Reliability of dental zirconia: effect of thermal history and grain size

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Statement of Purpose: The number of zirconia ceramics (3Y-TZP) available for dental applications is steadily expanding. These materials have a wide range of sintering temperatures, which dictate the mean grain size. Air abrasion with fine alumina particles is a recommended clinical step prior to cementation of dental restorations. Concurrently, it is well established that 3Y-TZP undergoes a stress-induced transformation from tetragonal to monoclinic (*t-m*) when surface treatments such as grinding or air abrasion are performed. It has also been shown that this transformation is reversible after heat treatment associated with staining and glazing steps for dental restorations. The aim of this study was to assess the effect of air abrasion and heat treatment after air abrasion on the depth of transformation, flexural strength and reliability of 3Y-TZP as a function of grain size. The hypothesis tested was that the depth of transformation and consequently the strength and reliability of dental zirconia are affected by both surface treatment and thermal history.

Methods: Cylindrical zirconia blanks (TZ3YE, Tosoh Corp. Japan) were prepared by cold isostatic pressing. Discs (1.5x18mm) were cut from the blanks, sintered at temperatures ranging between 1350 and 1600ºC for 2h, and randomly divided into 12 groups (n=20). For each sintering temperature, one group was left as sintered as control (AS), one group was air abraded (AA) and one group was air abraded and heat treated at 1250ºC/20 min. (AAHT). Crystalline phases and transformation depth were analyzed by grazing incidence x-ray diffraction (GIXRD). The mean biaxial flexural strength (BFS) was measured according to ISO standard 6872[1]. Strength results were analyzed by Weibull statistics. The microstructure was investigated by SEM. The real grain size was determined according to ISO standard 13356[2].

Results: Mean grain size, biaxial flexural strength, Weibull modulus and transformation depth for each treatment group are listed in Table 1. The mean grain size increased with sintering temperature from 0.35±0.03 μm for AS1350 to 0.96±0.12 μm for AS1600. The mean biaxial flexural strength was comparable amongst all as sintered groups. Air abrasion significantly increased the mean flexural strength for all groups. Heat treatment after air abrasion significantly decreased the flexural strength compared to air abrasion alone. However, the mean flexural strength of groups AAHT 1350 and AAHT1450 was significantly higher than that of matching as-sintered controls. Both air abrasion and heat treatment after air abrasion significantly increased the Weibull modulus for all treatment groups. Reliability was therefore highest for group AAHT1450, followed by AAHT1550, AA1450, AAHT1350 and AA1550. Group AAHT1450 had the best combination of mean flexural strength and reliability. GIXRD studies showed that the depth of the *t-m* transformation after air abrasion increased from 10.5 for AA1350 to 19.2 microns for AA1550. The presence of *m*-phase in group AS1600 led to a transformation depth of 13.5 microns for AAHT1600.

Group	Grain size (μm)	BFS (MPa)	Weibull modulus	Transf. depth (μm)
AS1350	0.35±0.03[a]	1000±146[a]	8.1±0.5	NA
AA1350		1553±201[e]	9.1±0.3	10.5
AAHT1350		1156±87[bc]	15.4±1.7	NA
AS1450	0.52±0.06[b]	1036±114[a]	13.3±0.5	NA
AA1450		1502±102[de]	17.6±0.7	13.2
AAHT1450		1187±97[bc]	20.9±1.3	NA
AS1550	0.76±0.07[c]	1147±89[bc]	15.0±1.4	NA
AA1550		1391±108[d]	15.2±0.6	19.2
AAHT1550		1258±115[ab]	19.8±0.9	NA
AS1600	0.96±0.12[d]	1015±143[a]	7.0±0.8	NA
AA1600		1258±115[c]	12.8±0.9	13.5
AAHT1600		1020±103[a]	11.6±1.0	NA

Identical letters denote no statistically significant difference (p>0.05).
Table 1. Grain size, flexural strength, W_m and transformation depth for the various groups.

Conclusions: Thermal history conditions grain size, phase assemblage and residual stresses in zirconia dental ceramics. Air abrasion promoted the *t-m* transformation while subsequent heat treatment reversed the transformation with an associated decrease in strength due to stress relaxation. Lower strength and reliability of the group sintered at 1600ºC is in line with a shallower transformation depth and the presence of a significant amount of cubic phase with lower toughness. Sintering at 1450ºC led to the highest strength and reliability after AA and AAHT.

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References

1. ISO-6872 (2008). International Standard - Dental Ceramics. *(E), Geneva, Switzerland* 1(6-15).
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