3M℠ Health Care Academy

Lava™ Zirconia Aging Stability

Y-TZP transformation toughening and low-temperature degradation

Zirconium dioxide (ZrO_2) exists in three crystalline states: Monoclinic at room temperature, tetragonal above 1170°C and cubic above 2100°C.[1,2] The “zirconia” used for dental applications is composed of zirconium dioxide crystals stabilized in the tetragonal state by the addition of yttrium oxide.

The tetragonal crystalline state is responsible for the high strength and fracture toughness of Y-TZP (Yttrium oxide stabilized Tetragonal Zirconium dioxide Polycrystals) materials. Once a crack propagates within a Y-TZP material, the energy supplied by the crack can trigger the tetragonal to monoclinic phase transformation in the surrounding grains. This phase transformation leads to a local compressive stress field that hinders further crack propagation.[1,2] This so-called “transformation toughening” is what makes Y-TZP the strongest ceramic material class used in dentistry.

It is well known[3,4] that, on Y-TZP material surfaces, the tetragonal to monoclinic phase transformation can also slowly occur by contact with water. The impact of this “low-temperature degradation” (LTD) on the lifetime of a medical device can be very different depending on the application and on the material composition and processing. Materials that are not well engineered might show the growth of the water-induced transformation zone. This may ultimately lead to bulk strength degradation and surface roughening. An often-cited example is the accumulated failure of zirconia femoral heads observed in 2001.[4] However, the main issue was recognized as processing errors for certain batches were all coming from a single manufacturer.

For dental applications, the key learning is the importance of manufacturing process control. Moreover, each particular Y-TZP material has to be tested for its phase transformation properties to assure long-term stability. The key question to be answered is: **Does water have a clinically significant influence on strength?**

It is important to note that the findings presented here are only valid for Lava™ Zirconia materials. Zirconia materials are not alike. Phase transformation properties strongly depend on the raw material, the grain size, the amount and distribution of dopands, the individual production process (i.e. the pressing and sintering parameters) and the quality of process control.[1,2]

Lava Zirconia material transformation toughening

Lava zirconia is thoroughly tested with respect to phase transformation properties. For Lava zirconia, efficient transformation toughening was demonstrated by space-resolved X-ray diffraction (XRD) analysis of fracture surfaces.[5] These findings are in accordance with the consistently high strength of Lava zirconia (Fig. 1).

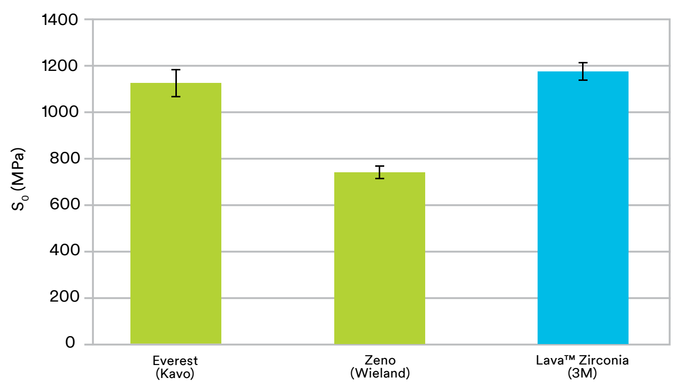

Fig. 1: Weibull’s characteristic strength determined with 3-point bending of polished bars in water.[6]

Influence of water on surface phase composition

Exposure of Lava™ Zirconia material to water leads to a superficial phase transformation detectable by XRD. The changes in crystal phase composition do not lead to a disintegration of the surface. Fig. 2 summarizes the surface phase compositions of as-fired Lava zirconia samples after storage in air, water and autoclaving.[7] The as-fired surface contains no monoclinic phase. The monoclinic phase reaches 21% for the samples aged by autoclaving. Here, besides the monoclinic, a significant amount of cubic II or "distorted cubic" phase is formed.

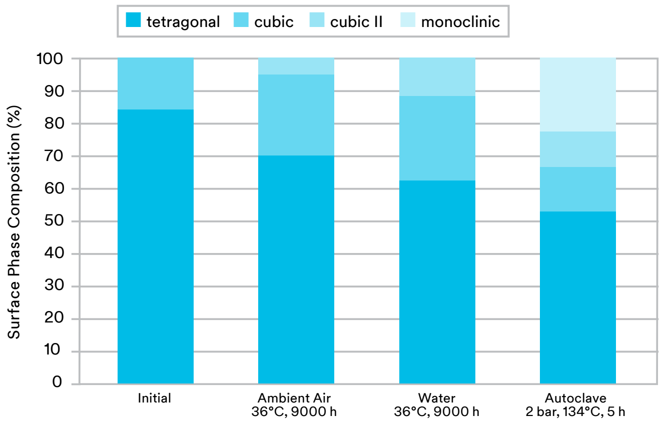

Fig. 2: Surface phase composition of as-fired Lava™ Zirconia material initially and after storage at ambient air, storage in water and autoclaving.

Autoclaving is an established method for the accelerated aging of Y-TZP materials. Autoclaving at 134°C for 5 hours is the standard aging protocol according to ISO 13 356, valid for Y-TZP implants for surgery, e.g. femoral heads. The content of monoclinic phase on Lava zirconia as-fired surfaces is below the ISO 13356 : 2008 limit of 25%.

