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Original Research

Influence of Cast Surface Finishing Process on Metal-Ceramic Bond Strength –In Vitro Study

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ABSTRACT

Because of the inherently brittle nature of ceramic, the susceptibility to their failure was identified at localized areas of high stress concentration on the ceramic surface, metal-ceramic interface or within the microstructure. The aim of this study was to investigate the influence of different cast surface finishing process on metal-ceramic bond strength.

Introduction

Increased patient awareness of esthetics has promoted research into understanding the brittleness and tendency of all-ceramic crowns to fracture under masticatory forces in service. One approach was to support the esthetic porcelain utilising a metal and the porcelain veneered to the whole metallic crown, or was applied only as a facial veneer. A second approach relied on improving the strength of existing porcelains, leading to the use of a strengthened porcelain or glass ceramic as a reinforcing core rather than a metal substructure.[1]

Dental ceramics are considered chemically inert restorative materials. However, many factors such as the composition, microstructure, chemical properties of the ceramic materials, erosive or acidic agents, exposure time, and the temperature, may influence the durability of dental ceramics.[2]

Ceramic for dental reconstructive work are multiphase silicate ceramics, glass ceramics or monophased glasses with varying compositions.[3,4] Structure composed of ceramic layers on a metal frame combined the strength of a metal substrate (dental alloy) with esthetic of a ceramic. Because of their inherently brittle nature susceptibility to their failure was identified at localized areas of high stress concentration on the ceramic surface, metal-ceramic interface or within the microstructure.[5]

A strong interface should provide sufficient stress transfer between the individual laminates to allow the applied loads to be transferred and accommodated. Conversely, a weak interface will frequently result in failure by a process of delaminating under an applied load possibly arising from crack initiation and propagation within and along the layer. [6]

Oxidation heat treatment of the metal is used to remove the entrapped gas, eliminate surface contaminants, and form the metal oxide layer. An alloy is deliberately given an oxidation treatment prior to ceramic application or

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whether it oxidizes during the portion of the firing cycle before flow of the ceramics begins, the fusing ceramics comes into immediate contact with oxide rather than with metal surface.[7,8]

The longevity of metal-ceramic restorations depends on the formation of a stable adhesive layer between the two materials. The adhesion mechanism between the metal and ceramic has not been completely defined, but it is believed to generally result from suitable oxidation of the metal and inter-diffusion of ions between the metal and ceramic.[9,10,11,12]

Airborne-particle abrasion of bonding surfaces increases the metal surface energy by improving the wettability of the opaque ceramic and consequently, the bond strength, through micromechanical bonding. Aluminium oxide (Al_2O_3) particles are the most commonly used air-abrasion particles for this purpose.[13]

MATERIALS AND METHODS

A total forty two plates, metal fused to ceramic samples of length 25mm width 3mm and thickness 2mm were prepared. Sandblasting was done with 100 and 250 µm aluminium oxide (Bego, Bermen Germany). Metal used was bellabond bego Germany. Ceramic used was VITA VMK MASTER (Bego, Bermen Germany) and the bonding agent used in group v was Tetric N Bond (Ivoclar Vivadent Bendererstr. Schaan, Liechtenstein.)

Fabrication of samples:

Forty two metal cast plates of 25x3x0.5 mm were casted according to manufacturer's instruction. These samples were grouped into six groups, the samples were cleaned and handled in same direction, and the surfaces to which ceramic was applied were treated by different procedures and combinations of procedures. Sandblaster used for sandblasting. (Santer Labo 16 – Confident Sales Pvt. Ltd.)

Grouping of samples:

Forty two metal cast plates were grouped into six groups.

Ist Group: oxidation of metal plate, then sandblasting with 100 µm aluminium oxide (Al_2O_3).

IInd Group: oxidation of metal plate, then sandblasting with 250 µm aluminium oxide (Al_2O_3).

