Original Article

# Effect of Color Shading Procedures and Cyclic Loading on the Biaxial Flexural Strength of Zirconia

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ABSTRACT

**Purpose:** Zirconia is the most preferred ceramic restoration in posterior areas because of its flexural strength. The aim of the study is the evaluation of biaxial flexural strength of different colored zirconium oxide core materials after cyclic loading. **Material and Methods:** Zirconia discs (12 mm diameter and 1.2 mm thickness) were divided into 6 groups of 12 discs each. Groups were colored according to the Vita Classic shade guide: A3 and D4. One group was not colored and left as control. Each group was randomly divided into subgroups and subjected to mechanical cycling prior to biaxial flexural strength test. Cyclic loading was applied as 50 N loads for 20,000 times for the loaded groups. Samples were subjected to biaxial flexural strength test in a universal testing machine with a crosshead speed of 1 mm/min. Two-way analysis of variance (ANOVA) and Tukey's HSD tests were used for comparisons of the groups. **Results:** Biaxial flexural strength values did not vary significantly depending on coloring procedure or loading process tested (p>.05). XRD analysis displayed that the monoclinic volume fraction of zirconia was highest in cyclic loaded D4 and was lowest in non-loaded control group. The SEM image revealed that A3 color solution created metallic coloring pigments at grain boundaries. **Conclusions:** Coloring procedures and cyclic loading did not affect the biaxial flexural strength of zirconia core material; however, microstructural analysis displays changes, which may weaken the zirconia structure on the long term.

**Keywords:** *Biaxial flexural strength, coloring, cyclic loading, zirconia*

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## Introduction

Increasing demand for the esthetic restorations has enhanced research in metal free ceramic frameworks. Due to their high flexural strength, chemical stability, and biocompatibility, zirconia frameworks can also be used in posterior restorations, and have been shown to superior properties to those of other dental ceramics.[1]

But zirconia has opaque white color which limits its esthetic properties. Coloring the zirconia can be used to overcome this disadvantage. Coloring procedure of the zirconia frameworks can provide more natural looking color match. Appropriate dentin color of the structure can be reflected from the inner layers. Direct advantages of colored zirconia frameworks are the reduction in veneer thickness required to mask the white color of the underlying framework and discarding the masking liner material, which is applied before layering the veneer ceramic.[2] Current studies has reported that coloring procedures affect the structure of the zirconia framework.[3-5]

High flexural strength of zirconia is based on the absorption of the mechanical stress due to a process called transformation toughening. In this mechanism, tetragonal phase of zirconia transforms to monoclinic phase of zirconia with a volume increase of % 3-5. Volume increase stops the propagation of microcracks in the zirconia.[6]

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Cyclic loading, such as mastication, is considered an important factor for the durability of all-ceramic restorations.[7] Therefore, it is important to know the amount of stress a material can with stand for material selection. High forces and repetitive stress during the chewing cycle may lead to fatigue of the material, which may eventually result in fracture in the oral environment.[8] Fatigue testing is important for evaluating the mechanical performance of a material.

Cyclic loading can also cause a degradation of the toughening mechanism of zirconia. Current studies reported that aging and cyclic loading cause decrease in strength of zirconia.[9,10]

The aim of the present study was to evaluate whether different coloring liquids or different cyclic loadings affect the strength of zirconia frameworks. The null hypothesis was that the application of different coloring liquids and cyclic loading does not affect the strength of zirconia framework.

## Materials and Methods

### Preparation of samples

Cylindrical shaped metallic mold with 15 mm diameter and 1.2 mm thickness were used for the fabrication of cylindrical shaped zirconia block with manual copy milling unit (Zirkograph 025 Eco, Zirkonzahn, South Tyrol, Italy).

Yttrium partially stabilized zirconium dioxide blocks (ICE Zirkon, Zirkonzahn, South Tyrol, Italy) were cut into discs by means of a low-speed diamond saw (Struers Ltd, Lanarkshire, United Kingdom). The 20% sintering shrinkage was considered as an approximate value. Finally 72 standardized zirconiadiscs (12 mm × 1.2 mm, ICE Zirkon, Zirkonzahn, South Tyrol, Italy) were prepared according to the ISO 6872 standard.[11]

The samples were divided into three groups: Uncolored, Vita A3 and D4 shades. Each group was then divided into two subgroups (n=12); cyclic loading or no cyclic loading.

Test samples (except for the control group) were dipped in the coloring liquid (Zirkonzahn) with plastic holders, held in liquid for 3 s, and dried under a warming lamp (Zirkonlampe 250, Zirkonzahn) for 45 min. After the shading procedure, samples were sintered in a sintering oven (Zirkonzahn) according to the manufacturer's instructions [Table 1].