Fig. 2 shows the aging behavior of as-fired surfaces. Ground, sandblasted or polished surfaces show a different behavior. A small fraction of monoclinic phase is formed by mechanical treatment.[8] The resulting surface subsequently shows a higher stability to water-induced phase transformation.[9]

Another factor greatly influencing the aging behavior of zirconia is the alumina content. Alumina increases the aging stability. However, it also decreases translucency. Some improved translucency zirconias are alumina free. For Lava™ Plus High Translucency Zirconia, 3M chose a different approach. The alumina content of Lava Plus zirconia is reduced to 0.1% to greatly increase the translucency. In addition, the alumina distribution was improved to fully maintain the aging stability (Fig. 3).[9, 10]

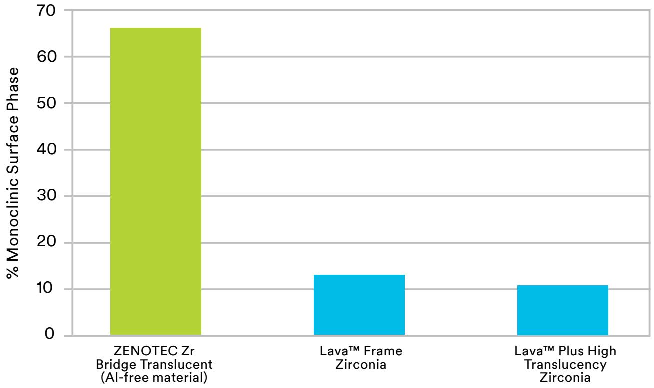

Fig. 3: Monoclinic surface phase content of polished samples after accelerated aging (30 hours autoclaving at 2 bars, 134°C).

Influence of water on strength

It is well documented that the storage of Lava zirconia samples in water does not lead to a significant decrease in strength.[11, 12, 13, 14] The hydrolytic stability proven for Lava zirconia material is very much in contrast to framework ceramics containing a glass phase like e.g. Empress 2, IPS e.max CAD, Empress CAD and Inceram Alumina, where a significant strength decrease was observed.[11, 13]

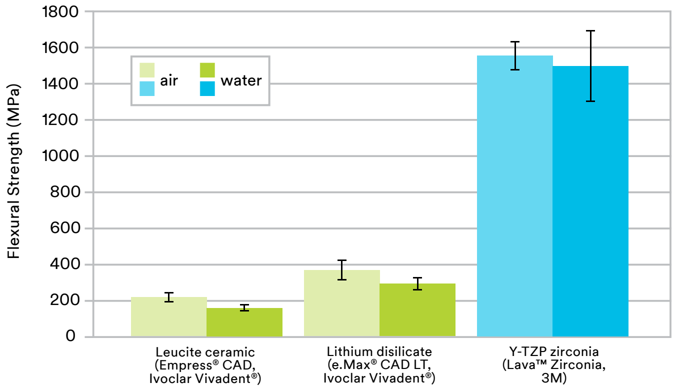

Fig. 4: Flexural Strength of Empress CAD, IPS e.max CAD and Lava™ Zirconia after 30 days of storage and subsequent testing in water. The glass containing systems showed a significant decrease in strength of 28% and 21%, respectively. No significant influence was found for Lava zirconia.[13]

Extensive autoclaving of 168 hours[15] or even 200 hours[16] was found to lead to a small decrease in hardness or strength, respectively. However, correlating these autoclaving times to real-time storage data for Lava zirconia material yields values of up to 80 years of exposure.

The superficial phase transformation induced by autoclaving following the ISO protocol has no significant influence on the bulk strength of Lava zirconia samples (Fig. 5).[7, 14, 17, 18] In contrast, the accelerated aging by autoclaving was found to increase the reliability factor (or Weibull module) m[7] and the fracture toughness,[19] respectively.

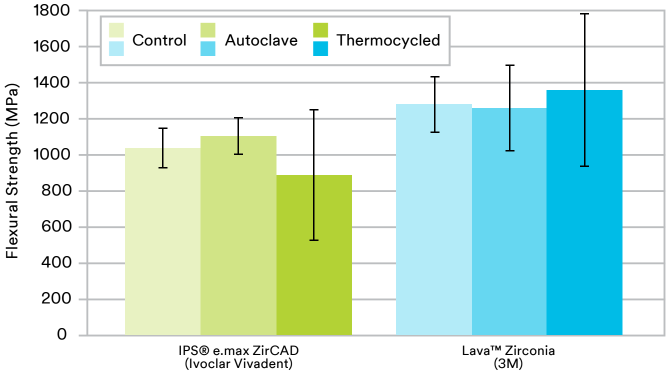

Fig. 5: Biaxial flexural strength of Y-TZP materials (Lava™ Zirconia, IPS e.max® ZirCAD) tested before and after thermocycling and autoclaving, respectively.[18]

Bottom line

Phase transformation is the mechanism responsible for the high strength of Y-TZP zirconia materials. Phase transformation also occurs on the surface after long-term exposure to water.

For Lava™ Zirconia materials from 3M, extensive research revealed that surface phase transformation leads to a new, stable surface with a mix of tetragonal, cubic, cubic II and monoclinic crystal phases. The superficial phase transformation did not decrease strength. Factors like framework design and finishing technique have a much higher influence on the restoration strength as compared to the surface phase transformation.

Looking back on over 15 years of clinical history, Lava zirconia material has proven its clinical performance in more than 10 studies. In sum, more than 1,500 Lava zirconia crown and bridge restorations were placed with follow-up times of up to 7 years. From thinnest coping anterior crowns[20] to posterior bridges,[21, 22] Lava zirconia frameworks showed excellent success rates.

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