



Laser Glazing Interaction with Dental Ceramics

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ABSTRACT

Objectives: The aim of this study was to investigate the hardness, morphology and structure of dental porcelain and In-Ceram Alumina after conventional glazing, XeCl excimer and CO₂ laser glazing techniques at two energy densities.

Materials and Methods: The materials used were conventional feldspathic porcelain Vitadur N and Vita In-Ceram Alumina. The hardness was measured using Vickers microhardness tester. SEM and X-ray diffraction analysis of the surface structure were conducted to detect the surface changes.

Results: The hardness of the porcelain specimens was not significantly increased with 2 watt CO₂ laser and 1.5 Joule/cm² excimer laser glazing. Also, the hardness of in-ceram alumina was not significantly increased with 2 and 10 watt CO₂ laser glazing. On the contrary, the hardness of porcelain was significantly increased with 10 watt CO₂ and 6.2 Joule/cm² excimer lasers, as well as the in-ceram alumina was significantly increased with excimer laser glazing. Scanning electron microscopy declared an increase in homogeneity and smoothness of laser glazed specimens especially those glazed with higher power setting of CO₂ laser and higher energy density of excimer laser when compared to the conventionally glazed specimens. X-ray diffraction charts of the control and laser glazed specimens were nearly identical, indicating that laser glazing had no effect on their internal microstructure.

Conclusion: It was concluded that, laser glazing improves the surface hardness and smoothness of ceramic surfaces without affecting their internal structures.

Clinical relevance: Laser technology can be used for glazing of ceramic restorations to improve its properties.

Keywords: Dental porcelain, In-ceram alumina, Hardness, Structure, Morphology.

INTRODUCTION

Today, as demands for esthetic dental restorations continue, new technologies will improve the material properties and develop new methods for its use¹. The development of ceramic systems more resistant to chewing stresses has allowed the fabrication of all ceramic restorations without metallic infrastructure².

The high crystalline content of metal-free ceramic framework prostheses and implant abutments made from glass-infiltrated ceramic core is often exposed to the oral environment without ceramic veneer. This is especially true for inadequate tooth preparation and bonded prostheses, in which the palatal extension of the prosthesis is usually uncovered³. Besides, the veneer layer over glass-infiltrated ceramic core may be subjected to brittle fracture and removed through use and accidental damage causing direct contact between the core material and the opposing human enamel or restoration⁴. The bulk of core materials could also be exposed in non-aesthetic areas that require additional strength, such as the connector regions⁵. In these cases, the framework ceramic surface should be as smooth as possible, with the aim of minimizing the bacterial colonization and dental biofilm formation⁶.

In fact, surface compression layer was found to occur on a wide range of ceramic materials following different treatment processes that acts to strengthen ceramic material. It can be achieved by thermal tempering, machining and polishing and the application of a glazing layer with a

lower coefficient of thermal expansion than the adjacent ceramic material⁷.

The special characteristics of XeCl excimer laser radiation, particularly its very high energy density and the possibility of guiding the laser beam through flexible quartz glass fibers, made this laser system very promising technique for working ceramic materials⁸. The CO₂ laser is well suited for the treatment of porcelain materials because its emission wavelength is almost totally absorbed by porcelain⁹. It was suggested that laser treatment of ceramic surfaces inhibits the formation of microcracks, leading to greater mechanical resistance of the ceramic¹⁰. Therefore, the aim of this study was to evaluate the surface hardness, surface morphology and structure of conventional dental porcelain and in-ceram alumina materials after glazing using XeCl excimer and CO₂ lasers with different intensities. The hardness was measured using Vickers microhardness tester. SEM and X-ray diffraction analysis of the surface structure were conducted to detect the surface changes.

MATERIALS AND METHODS

The materials used were conventional feldspathic porcelain Vitadur N (VITA Zahnfabrik H. Rauter GmbH and Co.KG. Postfach1338D-17880 Bad Sackingen. Germany) and Vita In-Ceram Alumina (VITA Zahnfabrik H. Rauter GmbH & Co. KG Postfach1338D-79704 Bad Sackingen. Germany). The main

composition of the dental porcelain is SiO₂, Al₂O₃, CaO, K₂O, Na₂O, TiO₂, ZrO₂, SnO₂, Rb₂O, BaO, ZnO, and other minor elements. The main composition of the In-Ceram Alumina is SiO₂, Al₂O₃, B₂O₃, TiO₂, La₂O₃, CeO₂, CaO.

Hardness testing

Preparation of porcelain specimens

The porcelain powder was mixed with distilled water to form slurry. The mixed slurry was loaded and condensed into a stainless steel mold (10 mm diameter and 2 mm thickness)¹¹. The water was expressed during condensation and periodically blotted away using an absorbent tissue. The specimens were placed on a firing tray, dried and sintered in a vacuum furnace according to the manufacturer's recommendations at 940° C¹². A total number of 25 disc-shaped specimens were prepared and divided as follows:

1. Five specimens of conventional dental porcelain were auto-glazed at 940 °C for 1 minute as a control group¹³.

2. Ten specimens of conventional dental porcelain were glazed with CO₂ laser device (Novapulse LX-20SP, Luxar, Bothell, Wash) with two different power settings, 2 and 10-watt, 5 specimens each in the super pulse mode (15 msec, 2Hz).

