Reinforcing Glaze Layer of Restorative Dental Zirconia by Adding Nano Alumina Ceramics

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Abstract

The objective of this work is to study the effect of adding varying ratios of Nano Alumina to the glazing powder on glazing layer of restorative dental ceramic. The effect of addition is examined by applying Vickers hardness and surface roughness tests on the glazing layer. The specimens have been cut in a cubic form. The specimens were placed in the furnace for sintering up to temperature of 1450 ºC. One specimen is glazed with glazing materials and the remaining three specimens are glazed with glazing materials but supported with varying ratios of Nano Alumina (10, 15 and 25 wt%) and all these specimens sintered at 850 ºC. It was found that Vickers hardness is increased with increasing the ratios of Nano Alumina but the surface roughness decreased with increasing the ratios of Nano Alumina. Weibull modulus increased with Alumina additive increases for glass coating layer.

Key Words: Dental Ceramics, Zirconia, Glazing, Weibull Modulus, Vickers Hardness.

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Introduction

Because of their aesthetic quality and a very well biocompatibility, Ceramics are the most materials used in the field of a dental restorative material (Albakry et al., 2004).

The procedure of glazing for ceramic dental restorations is applied to provide esthetic and smoothen rough surfaces (Aksoy et al., 2006). Fusing temperature of smoothing surface of the ceramic restoration is much lowers than that of the dentin and enamel ceramic (Shillingburg et al., 20012). Thin glaze layer has many significant properties. The micro-cracks and porosities on the ceramic surfaces glaze layer will be filled. The surface of ceramic material could be strengthening by Glazed surfaces (Fairhurst et al., 1992). It also prevents excessive wear of opposing teeth and minimizes bacterial plaque accumulation (Jagger and Harrison 1994). Glazed ceramic can mimic the characterization and gloss of the natural teeth (Al-Wahadni and Muir Martin 1998). The exposure of the ceramic restoration to the oral environment with glazed ceramic materials decreases and makes the restoration smother than unglazed ceramic (Aksoy et al., 2006).

Materials and Methods

Specimen's Preparation

Zirconia blanks (produced by VITA Company) are usually placed inside the CAD-CAM to make crown and bridge. The Zirconia blanks (produced by VITA company) after being milled by CAD-CAM will be used to make zirconia Specimens in another word, the remaining spaces that are not carved on zirconia blanks as shown in the figure (1) below will be utilized to make zirconia Specimens to be used in this work.

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Figure 1. Zirconia Disc that Milled by Copy Milling Machine

The Specimens are cut in a cubic form (one of the dimension is 10 mm and the remaining dimensions are variables). After the Specimens are being cut, they were placed in the ceramic furnace for sintering. The Specimens sintered up to 1450 °C at sintering process of ceramic foam shrinkage by necking and grain growth during densification will accrue with weight loss and the release of gaseous by-products. Therefore, shrinkage is an important parameter that engineers must consider when designing materials in practice. The linear shrinkage rate (LSH) (expressed as a percentage of the plastic length according to ASTM C326) after drying and firing the ceramic Specimen is as follows:

$$\mathrm{LSH}\ \% = \frac{L_o - L}{L_o} \times 100\ \% \qquad (1)$$

Where, L_o: plastic length of test specimen (before firing) and L: fired length of test specimen.

Glazing

After sintering was completed, the four zirconia Specimens are glazed by glazing material (glazing material produced from VITA Company). First Specimen is normally glazed without any additives as dental technician do and the remaining three specimens are glazed with addition varying ratios of Nano Alumina (Corandum-Al_2O_3, >99/99%, particle size (13 nm), sigma Aldrich, USA) to the glaze material as following:

- According to the manufacturers' instructions, weighting 100 wt% of glazing powder and mixing with tow drops of their own glaze liquids. The Specimen is then smoothly coated using a ceramic brush.
- The glazing powder is now mixed with varying ratios of Nano Al_2O_3 that will be used for glazing the remaining three Specimens.
- Weighting 90 wt% of glazing powder and then weighting 10 wt% of Nano Alumina and adding tow drops of glaze liquids and finally mixing them all together to become homogeneous. The second Specimen is then coated.
- Repeat the same procedure of previous step but adding 15 wt% of Nano Alumina to the glazing powder that is specified for the third Specimen. Repeat the same thing for the fourth Specimen but adding 25 wt% of Nano Alumina to the glazing powder.