### Cyclic loading

Twelve specimens for each group were subjected to mechanical cycling prior to biaxial flexural strength test. Mechanical cycling was performed in a mechanical cycling machine (CS-4, SD Mechatronik, Germany) that was simulating mechanical forces during the chewing cycle.

The specimens were fixed under the 3.2 mm diameter ball which was applied 50 N loads for 20,000 times. The test was performed in distilled water at 37°C.

### Biaxial flexural strength testing

Twelve specimens per group were placed in the Universal Testing Machine (TSTM 02500 Elista Ltd., Istanbul, Turkey) and biaxial flexural strength of the specimens wastested at a speed of 1 mm/ min [Figure 1].

Each disc specimen was placed centrally on three hardened steel balls (with the diameter of 3 mm, positioned 120° apart on a support circle with a diameter of 10mm) and upper dowel pin, (1.4 mm diameter) forced the center of the specimens until the fracture occurred.

Data were calculated according to guidelines of ISO 6872 to obtain biaxial flexural strength.

### X-ray diffraction analysis

The crystalline phases of the specimens were determined X ray diffractometry using Cu-kα radiation in the 2θ range of 20 to 40°, with a step width of 0.02° and scan speed of 1 degree per minute were used. The monoclinic phase content of different surfaces was calculated using the Garvie and Nicholson method from the integrated intensities of monoclinic and tetragonal peaks:[12]

$$X_m = [I_m(-111) + I_m(111)] / [I_m(-111) + I_m(111) + I_t(111)]$$

where I is the intensities at angular position 2θ degrees from the diffraction, t and m are the tetragonal and monoclinic peaks. The monoclinic volume fraction was then obtained using equation proposed by Toraya *et al.*[13]

$$Xv = \frac{1{,}311X_m}{1 + 0{,}311X_m}$$

### Scanning Electron Microscope *(SEM) Analysis*

The surface of the specimens were observed using a field emission SEM (JSM 6300F, Joel Ltd., Japan). Surface of the specimens were observed after different applications.

### Statistical analysis

The 'SPSS 15.0 for Windows' package program performed statistical analysis of the data. Statistical analysis was performed with Two-way (coloring procedure and loading process) ANOVA followed by Tukey's HSDs at a significance level set at $p<0.05$.

## Results

### Biaxial flexural strength test

Biaxial flexural strength of the samples according to different color shades and cyclic loading are given in [Table 2]. When the color-shaded groups were considered, statistically significant differences were not observed among the groups (p>0.05). When the loading process was considered, there were no statistically significant differences between the groups (p>0.05).

### Evaluation of XRD

X-ray diffraction analysis revealed changes from tetragonal to monoclinic phase in zirconia specimens after surface treatment. [Table 3] shows X-ray diffraction traces for the evaluated specimens. Monoclinic volume was increased in cyclic loaded groups and colored groups.

The results show that the monoclinic volume fraction of zirconia was highest in cyclic loaded D4 and was lowest in non-loaded control group.

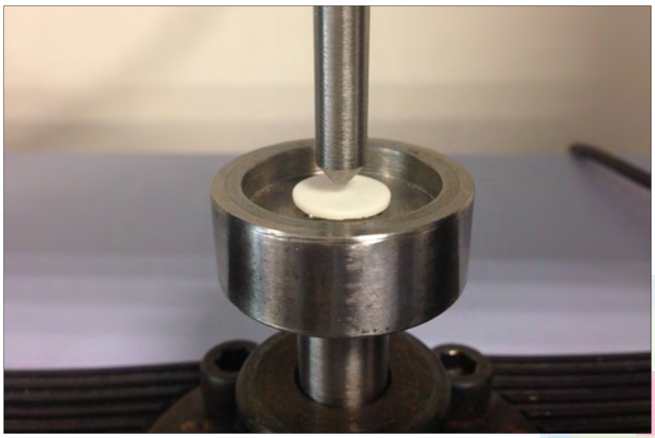

**Figure 1:** İllustration of biaxial flexural strength testing

**Table 1: ICE zirconia sintering program**

| Final temperature | 1500°C |
|---|---|
| Heating time | ~ 3 h |
| Standby time | 2 h |
| Cooling time | ~ 18 min |