3. Ten specimens of conventional dental porcelain were glazed with excimer laser device (Lambda-physik, Model Optex, Germany) filled with xenon and chlorine as the laser medium, emitting ultraviolet radiation at a wavelength of 308 nm with two different energy densities, 1.57 and 6.28 J/cm², 5 specimens each. The maximum energy of the radiation was 120 mJ, and the pulse duration was in the range of 60 ns. The repetition rate was 40 laser pulses per second.

Preparation of in-ceram alumina specimens

A total of 25 disc-shaped specimens were prepared from aluminum-oxide powder using stainless steel mold. Impression of the metal mold was made with the putty/wash technique using vinyl polysiloxane impression (Zetaplus; Zhermack Spa, Badia Polesine, Rovigo, Italy). The impression was poured with in-ceram special plaster (VITA Zahnfabrik). The aluminum oxide powder was mixed with a special liquid (Vita In-Ceram Alumina mixing liquid; Vita Zahnfabrik) according to the manufacturer's recommendations. The slurry mixture was then painted over the special plaster die and fired at 1120°C in the oven (In Ceramat; Vita Zahnfabrik) for 10 hours. A glass powder (silicate-aluminum-lanthanum) was mixed with distilled water to form a mixture for glass infiltration. The mixture was applied to the aluminum oxide frame work and fired in the furnace for 4 hours at 1100°C. Finally, the excess glass was removed using airborne-particle abrasion with 50µm alumina powder¹⁴. The prepared specimens were divided as follows:

1. Five specimens of in-ceram alumina were heat treated with two cycles; the first at 960°C/1 min and the second at 940°C/1 min as recommended by the manufacturer for auto-glazing of the veneering porcelain used on in-ceram alumina (as a control group)¹⁵.

2. Ten specimens of in-ceram alumina were glazed with CO₂ laser at two different power settings 2 and 10 watt (5 specimens each).

3. Ten specimens of in-ceram alumina were glazed with excimer laser with two different energy densities, 1.57 and 6.28 J/cm², (5 specimens each).

Before conducting the hardness test, surfaces of all specimens were thoroughly inspected by a magnifying lens to exclude specimens with any surface defect. A micro-

hardness tester (Digital Vickers Micro-hardness tester (FM-7) Japan) using a Vickers indenter and a load of 200 g for 30 seconds was used. Five measurements were made for each specimen, and the mean value of each specimen was calculated¹⁶.

Scanning electron microscope analysis

A total number of 10 photomicrographs were taken to 10 representative specimens (one specimen for each group) to observe the surface topography. The specimens were sputter-coated with 25 to 30 μm of gold (Hummer VII Sputtering System; Anatech Ltd, Alexandria, Va) and examined at original magnification $\times 200$ with scanning electron microscopy (Electron probe micro-analyzer operating at 30 KV Joel Type JXA-840A, Japan)⁵.

X-ray diffraction analysis

A total number of 10 specimens, 5 for each material were divided and treated as those used for the scanning electron microscopy. The specimens were then placed in the holder of a Siemens Diffractometer (Diffractometer D5000, Siemens, Germany) and scanned using Cu $K\alpha$ X-ray from 20 to 40° 2 θ degrees; a step size of 0.04° and 5 s-step interval were used⁵.

Statistical analysis

The data was collected and subjected to one-way ANOVA and LSD testes to determine the significant difference between the different glazing methods at $P \leq 0.05$.

RESULTS

Hardness

Mean hardness values of porcelain specimens glazed with conventional, CO₂ and excimer lasers are presented in Table 1. One-way ANOVA demonstrated a significant difference among the different

glazing methods of dental porcelain ($P \leq 0.001$). The statistical analysis of the results revealed a significant difference between the conventionally glazed group and 10 watt CO₂ laser as well as 6.2 Joule/cm² excimer laser groups ($P \leq 0.05$). Also, there was a significant difference between the two groups of CO₂ laser (2 watt, 10 watt), as well as between the two groups of excimer laser (1.5 Joule/cm² and 6.2 Joule/cm²) ($P \leq 0.05$). A significant difference was found between 2 watt CO₂ laser and 6.2 Joule/cm² excimer laser glazed groups, as well as between 10 watt CO₂ laser and 1.5 Joule/cm² excimer laser glazed groups ($P \leq 0.05$). On the other hand, there was no significant difference was found between the control group, 2 watt CO₂ laser and 1.5 Joule/cm² excimer laser groups. Also, there was no significant difference between 10 watt CO₂ laser and 6.2 Joule/cm² excimer laser groups.

Mean hardness values of in-ceram alumina specimens glazed with the different methods are presented in Table 2. One-way ANOVA demonstrated a significant difference in the mean hardness values of in-ceram alumina specimens glazed with different methods ($P \leq 0.001$).

The statistical analysis of the results revealed a significant difference between the conventionally glazed group and 1.5 Joule/cm² excimer laser glazing, as well as between the conventional glazing and 6.2 Joule/cm² excimer laser ($P \leq 0.05$). There was a significant difference between 2 watt CO₂ laser group and those glazed with the two groups of excimer laser (1.5 Joule/cm² and 6.2 Joule/cm²) ($P \leq 0.05$). A significant difference was found between 10 watt CO₂ laser group and 6.2 Joule/cm² excimer laser group ($P \leq 0.05$). On the other hand, there was no significant difference between the conventional glazing and those of CO₂ laser groups ($P > 0.05$). At the same time, there was no significant difference between 10

watt CO₂ laser and 1.5 Joule/cm² excimer laser, as well as between the two groups of excimer laser ($P>0.05$).

