Original Article

Assessment of Marginal Fit, Ceramometal Shear Bond Strength, and Fractographic Analysis of Sinter Cast Gold: An *In vitro* Study

Hetal Turakhia, Sabita M. Ram[1], Aruna Mehta[2], Bhushan R. Bangar[3], Abhishek Singh Nayyar[4]

Departments of Prosthodontics and Crown and Bridge and [4]Oral Medicine and Radiology, Saraswati-Dhanwantari Dental College and Hospital and Post-Graduate Research Institute, Parbhani, [1]Department of Prosthodontics and Crown and Bridge, MGM Dental College and Hospital, [2]Department of Prosthodontics and Crown and Bridge, Dr. DY Patil Dental College and Hospital, Navi Mumbai, [3]Department of Prosthodontics and Crown and Bridge, Maharasthra Institute of Dental Sciences and Research, Latur, Maharashtra, India

Abstract

Introduction: Fractographic analysis is performed by observing, measuring, and interpreting the fracture surface topography that can determine many features of the microstructure of materials and the mechanics of the fracture. This study was planned to put through investigation, marginal fit and ceramo-metal shear bond strength with fractographic analysis of the debonded surfaces. The aim of this study was to assess the marginal fit, ceramo-metal shear bond strength of sinter cast gold and to analyze the debonded sample by fractography. **Materials and Methods:** A total of 5 sinter cast gold copings were prepared on a stainless master die resembling a maxillary 1st pre-molar to receive a ceramo-metal restoration. Each of the copings were, then, assessed for marginal fit before and after ceramic firing. Also, a total of 5 ceramo-metal discs were fabricated for testing shear bond strength at the metal-ceramic interface. The samples were subjected to increasing shear load on a Universal Testing Machine with crosshead speed of 0.002mm/sec. The load at which, the samples debonded completely, was noted and the samples put-to fractographic analysis. **Results:** The mean marginal fit found in this study for sinter cast gold before ceramic firing was measured to be 11.5 µm with a standard deviation of ±4.64 µm and after ceramic firing was found to be 9.38 µm with a standard deviation of ±3.57 µm. The average shear bond strength at the sinter cast gold ceramic interface was 18.216 MPa. The fractographic analysis showed the globular microstructure of sinter cast gold on sintering, adhesive, and mix-mode type of fracture with the presence of both gold and ceramic. **Conclusion:** The marginal fit observed before and after ceramic firing was within clinically acceptable range while the ceramo-metal shear bond strength between sinter cast gold and ceramic was found to be inadequate.

Keywords: *Ceramo-metal restorations, fractographic analysis, shear bond strength*

Clinical Relevance to Interdisciplinary Dentistry

It is an *in-vitro* study with great clinical relevance in the field of dentistry.

Introduction

Modern science and technology in dentistry has traveled the lengths and breadths of many unimaginative firsts. Adaptation and a detailed study of our history gives birth to many such innovations. Ceramo-metal restorations, precious or, non-precious, have been popular for decades. Their success depends on many factors including the accuracy of the preparation, impression materials, techniques, laboratory

Access this article online

Quick Response Code:

Website: www.jidonline.com

DOI: 10.4103/jid.jid_55_16

Address for correspondence: Dr. Abhishek Singh Nayyar, 44, Behind Singla Nursing Home, New Friends Colony, Model Town, Panipat - 132 103, Haryana, India. E-mail: singhabhishek.rims@gmail.com

How to cite this article: Turakhia H, Ram SM, Mehta A, Bangar BR, Nayyar AS. Assessment of marginal fit, ceramometal shear bond strength, and fractographic analysis of sinter cast gold: An *In vitro* study. J Interdiscip Dentistry 2017;7:1-6.