IIIrd Group: sandblasting of metal plate with 100 µm aluminium oxide (Al_2O_3),then oxidation and again sandblasting of metal plate with 100 µm aluminium oxide (Al_2O_3).

IVth Group: sandblasting of metal plate with 250 µm aluminium oxide (Al_2O_3), then oxidation and again sandblasting of metal plate with 250µm aluminium oxide (Al_2O_3).

Vth Group: sandblasting of metal plate with 100 µm aluminium oxide (Al_2O_3),then oxidation and again sandblasting of metal plate with 100 µm aluminium oxide (Al_2O_3),then application of bonding agent.

VIth Group: sandblasting of metal plate with 100 µm aluminium oxide (Al_2O_3)then oxidation and again sandblasting of metal plate with 100 µm aluminium oxide (Al_2O_3) ,then etching with Hydrochloric acid solution. (50 ml distilled water and 50 ml of 32% hydrochloric acid). After etching samples were washed in distilled water and then in the compound of ethyl alcohol and acetone in the ratio of 1:1.

Then on the middle of metal plates ceramics (VITA VMK MASTER - Bego, Bermen Germany) was fired in the length of (8×3×1 mm). Specimens were fired in Ceramic Furnace Averon-007 SB (7x) (Ekaterinburg Russia) according to the manufacturer's directions. The following dentine porcelain firing schedule was applied: initial temperature, 403°C ,rate of rise, 60'per minute; final glazing temperature, 880X,Vacuum was initiated at 500°C and released at 880°C. Samples were fired only

Table 1: Group- wise distribution of the samples under study

Group distribution	Total no.
Group I	7
Group II	7
Group III	7
Group IV	7
Group V	7
Group VI	7

once. Surfaces were steam cleaned (Vaporklein Machine, Ivoclar). After fabrication, the specimens were finished with ceramic bur SHOFU Ceramaster (San Marcos USA) to flatten the ceramic surface and make it uniform.

A thin layer of surface glaze (Vita Akzent Plus Sackingen Germany) was mixed thoroughly and applied in the usual manner using a brush. Glazing was accomplished using an initial temperature of 403°C raised at a rate of 80X per minute to 890°C, The samples were glazed using a rapid firing cycle.

All specimens were tested with a 3-point bending test with a universal testing machine (Times Shijin Group-China, Model- WDE-5E).

The samples were set so that the surface with ceramics is turned opposite to the pin and the metal parts resting on the supports. The shift of pin was constant during testing and the testing was continued till the fracture that is till the full separation of the ceramics from the metal occurs.

The separation always started at the end of the sample and propagates towards the middle. Testing procedure was carried out according to the standard guidelines given in ISO 9693.

After testing the samples types of fracture surfaces were examined by the scanning electronic microscope (**FEI Quanta 200,Hillsboro-USA)**.

Data was analysed statistically by ANOVA, Post Hoc Tests (Bonferroni procedure) and Multiple Comparisons.

RESULTS AND OBSERVATIONS

The results in the present study are tabulated as follows:

Table no.1: Represents the group wise distribution of the samples under study.

A total of forty two samples were distributed into six groups each with seven samples.

Groups Were Coded As Group I, Group II, Group III, Group IV, Group V and Group VI.

Table No.2 And Graph No.1: Represents The Force At Which There Is Interfacial Fracture Between Metal And Ceramic.

The table no.2 and graph I shows the different forces at which the group samples have the fracture at interfacial surface the maximum force for fracture was seen in Group IV sample no.5 whereas the lowest force was noted in the Group VI sample no. 5.

Table no. 3: Represents the maximum force in a group.