Scanning electron microscope

SEM observations of the conventionally glazed porcelain specimen showed uneven and rather slightly granular surface feature (Figure 1). The scanning electron micrographs of CO₂ laser glazed porcelain specimens had rather more homogenous surfaces than the control specimens. In case of low power setting (2 watt), the peaks in the structure of the irradiated surface have been in part melted together. However, there were wide areas with fissures between the former peaks of the surface structure. On the other hand; at higher power setting (10 watt), the specimen had smoother surface and much more homogenous structure. The scanning electron micrographs of excimer laser glazed porcelain specimens appeared to have larger areas of fusion with much more smoothness and homogeneity in the surfaces especially with higher energy density (6.2 J/cm²). The arrows referred to the emergence of shallow splodgy areas of melting (Figure 2).

Figure 3 illustrated the scanning electron micrograph of conventionally glazed In-Ceram Alumina specimen. The figure showed slight alterations of the surface structure. The surface had wide areas without any changes except the presence of small crater like irregularities in and around glazed patches with numerous cracks formation. The surfaces of both CO₂ and excimer laser glazed In-Ceram Alumina specimens had much greater number of fused zones of melting and became more homogenous and smoother in shape with decreased number of craters and fissures especially at higher power setting of 10 watt CO₂ laser (Figure 4) and higher energy density of 6.2 J/cm² excimer laser.

X-ray diffraction analysis

The charts obtained from the specimens are presented in Figures 5 and 6 which are plots of relative intensity (counts per second) versus diffraction angle (2θ). The X-ray analysis of both porcelain and In-ceram alumina specimens detected diffraction peaks that corresponded to crystalline phases present in both materials indicating that the materials had predominantly crystalline structure.

In case of porcelain specimens, the charts showed the appearance of potassium aluminum catena-disilicate (leucite) crystals having a body-centered tetragonal lattice structure as indicated by the ASTM card # 22-675 (Figures 5a, b). The diffraction pattern of 2 watt CO₂ laser glazed specimen showed that there was no remarkable change in the intensities of potassium aluminum catena-disilicate bands when compared to those of the control specimen. On the other hand, the diffraction pattern of 10 watt CO₂ laser glazed specimen showed that there was a remarkable decrease in their intensities which appeared at (2θ) = 16.39°, 26.02°, 30.52° and 31.41° with their corresponding (d) space = 5.405Å, 3.424Å, 2.928Å and 2.847Å respectively (Figure 5 b). In case of 1.5 Joule/cm² excimer laser glazing, there was a remarkable decrease in the intensities which appeared at (2θ) = 30.52° and 31.41° with their corresponding (d) space = 2.928Å and 2.847Å respectively. Also, the intensities of the bands decreased at (2θ) = 30.52° and 37.96° with their corresponding (d) space = 2.928Å and 2.370Å respectively, in case of 6.2 Joule/cm² excimer laser glazing.

On the other hand, the charts of In-Ceram Alumina specimens dictated the appearance of aluminum oxide, corundum and calcium aluminum silicate bands having rhombohedral, hexagonal and triclinic structures respectively as indicated by the

ASTM card # 10-173 for both aluminum oxide and corundum and ASTM card # 20-20 for calcium aluminum silicate (Figure 6 a & b). The diffraction patterns of both 2 watt CO₂ and 6.2 Joule/cm² excimer laser (Figure 6 b) glazed In-Ceram Alumina specimens demonstrated a remarkable decrease in the intensities of corundum and aluminum oxide bands appeared at $(2\theta) = 35.12^\circ, 43.36^\circ, 52.55^\circ, 57.46^\circ, 66.46^\circ$ and 76.82° with their corresponding (d) space = 2.552Å, 2.085Å, 1.739Å, 1.602Å, 1.405Å, and 1.239Å respectively, when compared to those of the control specimens. While in case of 10 watt CO₂ laser glazed specimen, the band intensity decreased more at $(2\theta) = 76.82^\circ$ and (d) space = 1.239Å. Also, the intensities decreased remarkably at $(2\theta) = 43.36^\circ, 52.55^\circ, 57.46^\circ$ and 76.82° with their corresponding (d) space = 2.085Å, 1.739Å, 1.602Å and 1.239Å respectively, in case of 1.5 Joule/cm² excimer laser glazing.

The diffraction pattern of 2 watt CO₂ laser glazed In-Ceram Alumina specimen showed that there was a slight increase in the intensity of calcium aluminum silicate band appeared at $(2\theta) = 9.39^\circ$ and (d) space = 9.402Å when compared to that of the control specimen. While in case of 10 watt CO₂ laser glazed specimen, the band intensity increased more at $(2\theta) = 9.33^\circ$ and (d) space = 9.464Å. In case of 1.5 joule/cm² excimer laser glazing, there was a very remarkable increase in the intensity which appeared at $(2\theta) = 9.37^\circ$ and (d) space = 9.426Å. Also, the intensity of the band increased remarkably at $(2\theta) = 9.31^\circ$ and (d) space = 9.490Å in case of 6.2 Joule/cm² excimer laser glazing (Figure 6 b).

DISCUSSION

The effect of laser glazing of both dental porcelain and in-ceram alumina materials on their properties differ according to variation in energy density of excimer laser and power setting of CO₂ laser.

