after keeping the tail ends of two samples in approdimation so that the final test sample will consists of porcelain adhering to the two metal surfaces (fig 1). Lastly the porcelain was glazed. This procedure was repeated for all the samples of each group. The oxidation and porcelain firing was performed using a programmable vacuum porcelain-firing furnace. (ivoclar programat

,Germany ).The gauge dimensions of all the specimens of three groups were examined carefully on a profile projector (Nikon V12) and are used individually during the test to obtain the stress v/s strain plot. Ready test specimens were fastened individually on the "Hounsfield" universal testing machine (model type H5KS, serial number H5 KS-0195) using a special fixture to hold precisely the specimens in the axial direction. Figure 4 shows the tensile test arrangement.

## RESULTS

Tensile bond strength values of all the three groups of specimens subjected to three different oxidation/porcelain firing temperature cycles are presented in table 2. The group II specimens subjected to as per cycle II oxidation/porcelain firing temperatures has produced the greatest porcelain bond strength values to the base metal alloys. But, in this experimental study the bond strength values produced by the remaining two oxidation/porcelain firing temperature cycles found comparatively low to the values produced by the cycle III. The figure 2 shows the stress v/s Strain plots obtained while conducting the tensile test on three samples one from each group of base metal alloys. These curves indicating the percentage of elongation %EL varying from approximately 4% to 4.5%. This elongation includes the extension of the porcelain along with the base metal alloy together.

The Vickers hardness values of the three groups of base metal alloys, subjected to Oxidation/porcelain firing temperatures ranged between 220-230. The Fig 3 shows the polynomial nominal stress-strain curves for three groups of alloys when tested for porcelain bond strength to the alloy surface. The plot indicates that all the specimens of the three groups following the same type of path with slightly different fracture loads at the end. The mean tensile bond strengths of the porcelain to the alloy surface varying from 61.5 to 70.9 MPa. Also, this mean value is greatly dependent on a particular oxidation temperature for a metal alloy rather on the porcelain firing temperatures used in this study. This is evident from the hardness test results.

All the specimens were observed under SEM(scanning electron microscope) for fracture analysis. The failure of specimens of all the groups found to be due to the fracture at different fracture origin locations. The occurrence of the fracture line in few specimens found in the porcelain region and in the remaining at the metal-ceramic interface region. The results indicate that both cohesive strength of porcelain and bond strength at the metal-ceramic interface is to be accounted. Based on these bond strength values at the interface found more than the cohesive strength of the porcelain at the porcelain region.

## DISCUSSIONS

The tensile bond strength arrangement has been the most common laboratory technique for evaluating the bond of porcelain to metal surfaces. The important factor that will affect the measured bonding efficacy is the precise area of bonding. This is achieved in the present experiment, despite of extra precautions,

imperfections was still observed at the joint that may have affected the results. However the technical laboratory procedures, such as drying the porcelain, condensation, firing and handling the alloy surface have an effect on bond strength and fracture durability.<sup>2</sup>

This study compared the bond strength of porcelain fused to metal surfaces in three groups of specimens subjected to three oxidation/porcelain firing temperature cycles. Several scientists have shown the problems associated with most popular test arrangements used in the dentistry today, questioning the reliability of such measurement to provide useful information about the bond strength of porcelain to ceramic. Few studies explained that this tensile test helps to find the adherence of oxide layer to the porcelain and the metal surface.<sup>3</sup>

The principles of a tensile bond strength if to measure the strength of the bonding area between the two substrates by pulling them apart. A load / displacement curve; which is converted to a nominal stress-strain curve is generated during normal tensile testing. As reported in table 3 the Vickers hardness number increased in the range between 230 to 240 for group II specimens after porcelain firing. A careful interpretation of the failure mode is required to prevent inappropriate conclusions about the utility of any bond strength test configuration. Knowledge about failure mechanics concepts and the analysis of fracture events on the basis of metallurgical microscopic results will reduce the risk for data interpretation, such as the influence that the bond strength must exceed the cohesive strength of the porcelain when the fracture initiates away from the interface.<sup>4</sup>

The Fig 3 shows the polynomial stress-strain curves for three groups of alloys when tested for porcelain bond strength to the alloy surface. The plot indicates that all the specimens of the three groups following the same type of path with different fracture loads at the end.

## CONCLUSIONS

The following are the conclusion drawn after performing this experimental study.

- The metal-ceramic bond strength between NI-Cr-Mo (Wirocer, BEGO) base metal alloy and VITA VMK 95 (Low-fusing temperature) porcelain called dentin was evaluated using three different oxidation/porcelain temperature cycles.
- Specimens all the groups were subjected to an initial oxidation and porcelain temperature cycle under vacuum.(using programmable ivoclar furnace)
- When hardness was checked for all the groups of samples after porcelain firing the Vickers hardness

number of the base metal alloys of all the groups increased in the range between 220 to 230.