Table no.3 represents the maximum force load in a group required to separate or initiate the interfacial fracture between the metal and ceramic. Maximum force was noted in the Group IV in which sandblasting of metal plate was done with 250 µm aluminium oxide (Al_2O_3),then oxidation and again sandblasting of metal plate with 250µm aluminium oxide (Al_2O_3),while lowest force was recorded in Group VI in which sandblasting of metal plate was done with 100 µm aluminium oxide (Al_2O_3),then oxidation and again sandblasting of metal plate with 100 µm aluminium oxide (Al_2O_3),then etching with hydrochloric acid solution. (50 ml distilled water and 50 ml of 32% hydrochloric acid). After etching

Table 2: Represents the Force At Which There Is Interfacial Fracture Between Metal And Ceramic

Sr. No. (Samples)	Force in Newtons					
	Group I	Group II	Group III	Group IV	Group V	Group VI
1	38.62	48.28	42.48	59.28	36.6	34.28
2	36.12	52.2	40.76	54.16	36.86	36.9
3	38.24	45.92	42.46	58.9	39.34	36.16
4	39.2	46.63	41.58	56.48	38.22	34.14
5	37.92	49.76	43.72	62.92	35.75	38.92
6	35.14	54.24	44.34	60.14	35.82	38.82
7	37.4	48.38	42.9	60.82	37.46	34.4

samples were washed in distilled water and then in the compound of ethyl alcohol and acetone in the ratio of 1:1.

Table no. 4 and Graph no.2 Represents intergroup comparison mean scores and standard deviation in a group after application of force in Newton's.

- Mean force to fracture the interface between metal and ceramic in Group I was 37.52 Newtons, std. deviation was 1.43.
- Mean force to fracture the interface between metal and ceramic in Group II was 49.34 Newtons, std. deviation was 2.98.
- Mean force to fracture the interface between metal and ceramic in Group III was 42.60 Newtons, std. deviation was 1.21.
- Mean force to fracture the interface between metal and ceramic in Group IV was 58.95 Newtons, std. deviation was 2.88.
- Mean force to fracture the interface between metal and ceramic in Group V was 37.15 Newtons, std. deviation was 2.07.
- Mean force to fracture the interface between metal and ceramic in Group I was 36.23 Newtons, std. deviation was 8.52.
- Mean force required for fracture of the interface between the metal and ceramic irrespective of Group was 43.63 Newtons and std. deviation was 1.31.

Table no. 5 Multiple Comparisons between the groups after treatment of samples by different surface finishing procedure, Test applied ANOVA with Post hoc.

The group was compared with other groups and the mean difference was significant at the ($p<0.05$) level.

- On comparison of Group I with other groups there was statistically significant difference between Group II, Group III, Group IV.

- On comparison of Group II with other groups there was statistically significant difference with other groups.
- On comparison of Group III with other groups there was statistically significant difference with other groups.
- On comparison of Group IV with other groups there was statistically significant difference between all groups except Group no.VI.
- On comparison of Group V with other groups there was statistically significant difference between Group I, Group III, Group IV.
- On comparison of Group VI with other groups there was statistically significant difference between Group II, Group III .

SEM IMAGES;

The SEM revealed the surface topography of the prepared samples. The sandblasted samples exhibited a roughened irregular surface and with evidence of striations in etched samples. In our study, examination of the metal specimens revealed a mixed adhesive-cohesive mode of failure for Group I,Group V ,Group VI and adhesive failure for Group II, Group III, Group V.

IMAGE 1A, 2A, 3A, 4A, 5A, 6A Shows the long view that is long view magnification while IMAGEs 1B, 2B, 3B ,4B ,5B ,6B Shows the close view magnification.

IMAGE 1a- This view reveals the smooth surface on appearance.

IMAGE 1b- This view reveals the irregular surface with pits measuring 1mm on measuring scale. On further magnification the pores were visible.

IMAGE 2a- This view reveals some roughness on surface.

IMAGE 2b- This view reveals the spot on surface,the pores were 300µm on measuring scale.

IMAGE 3a- This view reveals the some rough surface on appearance.

IMAGE 3b- This view reveals the irregular surface with pits measuring 300µm on measuring scale. The pores visible were more in numbers.

IMAGE 4a- This view reveals the circular form of damage due to more force application.