Porcelain specimens which glazed with both 2 watt CO₂ laser and 1.5 Joule/cm² excimer laser showed an insignificant increase in their hardness values while those glazed with both 10 watt CO₂ laser and 6.2 Joule/cm² excimer laser showed a significant increase in their hardness values when all of them were compared to those subjected to conventional glazing. In-ceram alumina specimens exhibited an insignificant increase in their hardness values in all groups of glazed specimens except those glazed with both 1.5 and 6.2 Joule/cm² excimer laser.

The hardness results are in contrast with those of Mackert *et al.*, (1994) who indicated that the size of leucite particles in feldspathic porcelain increases during heating process with laser irradiation. This can increase the probability of microcracking thus decreasing the hardness of ceramic materials¹⁹.

The thermal effect of laser would melt a thin superficial layer of ceramic surface and this layer would fill in surface flaws, reducing their depth and blunting the flaw tips. This should provide an increase in hardness because, for a given ceramic material, strength and hardness would increase with decreasing flaw depth and sharpness¹⁸. The melted superficial layer of ceramic material has a lower thermal expansion coefficient than the leucite-rich interior. This would place the outer surface in compression when cooled. The compressive stress state would diminish the local tensile stress produced from applied loading at surface flaws, thereby necessitating the need for increased applied loading to initiate flaw propagation from the external surface²⁰.

Scanning electron microscope images declared the increase in homogeneity and smoothness of the surfaces of laser irradiated specimens especially those irradiated with higher power of setting of

CO₂ laser and higher energy density of excimer laser when compared to conventionally glazed specimens where more glassy matrix was dissolved resulting in an increase in fusion, homogeneity of crystallization and reduction of voids.

These results were attributed to the laser thermal effect which causes melting of a thin superficial layer of ceramics²¹. This leads to the deposition of high amounts of radiation energy in a well defined part of the ceramic surface over an ultrashort period of time, causing the accumulation of a very high energy density²². The radiation energy is thermalized and the temperature, in a thin superficial layer, rises²³. As the ceramic is manufactured with very small crystallites, its extremely low porosity may lead to scattering losses.²⁴ Schmage *et al.*, (2003)²⁴ found that a glazed surface layer on ceramics will be formed with Nd: YAG laser irradiation.

X-ray diffraction was performed to evaluate internal structure of the studied specimens. The diffraction patterns of both porcelain and In-Ceram Alumina specimens demonstrated sharp well-defined peaks which indicated that both materials having predominantly crystalline structure with very few broad bands which corresponded to the amorphous glassy phase. However, it is clear from the charts that the X-ray diffraction traces of both the control and laser glazed specimens are identical with each other; meaning that there was no change in the number of peaks nor their positions, indicating that laser glazing had no effect on the internal microstructure of both porcelain and in-ceram alumina and it was just a surface treatment. It was clear from the diffraction patterns of the specimens that the effect of laser glazing was just localized to changes in the intensities of the peaks.

In all X-ray diffraction patterns of porcelain specimens, the measured peak

positions belonged to peaks of potassium aluminum catena-disilicate (leucite) crystal which has a body-centered tetragonal lattice structure. The leucite peaks of 10 watt CO₂ laser, 1.5 Joule/cm² and 6.2 Joule/cm² excimer laser glazed porcelain specimens demonstrated a remarkable decrease in their intensities with the exception of 2 watt CO₂ laser glazed specimens which showed no change in their intensities. This could be explained by the thermal effect of laser in providing energy sufficient to make slight changes in the atomic positions or causing slight overlapping of some atomic planes over each others. In case of lower power setting of 2 watt CO₂ laser glazed specimens, the energy might be not sufficient enough to cause this effect.

The charts of in-ceram alumina specimens dictated the appearance of aluminum oxide, corundum and calcium aluminum silicate bands having rhombohedral, hexagonal and triclinic structures respectively. The peaks of both corundum and aluminum oxide in all In-Ceram Alumina specimens exhibited a remarkable decrease in their intensities. This could be explained as mentioned before in case of porcelain specimens by the thermal effect of laser in providing energy sufficient to make slight changes in the atomic positions or causing slight overlapping of some atomic planes over each others. However, calcium aluminum silicate bands exhibited a remarkable increase in their intensities in all In-Ceram Alumina specimens except 2 watt CO₂ laser glazed specimens which exhibited a slight increase in the intensities. This could be explained by the effect of thermal energy of laser in rearranging the atomic positions which increased the degree of ordering in the structures and consequently the intensities increased.

CONCLUSIONS

Depending on the results and within the limitations of this study, the following conclusions could be deduced:

1. The surface roughness of both dental porcelain and in-ceram alumina was decreased through glazing of dental ceramics by either CO₂ or excimer lasers.
2. The hardness of dental ceramics were increased with increasing the power setting and energy density of both CO₂ and excimer lasers.
3. In general, excimer laser exhibited more pronounced effect than CO₂ laser in improving the mechanical properties of both dental porcelain and in-ceram alumina.

The diffraction patterns dictated that laser glazing had no effect on the internal microstructure of both dental porcelain and in-ceram alumina but only making slight changes in atomic positions or increasing the degree of ordering between the atoms.

