



## BIO- AND MEDICAL TECHNOLOGY

# CRYSTALLIZATION BEHAVIOR OF LITHIUM DISILICATE VENEERING CERAMICS

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### Lithium disilicate glass ceramics as veneering ceramics

Lithium disilicate glass ceramics have been known for a long time. The processing is carried out by milling and hot pressing from blanks. For the developed veneering ceramics, powders are used. At a temperature of 900 °C, lithium disilicate (LDS) crystallizes at a rate of approximately 10 μm/sec [1]. If powdered material is used, the corresponding particle is crystallized in a very short time. An increase of the viscosity is connected to the crystallization, which reduces the rate of reaction with both the adjacent grain and the TZ3Y substrate. In order to take advantage of the excellent properties of the lithium silicate glass ceramics as veneering ceramics (VBK), it is necessary to control the proportion of crystal and glass phase.

### Ratio $\text{Li}_2\text{O}:\text{SiO}_2$ on the crystallization behavior

The TEM images [2] (Figure 1a/b) show the change in the morphology of lithium disilicate in dependence of the ratio  $\text{Li}_2\text{O}:\text{SiO}_2$ . Based on the molar ratio of 1:2 from the LDS, an excess of  $\text{SiO}_2$  (1:2.6) can be seen in Figure 1a and a lack of  $\text{SiO}_2$  (1:1.4) in Figure 1b. The  $\text{Li}_2\text{O}$  excess leads to sharper crystal contours, many stacking faults and inclusions in the crystal (1b). For veneering ceramics, the sample with higher  $\text{SiO}_2$  content, less pronounced grain boundaries or the continuous transition between crystal and glass phase is better suited. In the crystals of the sample (1a), TEM micrographs show no inclusions and only a few stacking faults.

### Interface/surface effects and diffusion processes

#### a) Interface between veneering ceramics and zirconium oxide

$\text{ZrO}_2$  diffuses out of the ceramic framework into the veneering ceramics. TOF-SIMS studies [2] show that the ionic components of  $\text{Li}_2\text{O}$ ,  $\text{Na}_2\text{O}$  and  $\text{Nb}_2\text{O}_5$  diffuse into the zirconium oxide ceramics. This leads to a depletion of  $\text{Li}^+$  ions in the boundary

region of the veneering ceramics and, thus, to the emergence of a glass phase. The diffusion behavior enables a very intense adhesive bond between the framework of yttrium-stabilized zirconium oxide and the veneering ceramics.

#### b) Interface between LDS crystal and the adjacent glass or crystal phase

For the developed veneering ceramics,  $\text{Nb}_2\text{O}_5$  is an essential part. With the crystallization of LDS and the associated concentration shifts, sodium niobate crystals are formed directly at the LDS crystals (Figure 2). Raman spectroscopy shows an intense peak at the wave number of 860  $\text{cm}^{-1}$  for both the niobate crystal and in the amorphous region. The niobate-analog structural units favor the melting of the powdered LDS glass ceramics and the formation of a smooth surface.

#### c) Surface of the veneering ceramics and application

After annealing process, the surface of the veneering ceramic is smooth, glassy and does not require glaze firing. The developed veneering ceramics type was handed to the project partners for the market launch phase. Figure 3 shows a dental restoration with sprayed veneering ceramics.

### Sources

- [1] T. Honma and T. Komatsu, Journal of the Ceramic Society of Japan 116(12) 1314-1318 2.
- [2] TEM/TOF-SIMS in Kooperation mit Fraunhofer IWM / Prof. Höche.



1 Influence of  $\text{Li}_2\text{O}:\text{SiO}_2$  on the crystal structure of LDS (TEM).

2 Interface between veneering ceramics and  $\text{ZrO}_2$  framework (REM).

3 Zirconium oxide bridge with sprayed veneering ceramics.