IMAGE 4b- This view reveals the irregular surface with pits measuring 300µm on measuring scale. The pores visible were more in number in compare to other samples. The roughness was more in this sample.

IMAGE 5a- This view reveals the cracks on the surface of the sample.

IMAGE 5b- On magnification there was circular form of damage with pores measuring 500µm on measuring scale.

IMAGE 6a- This view reveals the scaling on the sample surface.

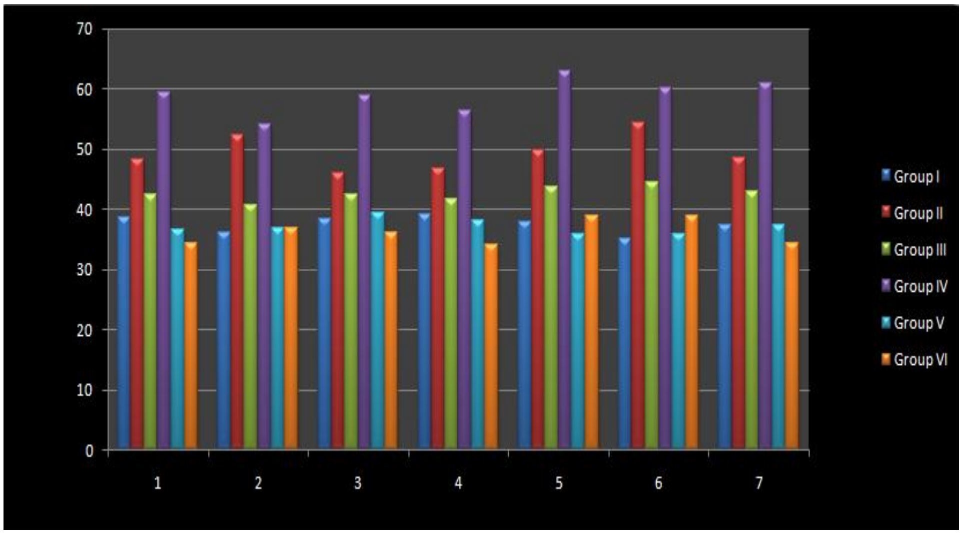
IMAGE 6b- on magnification cracks were seen in the samples, reveals the breakage of sample on low force.

Discussion-

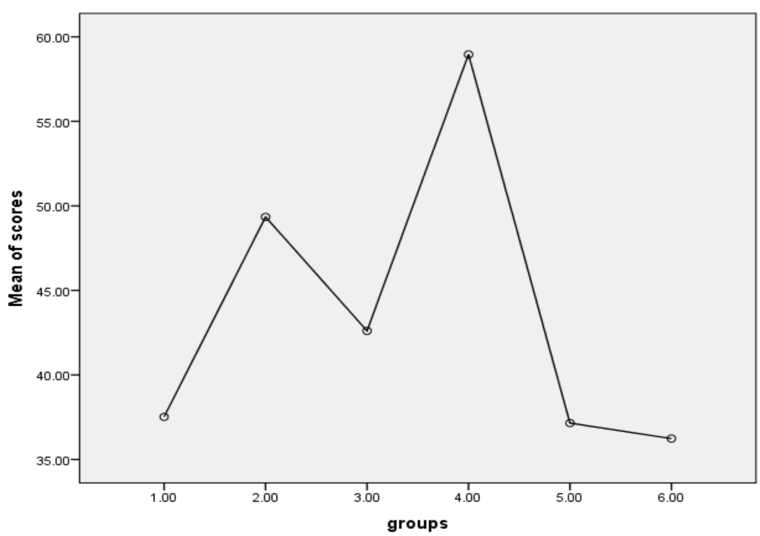
Because of their inherently brittle nature susceptibility to their failure was identified at localized areas of high stress concentration on the ceramic surface, metal-ceramic interface or within the microstructure.[5] A strong interface should provide sufficient stress transfer between the individual laminates to allow the applied loads to be transferred and accommodated. Conversely, a weak interface will frequently result in failure by a process of delaminating under an applied load possibly arising from crack initiation and propagation within and along the layer .[6]

The surface treatment of metal substrates plays an important role in the formation and thickness of the interactive oxide layer which directly affects the bond between ceramic and metal. It was reported that the

Graph 1: Represents the Force (in Newton) At Which There Is Interfacial Fracture Between Metal And Ceramic.