procedures, type of alloys, ceramics and also, the skill of the operator.[1,2] Marginal fit is of prime importance to provide a seal at the margins and reduce the thickness of luting cement. There are many theories proposed in literature to explain the improper fit of ceramo-metal restorations when they are fabricated by the lost wax technique.[1,2] There are various steps involved in the fabrication of a ceramo-metal restorations. Distortion may creep-in at any of the stages during the fabrication procedures which may ultimately lead to failure in the final restoration. The ceramo-metal bond strength is, also, an important parameter in the success of the restoration. This is because during intra-oral function, the restoration is subjected to constant masticatory load and thermal cycling. Although many theories and concepts have been offered relative to the actual mechanism of bonding, the exact mechanism, although, still remains uncertain.[3,4] The accurate measurement of the bond strength at the metal ceramic interface is most complex and various test methods have been used over the years.[5,6] The ceramo-metal interface of debonded restorations can be visualized by fractography which is done by observing, measuring and interpreting the fracture surface topography that can determine many features of the microstructure of materials and the mechanics of the fracture.[7] In the existential scenario of breakthrough technology, further implication of the same, have been researched and discovered to bring foil metal technology in the form of sinter cast gold which is 99 .9% pure gold for the field of dentistry. It has helped to eliminate the lost wax technique which has been popular for years now.[8] Therefore, this study was planned, to put through investigation, two of the important properties of the material- marginal fit and ceramo-metal shear bond strength with fractographic analysis of the debonded surfaces. The aim of this study was to assess the marginal fit, ceramo-metal shear bond strength of sinter cast gold and to analyze the debonded sample by fractography.

Aims and objectives

The main aims and objectives carried out were as follows:

1. To evaluate the marginal fit of sinter cast gold copings before ceramic firing;
2. To evaluate the marginal fit of sinter cast gold copings after ceramic firing;
3. To evaluate the shear bond strength at the interface of sinter cast gold and ceramic; and
4. Fractographic analysis of the surface of the debonded samples of sinter cast gold.

Materials and Methods

Materials used for the test samples

- Sinter cast gold sheet (Nobel metal) composition:
    - Cold (Au) - 99.9%;
    - Trace elements (Ir, Rh, Ta) - 0.1%.
- Properties of sinter cast gold as claimed by the company:
    - Color - yellow;
    - Density - 19.3;
    - Coefficient of thermal expansion - 14.2–14.6;
    - Vicker's hardness - 90–95 VHN;
    - Melting range - 10600–10750;
    - Thickness - 1 mm.

Materials and instruments for fabrication of the test samples

A. Sinter cast gold for adaptation:
- Die Lubricant-Picosep;
- Bard parker blade no. 15 with handle;
- Titanium spatula (Nobel metal).

B. Investing and sintering process:
- Refractory die material (Nobel metal);
- Acetone more than 99% pure (S.D. Fine Chem Limited);
- Aluminum oxide;
- Rubber cup;
- Stainless steel cement spatula;
- Glass slab;
- Hemostat;
- Ceramic furnace plate; and
- Ceramic furnace (Centurion).

C. Recovery and cleaning of sintered samples:
- Hemostat;
- Rubber bowl with cold water; and
- Bard parker blade no. 15 with handle; along with
- Ultrasonic cleaner (Electrosonic, Germany).

D. Finishing of the test samples:
- Sintered diamond burs;
- White messy wheels; and
- Ball burnisher.

E. For ceramic application and firing:
- Sable Paint Brushes (Ivoclar, Vivadent);
- Wash Opaque Ceramic (Vita VMK, Ivoclar);
- Dentin Ceramic (Vita VMK, Ivolclar);
- Hemostat;
- Ceramic furnace plate;
- Ceramic Furnace (Centurion).

F. Finishing of the ceramic surfaces:
- Green Stones (Shofu, Japan); along with
- White Stones (Shofu Japan).

G. Instruments for Measurement:
- Electronic Digital Vernier Caliper.

H. Equipment for testing:
1. For marginal fit:
    - Clay; and
    - Optical microscope.

2. For bond strength:
   - Fixture to hold the sample; and
   - Special jig assembly with rods; and
   - Universal testing machine with Load Analyzer (Bliss, Bengaluru).
3. For fractography:
   - Scanning electron microscope (SEM) (FEI, Quanta 2000).