After all Specimens being coated, the Specimens are placed in the ceramic furnace in order to achieve adhere of the glazing layer on the specimen at temperature of 450 °C for half an hour. The furnace temperature was then increased to 850 °C for two hours according to the manufacturers' instructions.

Vickers Hardness Testing

The Vickers hardness number (HV) of the Specimen surface was examined by using Vickers hardness testing under load of 100 g which is equal to 0.98 N for time period of (5 s) for each zirconia Specimen.
The result of specimen's hardness is quite scattered, because the reason is the scale distribution behavior which different from that of metals causes the failure. This behavior is very. Therefore, when using ceramics, different design methods must be adopted (Carter and Norton 2007).

Weibull survival probabilistic analysis has been developed as an engineering design method for components made of materials such as ceramics. The failure load of a ceramic component depends on the size of the defect and is therefore a characteristic of the specimen, not a characteristic of the material. The defects have a size distribution, so the failure load is variable. Another way to describe this situation is that under any given load, a portion of the test specimen will survive. Weibull describes this score as the probability of survival (Ps) under any tensile stress as (Equation (2)): (Burrow et al., 2004).

$$P_s = \exp\left\{-\left(\frac{\sigma_{max}}{\sigma_o}\right)^m\right\} \qquad (2)$$

Where σ_{max} is the maximum strength ($\sigma_{max}=HV_{max}$ for hardness test), σ_o = the characteristic strength for which the survival probability is 0.37 (1/e) (so, either $\sigma_o = \sigma_{oE}$ for electrical strength or $\sigma_o=\sigma_{oM}$ for mechanical strength). To determine Weibull modulus it must take a natural logarithm of both sides of equation (1)

$$\ln\left(\ln\left|\frac{1}{P_s}\right|\right) = m\ \ln \sigma_{max} - m.\ln\sigma_o \qquad (3)$$

We will get a straight line of slope (m) from ploting $\ln(\ln|1/P_S|)$ versus $\ln\ \sigma$. The higher the Weibull modulus the lower is the variability of strength (Quinn Quinn 2010).

Surface Roughness

The surface roughness of the specimens was examined by using surface roughness instrument (TR220 hand held roughness).

Result and Discussion

Linear Shrinkage

It is noticed from the table (1), the mean shrinkage when sintering at temperature of 1350 °C is 18.69 %.

Table 1. The mean shrinkage when sintering at 1350, 1400 and 1450 °C

Specimen	L_o (mm)	L after sintering at 1350 °C (mm)	L after sintering at 1400 °C (mm)	L after sintering at 1450 °C (mm)	LSH % at 1350 °C	LSH % at 1400 °C	LSH % at 1450 °C
1	10.13	8.15	8.1	8.1	19.54	20.03	20.03
2	10.23	8.35	8.2	8.2	18.37	19.84	19.84
3	10.11	8.3	8.15	8.15	17.90	19.38	19.38
4	10.12	8.2	8.15	8.1	18.97	19.46	19.96
	LSH_{av}				18.69	19.68	19.80

When sintering temperature is raised to 1400 °C, the mean shrinkage is continually increasing up to 19.68 % as indicated in table (2). The difference in shrinkage between (1350 °C and 1400 °C) is 5.2 % only.
The sintering temperature is again raised to 1450 °C, the mean shrinkage becomes 19. 8 % as indicated in table (1). The difference in shrinkage between (1400 °C and 1450 °C) increased to 0.6 %.
When shrinkage by dimensions is increased that is considered as an indication to continue increasing sintering temperature in order to get best mechanical properties. When the shrinkage is stopped this imply that increasing sintering temperature is no longer useful and any increase in sintering temperature will increase the grain size growth as a result decrease the toughness of zirconia which is unacceptable.

Vickers Hardness (HV)

The results of Vickers hardness tests of glazing layer of the specimens are shown in the table (2). The Vickers hardness number (HV) has increased as the quantity of Nano Alumina is increased as clearly indicated in the figure (2). The increase in Nano Alumina content will increase the formation of crystal phases which is responsible for the increase in Vickers hardness number of the glazing layer.
It has also been noticed that the Vickers hardness numbers (HV) at ratios of 15 wt% and 25 % are approximate. That implies any increase above 25 % wills no longer enhancing the Vickers hardness number (HV). Therefore, the addition was stopped at the ratio of 25 %. That means there is a limit of adding Nano Alumina. The mechanical properties of glazing layer are enhanced by adding Nano Alumina and this certainly means increasing the life time of artificial teeth made from zirconia and increasing surface resistance of zirconia.