Graph 2: Means Plots



thinner the oxide layer on the metal surface, the stronger the bond between the two materials.[14]

Evidence suggests that this roughened surface can also provide mechanical interlocking and increase the surface area for porcelain-metal bonding.[15]

Sandblasting has been shown to affect oxide formation; the oxide layer formed before sandblasting differs from the one obtained after sandblasting.[16] Investigations have found that a roughened metal surface yields the highest porcelain-metal bond strengths.[17]

The metal oxides have been studied extensively and are thought to play an important role in ceramometal bonding.[9,18-22]

It is widely believed that the fusing porcelain dissolves away the oxide originally formed and produces an interaction zone responsible for the formation of a bond.[23]

Graham suggested final finishing process in the order: sandblasting, grinding, sandblasting and oxidation.[24]

Smoother surface achieved the lowest values of bond strength and bonding agent did not improve bond strength because of hermitical sealing of cast surface. [25]

Conclusion

It is difficult to quantify the real bond strength because in vitro testing is not usually in correlation with ceramic breakdown in function.

It can be concluded that the analysis of all the parameters used in assessing the strength of the bond between metal and ceramics has confirmed that the bond is the strongest in the surface treatment procedure sandblasting with 250 µm (Al_2O_3), oxidation and sandblasting again with 250 µm and significantly weaker in the etched sample. The metal samples revealed an adhesive mode of failures on the most part of surface and adhesive-cohesive on the edges.

Table 3 : Maximum force applied in a group sample leading metal-ceramic interface fracture.

Group	Maximum Force (Newtons)
1.00	39.20
2.00	54.24
3.00	44.34
4.00	62.92
5.00	39.34
6.00	38.92
Total	62.92

Table No 4: Intergroup comparison mean scores and standard deviation in a group after application of force in Newton's

Groups	N	Mean	Std. Deviation	Std. Error	95% Confidence Interval for Mean		Minimum
					Lower Bound	Upper Bound	
1.00	7	37.5200	1.43467	.54225	36.1932	38.8468	35.14
2.00	7	49.3443	2.98473	1.12812	46.5839	52.1047	45.92
3.00	7	42.6057	1.21324	.45856	41.4837	43.7278	40.76
4.00	7	58.9571	2.88184	1.08923	56.2919	61.6224	54.16
5.00	7	37.1500	1.30081	.49166	35.9470	38.3530	35.75
6.00	7	36.2314	2.07836	.78555	34.3093	38.1536	34.14
Total	42	43.6348	8.52446	1.31535	40.9784	46.2912	34.14

Table 5: Multiple Comparisons between the groups after treatment of samples by different surface finishing procedures by Test applied ANOVA with post hoc.

Groups	Mean	SD	P value
1	37.52[a]	1.43467	0.001
2	49.34[b]	2.98473	
3	42.60[c]	1.21324	
4	58.95[d,f]	2.88184	
5	37.15[a,e]	1.30081	
6	36.23[a,f]	2.07836	

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Figures

Figure -1 Material used

Figure-2 Armamentarium used

Figure 3- Sandblasting Machine (SANTER LABO 16 CONFIDENT PVT.LTD)