Methods

Assessment of marginal fit

A Stainless steel replica of a prepared maxillary 1st premolar was prepared to receive a ceramo-metal restoration following guidelines given by Tylman.[9] Eight vertical indentations were engraved on the base of the die approximately 1mm from the finishing margin. These acted as reference points for the measurements of marginal fit under optical microscope. For the fabrication of the copings, sinter cast gold strips were carefully cut from the sheet and adapted to the die. The copings were, then, invested and sintering process was carried-out. The sintered copings were, then, placed on the stainless steel master die and burnished at the margins [Figure 1]. The burnishing was done using ball burnisher as pure sinter cast gold was pure gold sheet and gold is malleable and ductile. In a similar manner, the remaining 4 sinter cast gold copings were fabricated and numbered 1 to 4. Each of the copings were seated on the master die one after the other and assessed for marginal fit before ceramic firing. The fit was observed under an optical microscope at 100X magnification [Figure 2]. The observations were noted and tabulated. Ceramic build-up was done on all the 5 copings [Figure 3]. The marginal fit was assessed in the same manner as done before ceramic firing in the first case and the observations were tabulated [Figure 4].[9-11]

Assessment of ceramo-metal shear bond strength

A total of 5 sinter cast gold discs were fabricated from a circular mould with nut screw assembly for easy retrieval for testing shear bond strength at the metal-ceramic interface [Figure 5]. Ceramic build-up was done on the sintered discs. The samples were numbered 1 to 5. The samples were, then, subjected to increasing shear load on a Universal Testing Machine with crosshead speed of 0.002 mm/sec at the metal ceramic interface [Figure 6]. The loads at which, the samples debonded completely, were noted and the observations were tabulated [Figure 7].[9-11]

Fractographic analysis

The sinter cast gold parts of the debonded samples were cleaned ultrasonically using acetone and examined under scanning electron microscope (SEM). The analysis was seen as images which were recorded photographically [Figure 8].

Figure 1: Sinter cast gold coping on master die

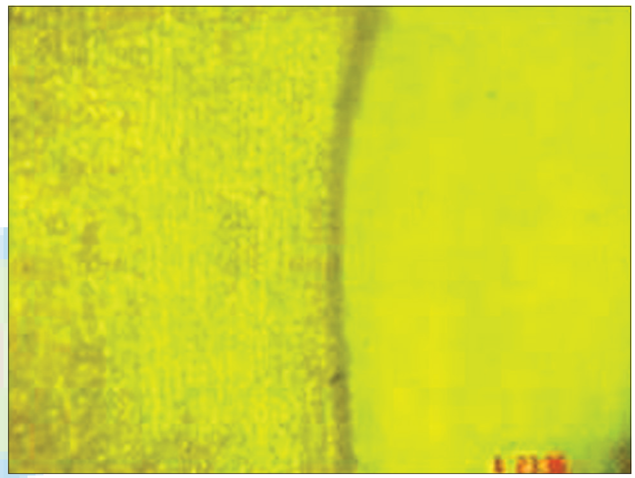

Figure 2: Optical micrograph showing marginal fit of sinter cast gold before ceramic firing

Figure 3: Sinter cast gold coping on master die after ceramic firing

Statistical test used

The data obtained was subjected to statistical analysis. The statistical test used was student's t-test while