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Table 1. Mean hardness values (VHK) Kg/mm² of dental porcelain with different glazing methods

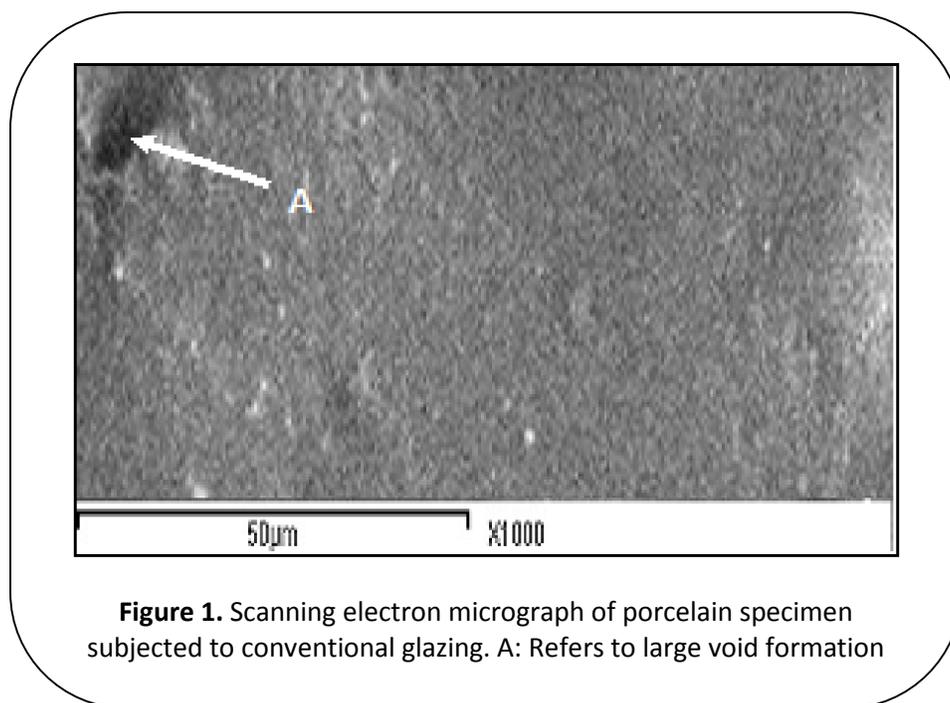
| Treatments | Mean ± SD | F-value | P-value | LSD |
|---|-------------------------------|---------|---------|-------|
| Conventional glazing (Control) | 422.6±34.3175 ^{DB} | 13.455 | 0.001 | 68.96 |
| 2 watt CO ₂ laser | 455.6 ± 63.2528 ^{CB} | | | |
| 10 watt CO ₂ laser | 539.1 ± 43.3307 ^A | | | |
| 1.5 Joule/cm ² excimer laser | 470.2 ± 41.5707 ^B | | | |
| 6.2 Joule/cm ² excimer laser | 595.0 ± 14.2824 ^A | | | |

Means with different superscripts are significantly different at P-value ≤ 0.05.

Table 2. Mean hardness values (VHK) Kg/mm² of In-ceram alumina with different glazing methods

| Treatments | Mean ± SD | F-value | P-value | LSD |
|---|-------------------------------|---------|---------|-------|
| Conventional glazing (Control) | 768.6±70.1413 ^{DC} | 7.652 | 0.001 | 119.2 |
| 2 watt CO ₂ laser | 796.6 ± 64.8984 ^C | | | |
| 10 watt CO ₂ laser | 860.8± 64.8128 ^{BDC} | | | |
| 1.5 Joule/cm ² excimer laser | 961.4 ± 92.4219 ^{AB} | | | |
| 6.2 Joule/cm ² excimer laser | 980.0 ± 87.4643 ^A | | | |

Means with different superscripts are significantly different at P-value ≤ 0.05.



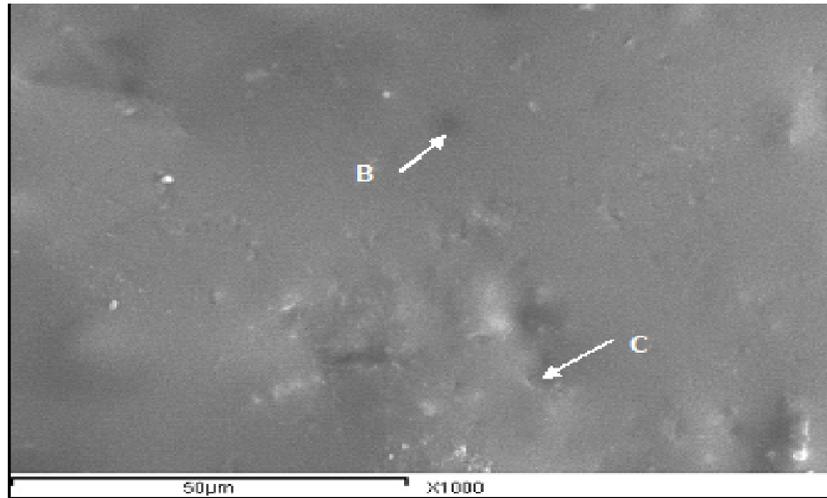


Figure 2. Scanning electron micrograph of porcelain specimen subjected to 6.2 Joule/cm² excimer laser glazing. B, C: refer to shallow splotchy areas of melting