- From the experimental results it is clear that the oxidation layer formed due to a particular oxidation temperature is the deciding factor controlling the tensile bond strength values.
- So, it was suggested to the laboratory technicians at Bapuji College of Dental Sciences to perform oxidation/porcelain temperature cycle as per cycle II.
- Though we use the results of tensile bond strength of porcelain to the base metal for the comparison purpose the compression strength lies few times more than the tensile bond strength.
- According to Maeda the maximal occlusal force in



the anterior intraoral quadrants ranged from 260 to 380 N for an individual tooth. So, when the bond strength of porcelain to the base metal alloy found approximately 71Mpa, the forces exerted on the metal-ceramic are insufficient to cause any subclinical fracture.

**Table.1** Oxidation/Firing temperatures in °C cycles

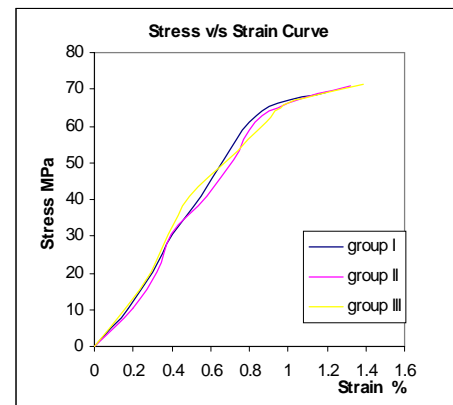
Stage	Cycle I Group I samples	Cycle II Group II samples	Cycle III Group III samples
<b>Fig 1</b> Fused specimen			
Oxidation	930	960	990
Opaque I	930	950	970
Opaque II	900	910	920
Dentin I	900	910	910
Dentin II	890	900	900
Glaze	830	830	830

**Table.2** Tensile bond strength values.

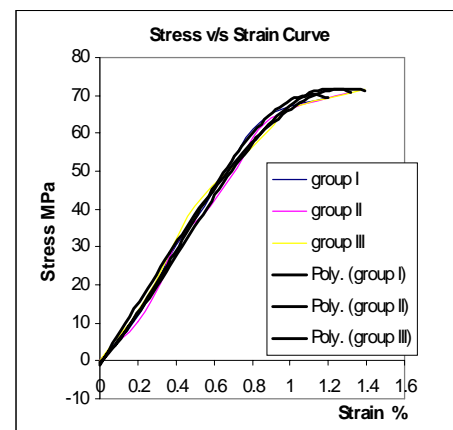
Alloy group	Mean tensile bond strength values in, MPa	Standard deviation $\sigma$
Group I	61.5	5.56
Group II	70.9	2.97
Group III	67.5	4.45

**Table 3** Materials used in this study.

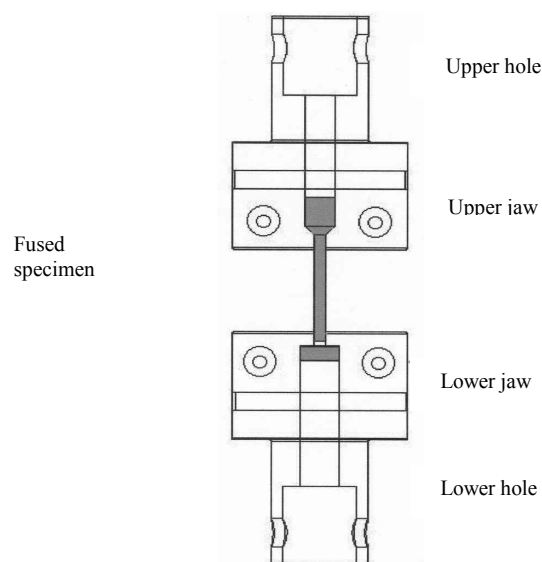
Brand name	Description	Manufacturer (lot No)
VITA VMK 95 metal ceramic OPAQUE	Opaque ceramic shade A1	VITA Zahnfabrik H Rauter GmbH & Co KG, Germany. (J017 B306150 5225)
VITA Opaque Fluid	Liquid	VITA, Germany. (5020)
VITA Modeling Fluid for VITA VMK 95	Liquid	VITA, Germany. (J017BMF 200/\$ 6667)
VITA VMK 95 Metal Ceramic, Dentin	Leucite-based shade A1	VITA, Germany. (J017 B301550 B 5446)
VITA Algent Akz 25 Glaze	Powder	VITA, Germany. (+J017BAT257 60 6229k)
VITA Algent	Liquid	VITA, Germany. (+J017BATF22 64721)
WIRO CER	Pellets	BEGO, Germany. (1770)
BELLVEST T	Phosphate bond Graphite free precision casting investment	BEGO, Bat Nr 12347.k 1768)
BEGO SOL	Mixing liquid	BEGO (Batch No 80204)
SILICAST	Ceramic Crucibles	(15300)



**Fig 2** Stress v/s Strain plots for three groups.



**Fig 3** Polynomial Stress v/s Strain plot for three groups



**Fig 4** Tensile test arrangement

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## NOMENCLATURE

Symbol	Meaning	Unit
$\sigma$	Standard deviation	----