Table 2. The Vickers hardness number (HV) for different ratios of Al_2O_3

The ratio of Nano Alumina %	Vickers hardness number (HV) MPa
0	569.8
10	673.7
15	726.2
25	730.2

The discrepancy in hardness values was studied according to the statistical analysis model. Weibull modulus was calculated for ten hardness readings on a single specimen surface, after which the relationship between |lnln(1/Ps)| and |lnHV| was plotted. The slope of the straight line represents the Weibull modulus. It was noticed from Figure 3 that the increase in the addition of alumina to the glaze led to an increase in the Weibull modulus and its value ranged from 36.26 to 61.4 (figure 4).
The reason for the increase in the Weibull modulus is the role of alumina grains in hindering the growth of micro-cracks, thus increasing the hardness and reducing the length of cracks arising at the edges of the Vickers indentation. Thus, the hardness of the glaze layer is more accurate by adding alumina,

which means increased durability (Zaidan and Al Ani 2020).

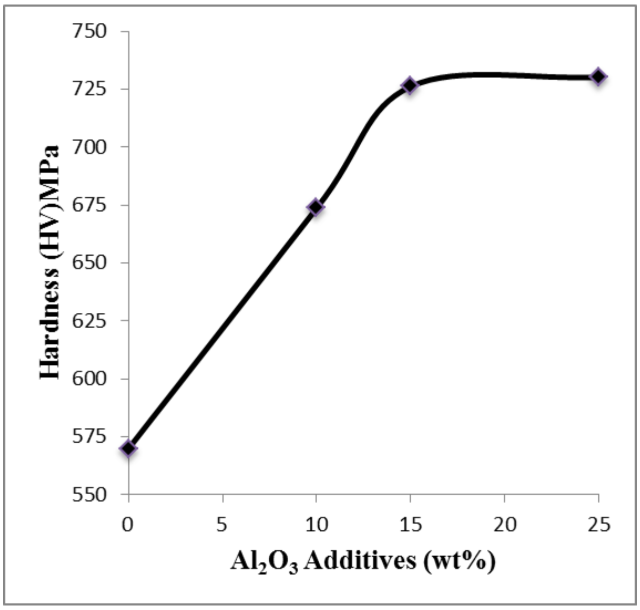

Figure 2. Vickers hardness change with increasing the ratio of Nano Alumina

Surface Roughness

The results of surface roughness test for different glazing layer are shown in the table (3). As clearly indicated in the figure 5 that the surface roughness increases with increasing the ratios of Nano Alumina but the values of surface roughness do not reach inacceptable limits in the prosthodontics field. The increase in the surface roughness is due to crystalline phases that are formed by adding Nano Alumina and hence it is expected that the wear of opposing teeth is increased because increasing the surface friction. But the increasing in hardness will reduce the effect of roughness and it is possible that the glazing layer will maintain its toughness (Salman et al., 2020). The surface roughness increment can be treated either by milling and polishing the surface or by increasing sintering temperature higher than the manufacturers' instructions because the addition of Nano Alumina increases the melting point of the specimen.