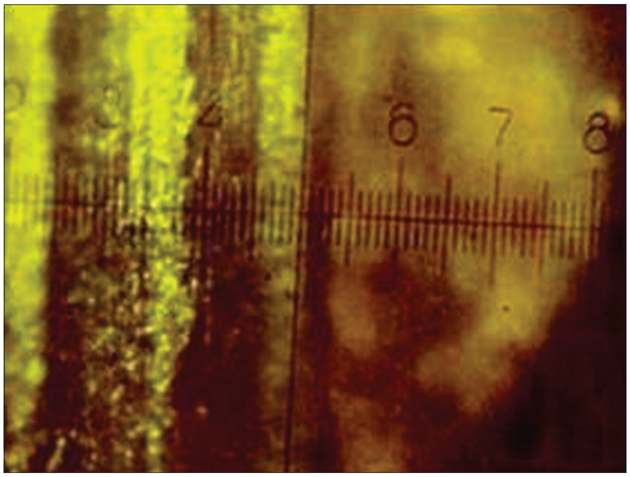

Figure 4: Optical micrograph showing marginal fit of sinter cast gold after ceramic firing

Figure 6: Sample position on universal testing machine for shear bond strength

P value of 0.05 was considered to be statistically significant.

Results

The mean marginal fit found in this study for sinter cast gold before ceramic firing was measured to be 11.5 µm with a standard deviation of ±4.64 µm and after ceramic firing was found to be 9.38 µm with a standard deviation of ±3.57 µm. The change in marginal discrepancy before and after ceramic firing was 2.12 ± 2.93 µm [Table 1]. The average shear bond strength at the sinter cast gold ceramic interface was 18.216 MPa [Table 2]. The fractographic analysis showed the globular microstructure of sinter cast gold on sintering, adhesive and mix-mode type of fracture with the presence of both gold and ceramic [Figures 9-11].

Discussion

Sinter cast Gold, the latest entrant into the arena of foil metal techniques, was introduced with an idea to make