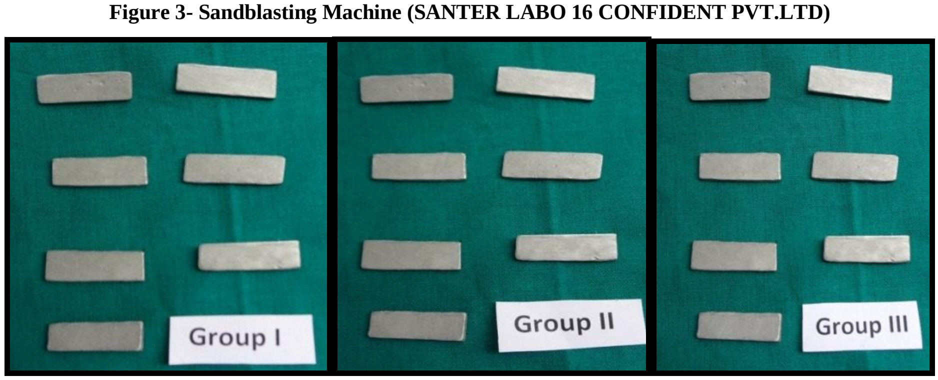


Figure 4– Metal plates after different surface finishing procedure

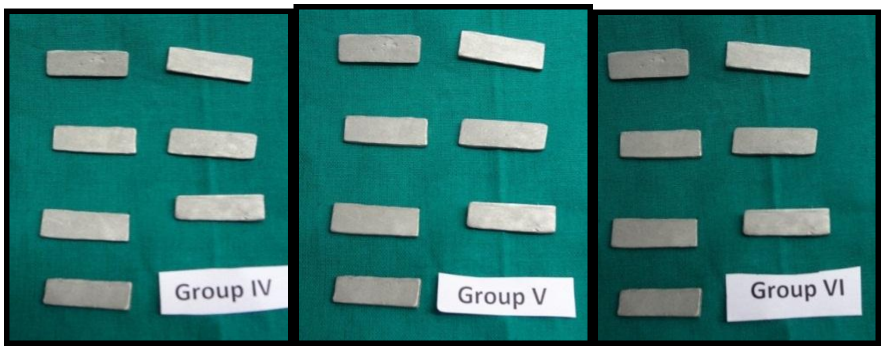


Figure 5- Opaque layer application

Figure 6- (a)Opaque on plates

Figure 6- (b) Opaque on plates

Figure7- (a) Opaque layer after heat treatment

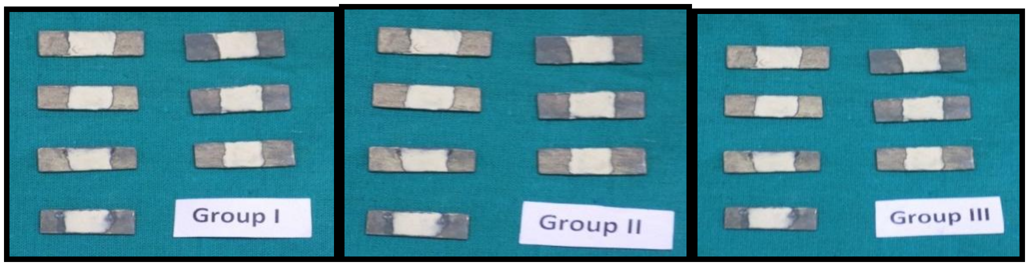


Figure 7- (b) Opaque layer after heat treatment