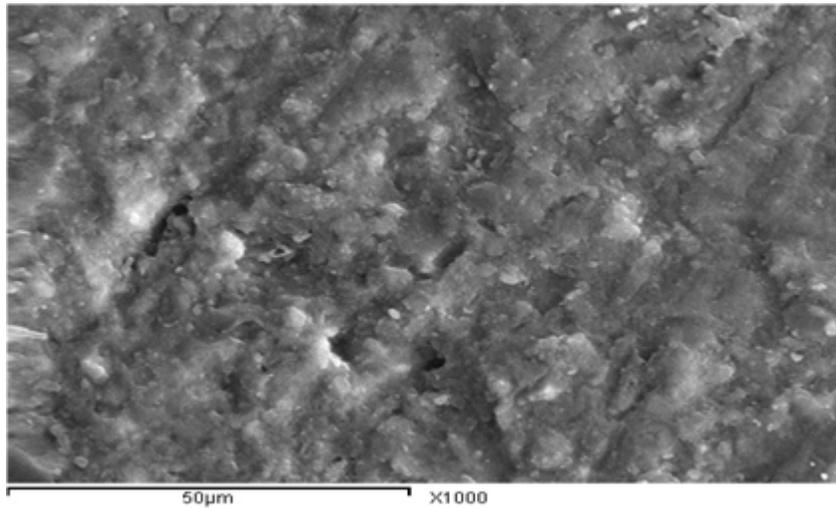


Figure 3. Scanning electron micrograph of In-ceram alumina specimen subjected to conventional glazing. D: refers to glazed patches, E: shows crater like irregularity and F: shows crack formation



Figure 4. Scanning electron micrograph of In-ceram alumina specimen subjected to 10 watt CO₂ laser glazing

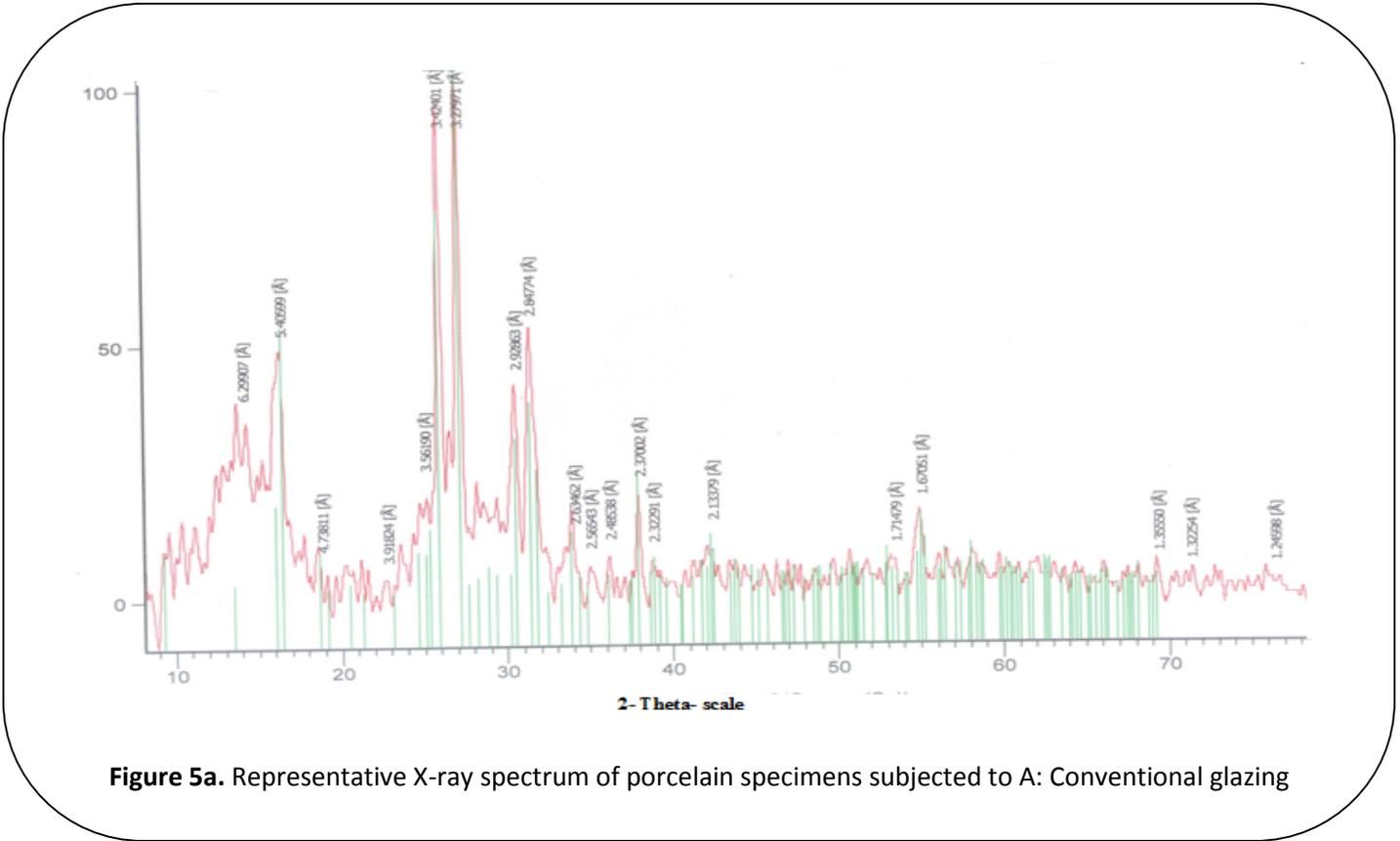


Figure 5a. Representative X-ray spectrum of porcelain specimens subjected to A: Conventional glazing