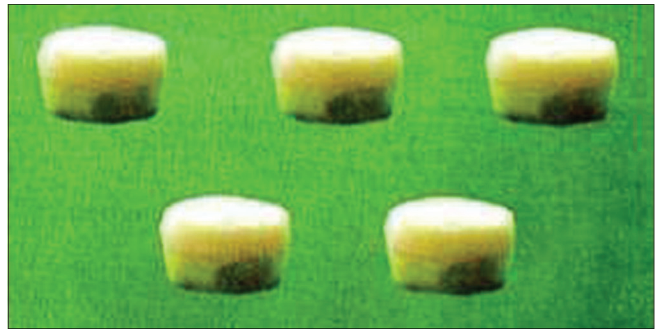

Figure 5: Ceramo-metal shear bond strength test samples

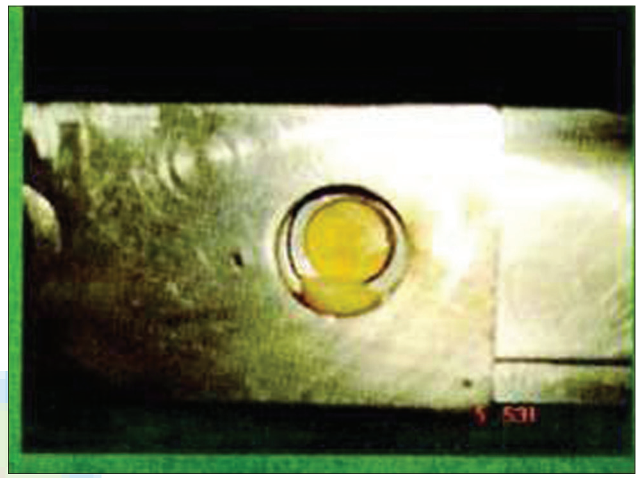

Figure 7: Debonded sample

the laboratory procedures leading to the fabrication of a ceramo-metal restoration simpler and less time consuming. Sintering process is the transformation of metal powders into a single bonded mass without getting to the melting process.[8] The process yields a rugged surface texture suited for mechanical interlocking with ceramic. This method produces uniform thin gold copings of 0.3mm thickness, thus, providing ample space for porcelain to be built-up, which would, in turn, enhance the esthetics of the restoration. A marginal adaptation below 30 µm can be obtained consistently. The ideal cement film thickness ranges from 25-40 µm. The practical range for clinical acceptability of marginal fit is around 50-75 µm. Marginal opening is almost 8 times more in non-precious alloys due to thick oxide layers. The absolute marginal discrepancy for cast restorations has been found to be 64 ± 24.1µm and for electro-formed gold restorations to be 36 ± 32.7µm.[12,13] Captek produces a mean marginal fit of 68µm while those of electro-formed crowns, it has been found to be 32µm and that of high noble alloys, 31 µm.[14] It can, therefore, be seen from the results that sinter cast gold yielded copings with good marginal adaptation and fit. It may be due to elimination of errors as in lost wax technique and because it is burnishable. The range of

Figure 8: Fractographic analysis scanning electron microscope showing globular microstructure of sinter cast gold on sintering

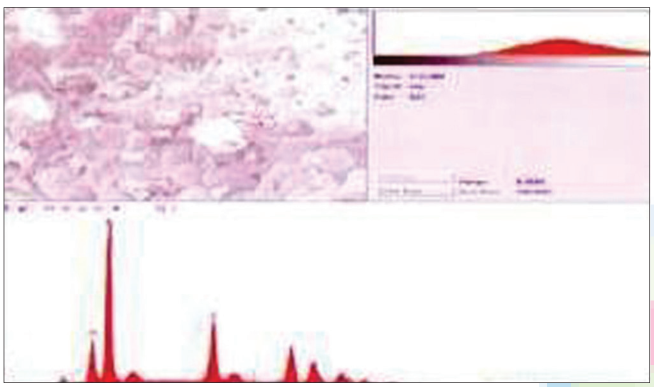

Figure 10: Fractographic analysis showing ceramic constituents on debonded surface

Table 1: Comparison of mean marginal fit before and after ceramic firing

	Mean±SD (µm)	*P*	*t*
Before firing	11.50±4.64	-	-
After firing	9.38±3.57	-	-
Change	−2.12±2.93	>0.05 (NS)	0.72

*Statistical test used: Student's *t*-test. SD=Standard deviation, NS=Statistically nonsignificant (*P*>0.05)

Table 2: Mean shear bond strength, standard deviation and range

	Bond strength (MPa)
Mean±SD	18.216±0.285
Range	17.834-18.598

SD=Standard deviation

ceramo-metal shear bond strength has been rated between 25-55 Mpa depending on the type of porcelain, alloy and the preparation used. Captek, a similar ceramo-metal material, has a shear bond strength of 56.08 ± 9.47 Mpa with a thin layer of bonding agent while increase in the layer of bonding agent decreased the bond strength to

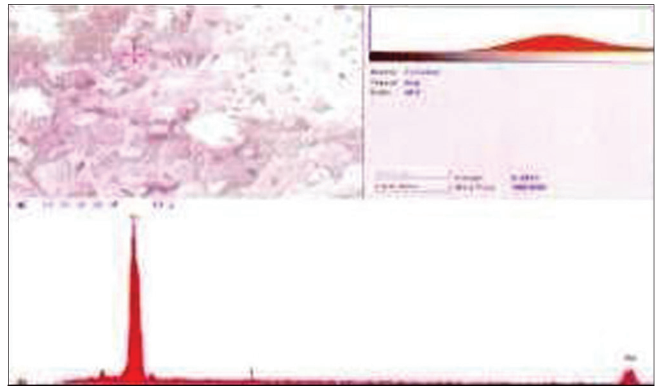

Figure 9: Fractographic analysis showing element on the debonded surface

Figure 11: Fractographic analysis showing globular microstructure of sinter cast gold on sintering, adhesive and mix-mode type of fracture