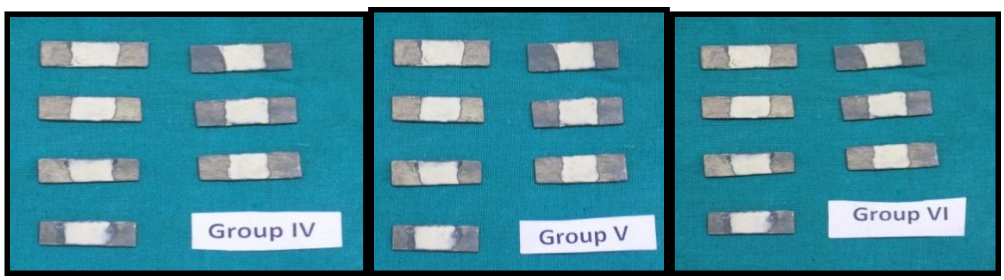
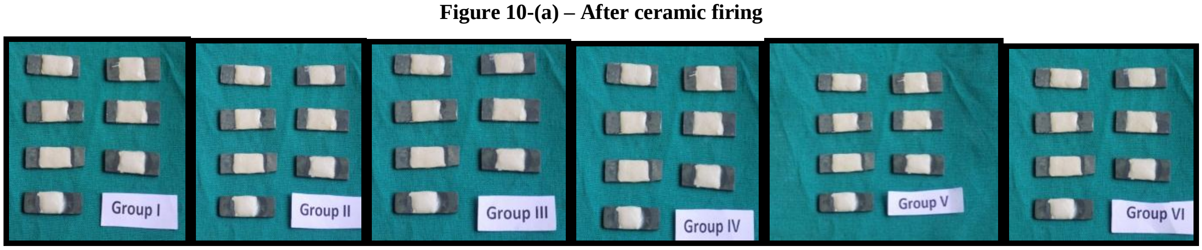


Figure 8- Ceramic firing in furnance

Figure 10-(a) – After ceramic firing



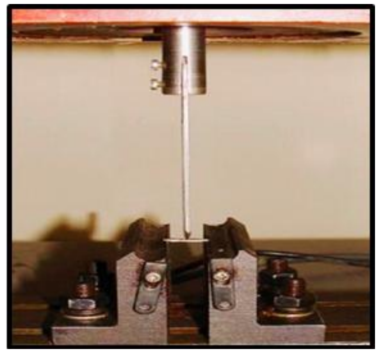
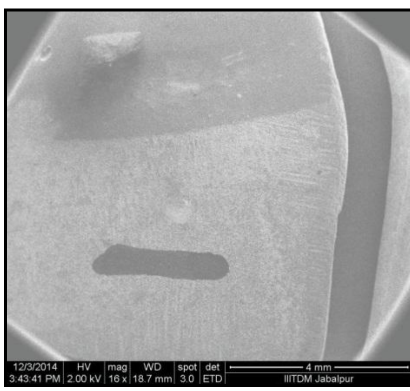
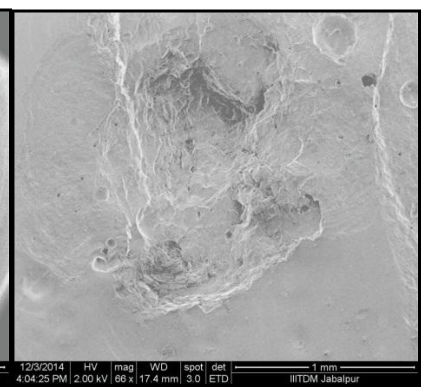


Figure 11- Plate in universal testing machine

SEM PHOTOGRAPHS

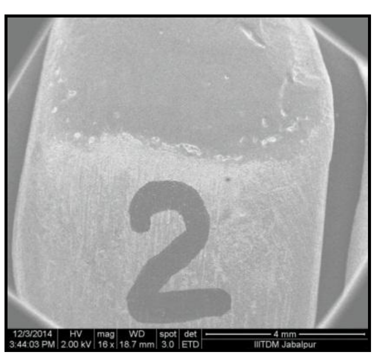
IMAGE 1 (a and b)