**Table 2: Biaxial flexural strength of various groups**

| Groups | Mean (MPa) | SD (±) | Min (MPa) | Max (MPa) |
|---|---|---|---|---|
| Control | 992.07 | 114.15 | 804.62 | 1196.60 |
| A3 | 980.56 | 109.27 | 787.11 | 1161.64 |
| D4 | 954.62 | 91.26 | 850.08 | 1030.13 |
| Control (Cyclic Loaded) | 915.01 | 117.67 | 753.81 | 1032.45 |
| A3(Cyclic Loaded) | 952.26 | 87.47 | 775.12 | 1071.80 |
| D4 (Cyclic Loaded) | 939.17 | 88.04 | 758.12 | 1018.35 |

**Table 3: XRD monoclinic volume fraction of the groups**

| Groups | Non Loaded | Cyclic loaded |
|---|---|---|
| Control | 4.7 | 5 |
| A3 | 5 | 5.1 |
| D4 | 6.2 | 6.6 |

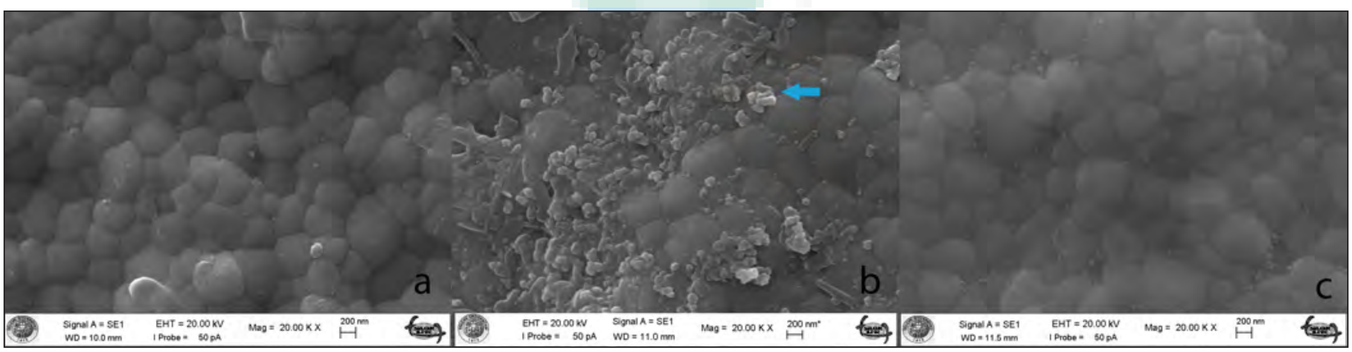


**Figure 2:** SEM images at 20.000 K magnification non-loaded. (a) Control, (b) A3 group, (c) D4 group

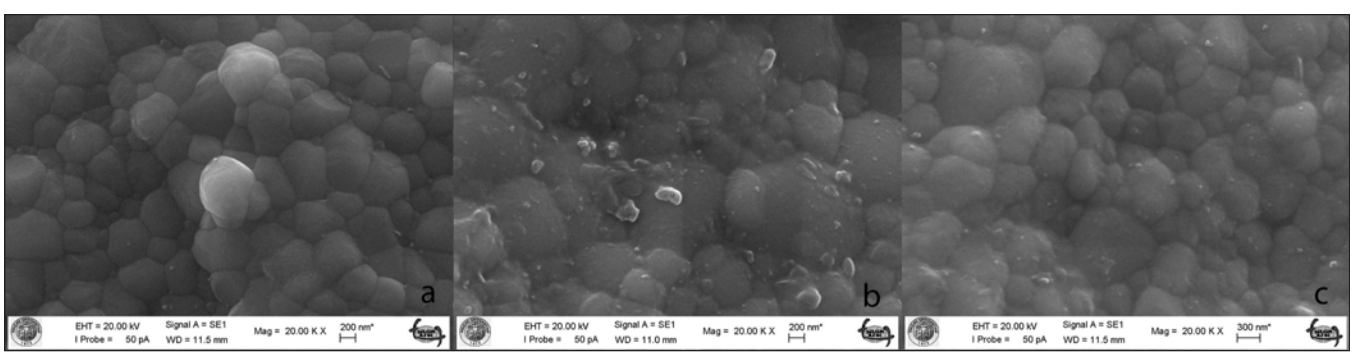


**Figure 3:** SEM images at 20.000 K magnification cyclic loaded. (a) Control, (b) A3 group, (c) D4 group

### SEM analysis

The SEM analyses of the specimen surfaces are shown in [Figures 2 and 3]. The SEM image revealed that A3 color solution created metallic coloring pigments at grain boundaries which can be observed both at non-loaded [Figure 2-b] and loaded [Figure 3-b] specimens where there was not any obvious accumulation in the control group [Figures 2-a, 3-a] and D4 group [Figures 2-c, 3-c].