27.28 Mpa. The shear bond strength of base metal alloy, on the other hand, has been rated to be 29.93 Mpa while ceramic alloy as 21.92 Mpa and palladium base ceramic alloy as 30.49 Mpa.[15,16] The present study found ceramo-metal shear bond strength of sinter cast gold as 18.216 ± 0.285 Mpa which was lesser when compared with the values of similar materials from the previous studies. It was, also, seen in this study that no bonding agent was used for this system. Also, it being 99.9% pure gold, did not have any oxide layer formation at the metal-ceramic interface resulting in a much weaker van der Waal's bond. However, comparison with other type of alloys and influence of time on the clinical use must be evaluated.

Conclusion

Within the limitations of the present *in vitro* study, the conclusions drawn were:

- The change in marginal fit before and after ceramic firing was minimal and statistically insignificant;
- The marginal fit observed before and after ceramic firing was within clinically acceptable range;
- The ceramo-metal shear bond strength between sinter

cast gold and ceramic was found to be inadequate; and
- The fractographic analysis showed mix-mode adhesive fracture between sinter cast gold and ceramic structure.

The system, being new, requires further research and clinical trials to enhance properties of the material.

Financial support and sponsorship

Nil.

Conflicts of interest

There are no conflicts of interest.

References

1. lver M, Klein G, Howard MC. An evaluation and comparison of porcelain fused to cast metals. J Prosthet Dent 1960;10:1055-64.
2. Mumford G. The porcelain fused to metal restoration. Dent Clin North Am 1965;23:241-9.
3. Hamaguchi H, Cacciator A, Tuerler VM. Marginal distortion of porcelain-bonded to metal complete crown: An SEM study. Prosthet Dent 1982;74:146-53.
4. Campbel SD, Sirakian A, Pelletia LB, Giordano RA. Effects of firing cycle and surface finishing on distortion of metal ceramic casting. J Prosthet Dent 1995;74:476-81.
5. Gemalmaz D, Alkumru HN. Marginal fit changes during porcelain firing cycles. J Prosthet Dent 1995;73:49-54.
6. Rajagopal P, Chitre V, Singh RK, Aras M. Tests for ceramometal bond strength. J Ind Prosthet Soc 2003;3:20-5.
7. Derek Hull. Fractography. 3rd ed. New York: ASTM Press; 1998.
8. Mola P. Sintering also in the dental laboratory. J Dent Univlla Dent Assoc 2001:47:49-60.
9. Malone WFP, Koth DL. Tylman's Theory and Practice of Fixed Prosthodontics. 8th ed. St.Louis: Ishiyaku EuroAmerica. Nasher; 1994.
10. Morris HF. Department of Veterans Affairs Cooperative Studies Project No. 242. Quantitative and qualitative evaluation of the marginal fit of cast ceramic, porcelain-shoulder and cast metal full crown margins. Prosthet Dent 1992;67:198-204.
11. Rosenstiel SF. Contemporary Fixed Prosthodontics. 2nd ed. Nodia: Mosby; 1998. p. 604.
12. Buchanan WT, Svare CW, Jurner KA. The effect of repeated firings and strength on marginal distortion in two ceramometal systems. J Prosthet Dent 1981;45:502-6.
13. Holmes J Robert. Marginal fit of electroformed ceramometal crowns. J Prosthod 1996;5:111-4.
14. Juntavee N, Nathanson D, Giordano R. Marginal fit of Captek and conventional metal-ceramic restorations. J Dent Res (Special Issue) 74, 1995, Abstract No. 167.
15. Hung SH, Hung KS, Eick JD, Tirade, Chappel RP. Marginal fit of porcelain fused to metal and two types of ceramic crown. J Prosthet Dent 1990;63:26-31.
16. Petteno D, Schierano G, Bassi F, Bresciano ME, Carossa S. Comparison of marginal fit of three different metal- ceramic systems: An *in-vitro* study. Int J Prosthodont 2000;13:403-8.