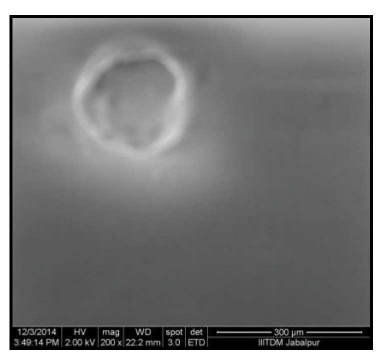




A **b**

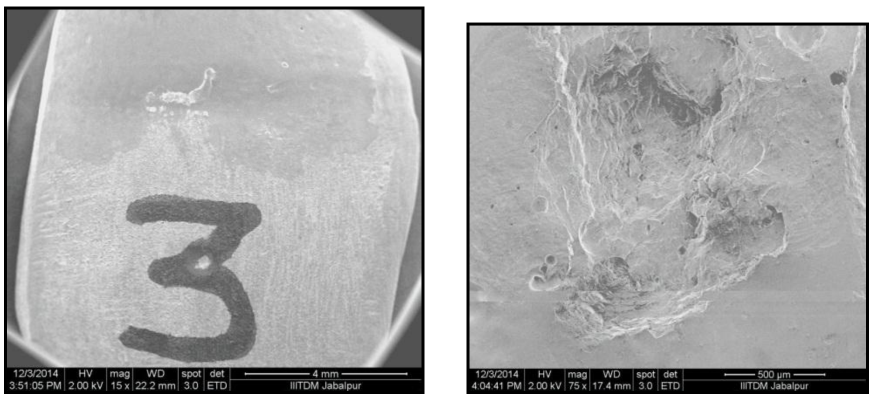
IMAGE 2 (a and b)





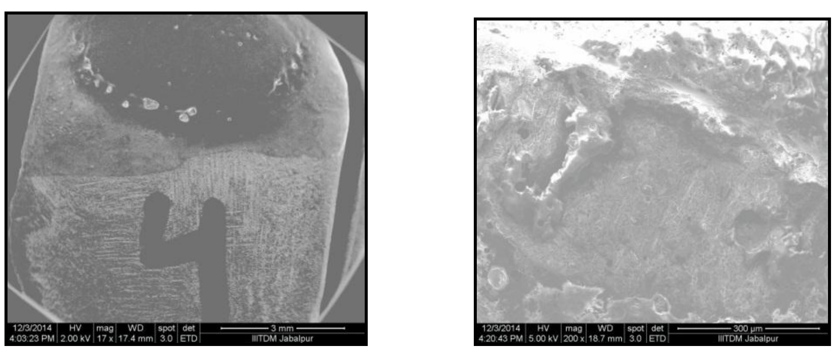
A **b**

IMAGE 3 (a and b)



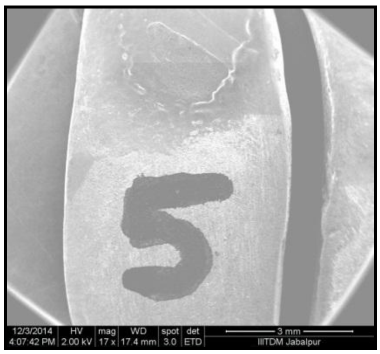
A b

IMAGE 4 (a and b)



A b

IMAGE 5 (a and b)



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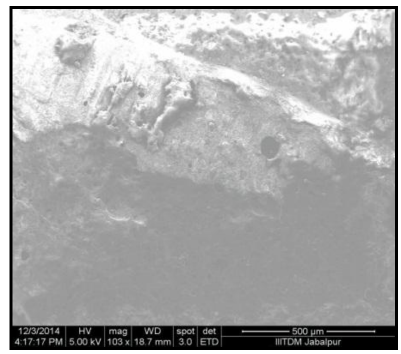
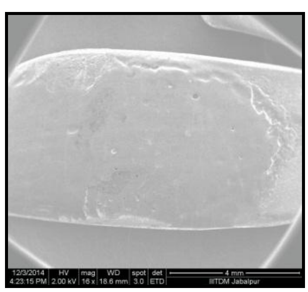


b

IMAGE 6 (a and b)



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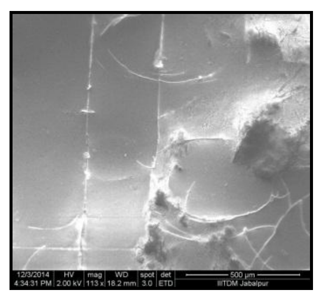

b