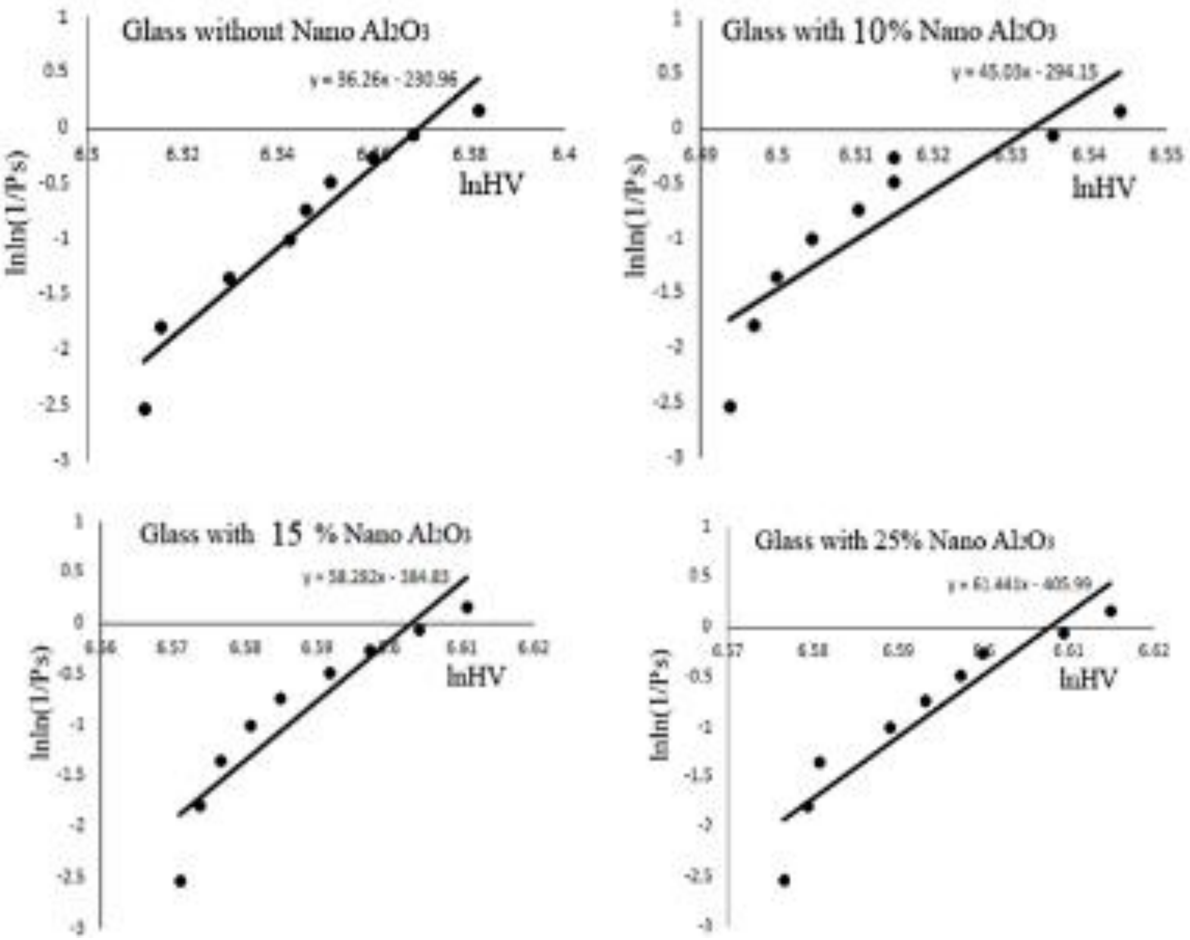

Figure 3. Weibull distribution of the hardness for glass coated layer with difference Nano Al_2O_3 additives

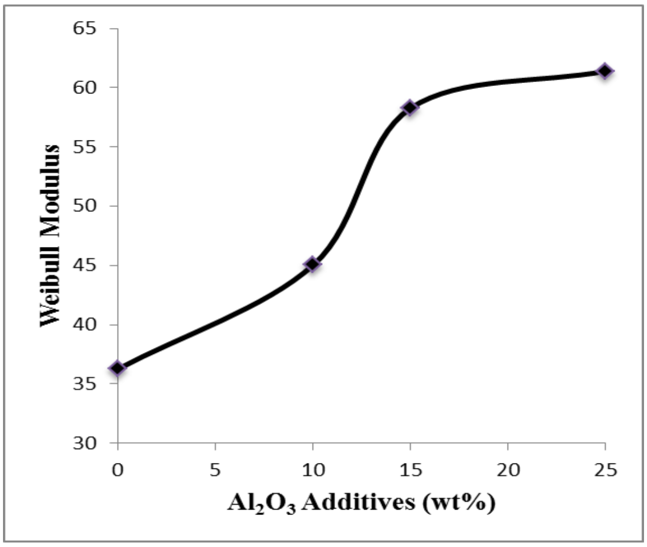

Figure 4. Weibull modulus with increasing the ratio of Nano Al_2O_3

Table 3. The values of surface roughness for different quantity of Nano Al_2O_3

The ratio of Nano wt% Al_2O_3	Surface roughness (μm)
0	0.459
10	0.594
15	0.903
25	0.951