Counts per second

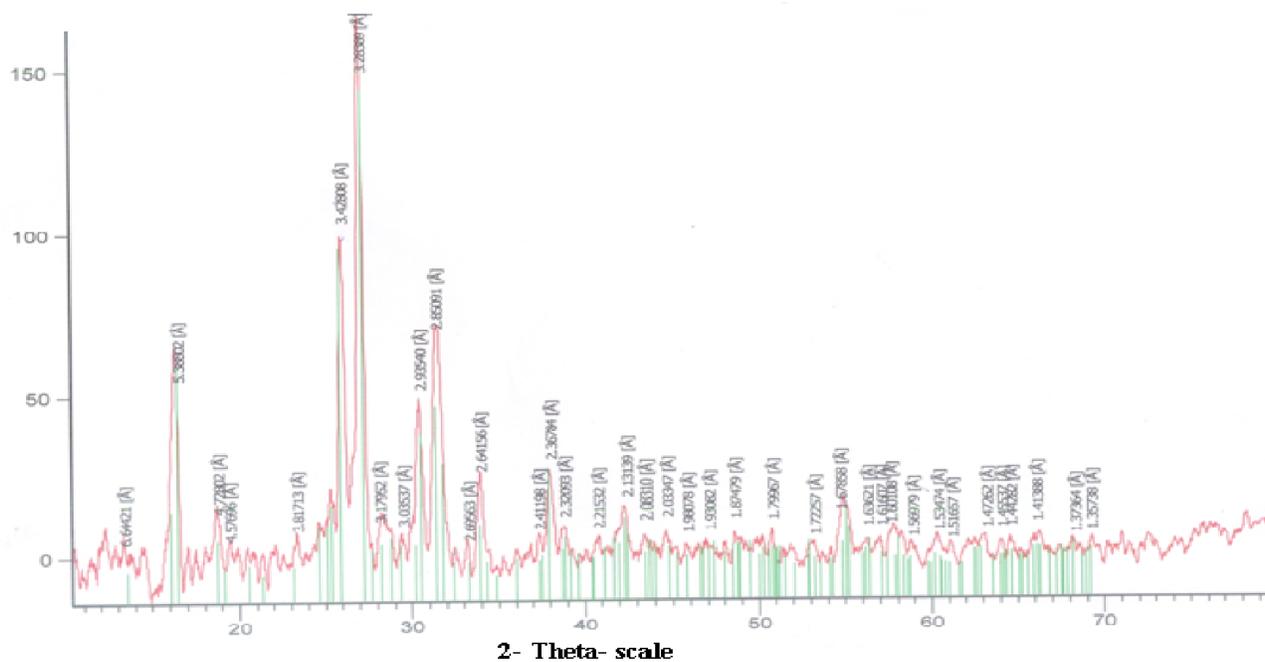


Figure 5b. Representative X-ray spectrum of porcelain specimens subjected to B: 10 watt CO₂ laser glazing