## Discussion

Mechanical strength of the dental materials is the most important factor for the ceramic restoration. It has a flexural strength of 900-400 MPa, a fracture toughness up to 10 $MN/m^{3/2}$ and a modulus of elasticity value of 210 GPa.[14-16] Flextural strength of the zirconia based on the T→M phase transformation. This transformation is affected by temperature, moisture, grain size, micro and macro cracking of the material, concentration of stabilizing oxides, and fabrication and veneering techniques.[14,17-20] In the current study, the effects of cyclic loading and coloring procedures on the biaxial flexural strength of the zirconia was evaluated.The hypothesis that application of different coloring liquids and cyclic loading to the zirconia framework does not affect the strength of framework was accepted.

High and repetitive stresses during the mastication cause crack growth in the ceramic material. Fracture is the result of these cracks, which cause the degration of the strength of the zirconia.[10,21] Physiological loading conditions can be generated by the chewing cycle simulation.[7,8,22-28] In the present study, the physiological values of the mastication parameters used for the cyclic fatigue loading in the mastication simulator were evaluated according to the current literature.[29,30] Several *in-vitro* studies used a cyclic loading force of 49 N for load-to-failure testing of dental restorations.[31,32] These studies have considered functional forces that arise during mastication or swallowing, which usually range between 2 and 50 N.[29,30] Hence, a cyclic loading force of 50 N was applied in the present study to simulate a clinically relevant situation.

Although zirconia frameworks are more esthetically acceptable compared with metallic frameworks, their opaque and whitish appearance remains a handicap. Thus, colored zirconia frameworks were introduced to obtain a more natural-looking color match. The main advantage of colored zirconia ceramics is that they enable color to be reflected from the inner layer, as in the dentin and enamel structure of natural teeth.[4,19]

According to the results of current study, there was no statistically significant difference among the colored groups and the non-colored control group. Hjerppe *et al.*[4] evaluated the effects of different coloring solutions (A3, B1, C4, D2, D4) and dipping periods on the fracture resistance of zirconia and found that the coloring procedure had a negative effect on the fracture strength of zirconia, which is in contrast to our findings. They demonstrated that shrinkage percentages of non-colored groups are higher than the colored groups resulting in the higher density of the colored groups. It was claimed that the non-colored groups fracture strength is higher than the colored groups due to this increase in density. The contrast between the findings may be due to the variation between the thicknesses of the samples. In the study by Hjerppe *et al.*[4] the thickness of the samples was 1 mm where in the current study it is 1.2 mm as mentioned in the ISO 6872 standard.[11] As the application method of the coloring liquid is immersion, it can be considered that the samples are facially affected by the coloring liquids. Thus there is a thicker unaffected part in the middle of the 1.2 mm thick samples of the current study.

Color solutions, which contain metallic pigments, are applied with the dipping of zirconia framework before sinterization. It was reported that concentration of the coloring pigments at grain boundaries reduce the stabilizing element (yttrium).[33] Chen and Chen[34] explain that melting point of coloring pigment, is much lower than the melting point of yttrium and hafnium oxides (2410 and 2751°C, respectively), so displacement of the stabilizing elements by the metallic pigments can occur during the sintering of zirconia frameworks.[19] Reduction in the percentage of the stabilizing elements would result in higher percentage of tetragonal-monoclinic transformation and that affects the mechanical properties of the zirconia. In the current study, XRD analyze results show that tetragonal-monoclinic phase transformation increased in the colored groups. The percentage of tetragonal-monoclinic transformation in the colored groups cause reduction in strength, toughness and density, which is more than non-colored groups. Phase change causes micro and macro cracking of the material.[35] Phase changing increase roughness, grain pull out and possible premature failure[33] Excessive T→M phase transformation may cause surface elevations and craters can be observed.[19] In this manner, potential risk factors for the non-colored groups were lower than the colored groups. XRD analyze results also show that tetragonal-monoclinic phase transformation increased in cyclic-loaded groups. Dynamic loading can perform T→M phase transformation and micro cracks, which cause the degration of the strength of the zirconia. Although no significant differences were observed between the fracture strength of colored and non-colored specimens, the increase in phase transformation may suggest a decrease in the mechanical properties on the long term.

Evaluating one trademark of zirconia and color solutions can be considered as a limitation of the current study. Another limitation was that the chemical composition of the coloring liquid was not investigated. Future researches can evaluate the effect of different color solutions and zirconia.

## Conclusion

Coloring liquids and loading process did not affect the biaxial flexural strength of the structure of zirconia framework in the early period. However, micro analysis suggests that coloring liquid may affect the phase transformation and mechanical properties negatively on the long term.

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### Conflicts of interest

There are no conflicts of interest.

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