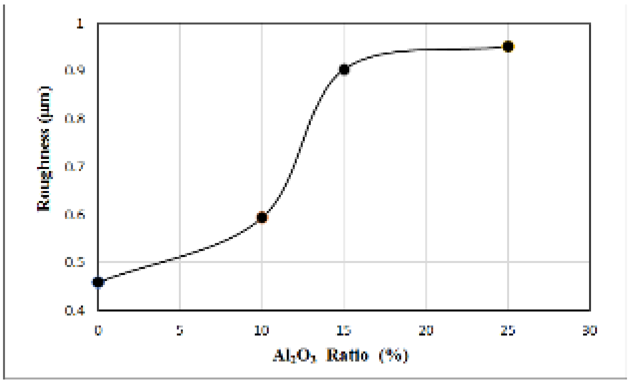

Figure 5. Surface roughness change with increasing the ratio of Nano Alumina

Microscopic Image of the Surfaces

As indicated in the microscopic images as shown in figure 6. It is noticed that forming dark areas on the specimen's surfaces with increasing the ratio of Nano Alumina and these dark areas represent crystals of Nano Alumina that are formed on the surface. The crystalline grains increase the roughness of the surface, but they are almost constant for values of addition greater than 25%.

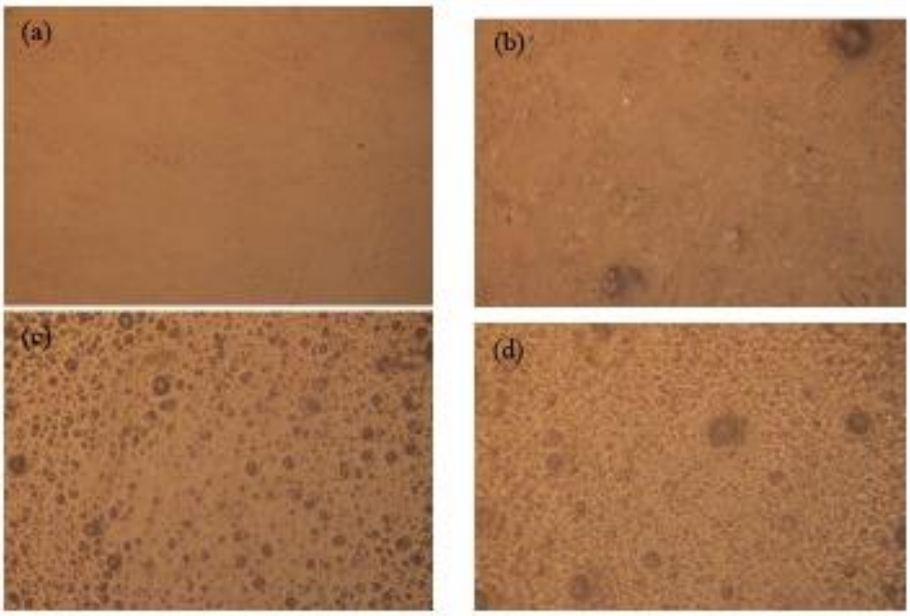

Figure 6. Optical Microscopic image of the glazing layers: (a) without Al_2O_3, (b) with adding (10%) of Al_2O_3, (c) with adding (15%) of Al_2O_3, (d) with adding (25 %) of Al_2O_3

Conclusions

- The addition of Nano Alumina to the glazing material increased the Vickers hardness of specimen surface and the enhancement in hardness is beneficial in the prosthodontics field because increasing the hardness prevents the formation of bores on the teeth surface.

- The surface roughness has increased due to the addition of Nano-Alumina which is undesirable properties and it is because Nano Alumina insoluble in water. T
- he second negative property that is resulted due to addition of Nano Alumina is the opacity increment of the surface, in another word, the translucency of teeth is decreased.
- The reason behind this opacity is the formation of Nano Alumina crystals on the specimen surface as clearly shown on the microscopic images.
- The opacity is clearly increased at higher ratios of Nano Alumina.
- These negative results can be treated either by finishing process or by heat treatment of glazing layer and the heat treatment can be done either by laser or plasma.

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