Counts per second

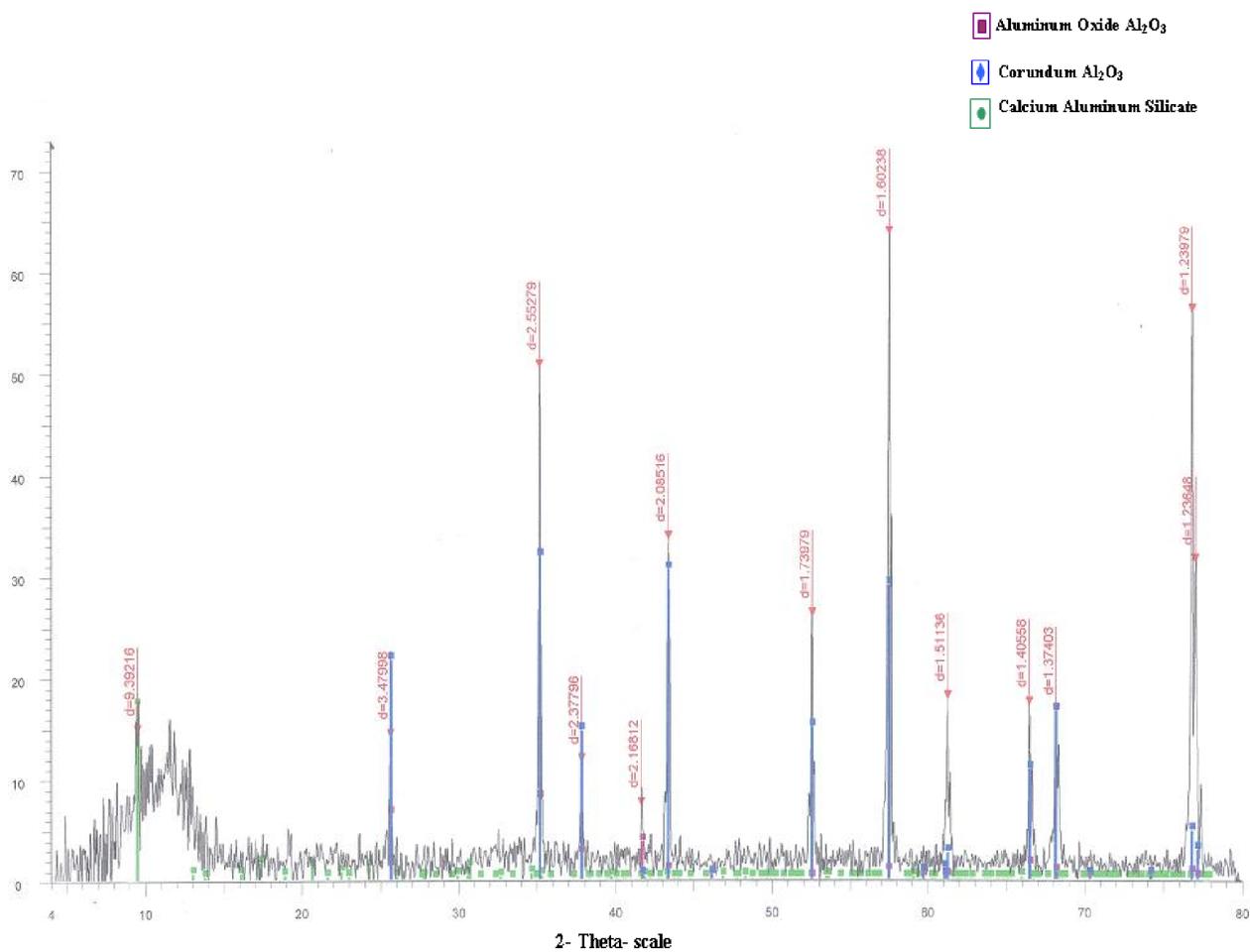


Figure 6a. Representative X-ray spectrum of In-ceram alumina specimens subjected to A: Conventional glazing

Counts per second

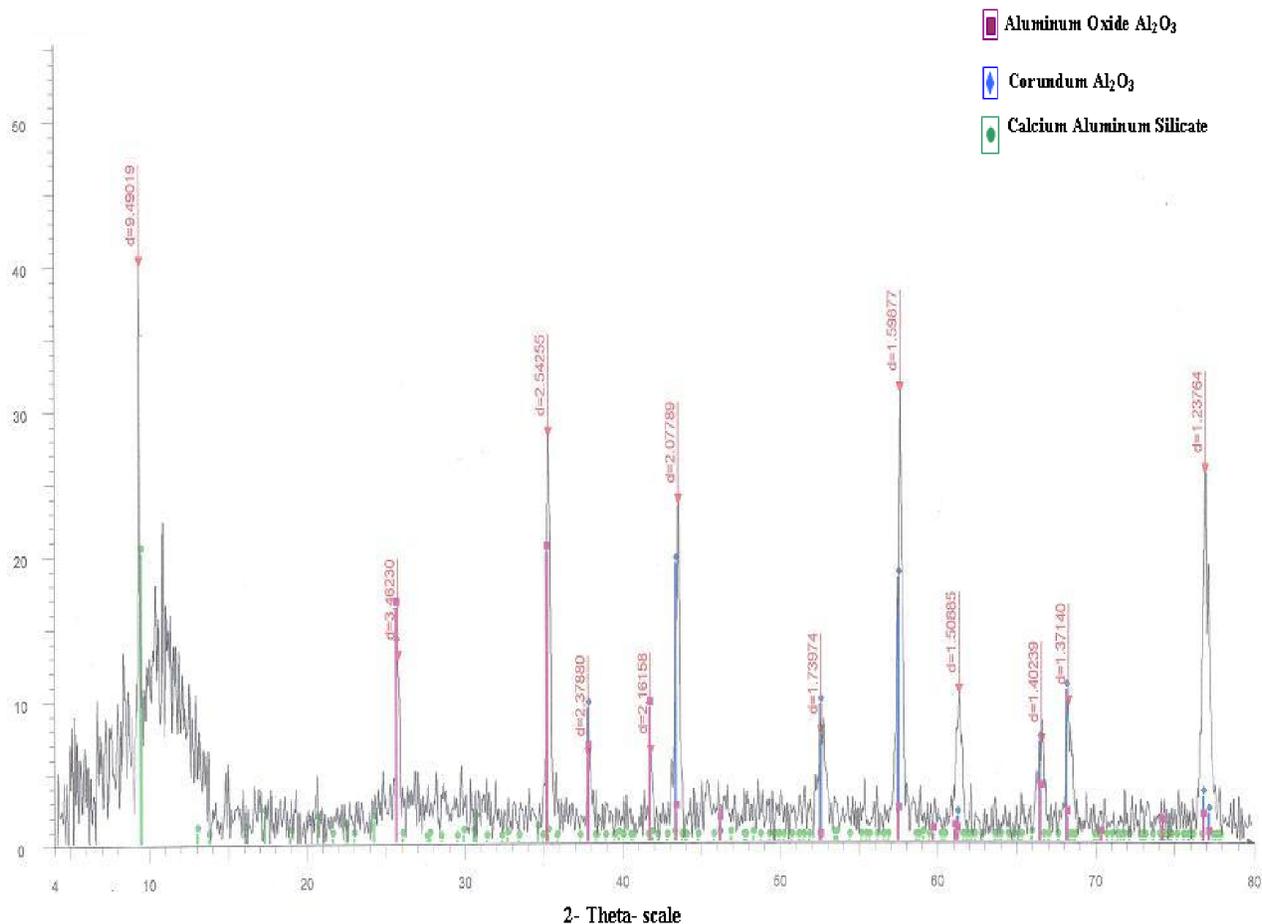


Figure 6b. Representative X-ray spectrum of In-ceram alumina specimens subjected to B: 6.2 Joule /cm² excimer laser glazing

Counts per second