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Chemical analysis of the crystal structure of the In-Ceram compound

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Introduction

Chemical analysis of the quality of the In-Ceram-system presumes a complete understanding and knowledge about its microstructure and its chemical and macroscopic composition. As far as science is concerned, the knowledge about ionic exchange in the borderline of compound materials (such as glass - infiltered aluminium oxide and ceramics) is mandatory. This knowledge could be the base for further results in the research about the persistency, the process of erosion and the wearing-out, as well as the mechanisms of fracture of the In-Ceram in the oral cavity.

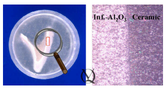

Fig.1: Ground section showing a crown manufactured with the In-Ceram system

Material and Methods

For the chemical analysis, testing instruments were manufactured according to the instructions of the Vita Company in Bad Saeckingen, Germany. Squares made of Al2O3-suspension with the size of 5 x 10 x 1 mm were used as a framework These were reprocessed in the following steps:

- sintering
- glassinfiltration
- removing of the surplus glass
- veneering with Vita(r) Alpha ceramic (thickness 1 mm)

Finally, with the help of a diamond-coated saw, the testing instruments were cut in two. On the surfaces of the ground sections, corresponding measuring spots were chosen in the centre of the ceramic layer and the infiltered Al2O3 (Fig.2).

Quantitative analysis The quantitative analysis of characteristic elements was realized by X-Ray induced Photoelectron Spectrometry (XPS) (Fig.3).

XPS line scan The interface between ceramic and infiltered Al2O3 was scanned with a line scan (lateral extension: 20 µm) to determine the concentration of the elements O, Al, Si, C, La and Na. The tested area had the size of 1500 µm x 60 µm with an information depth of 20 µm (Fig.4).

Extensive Electron Micro-scanning Analysis With the help of the Electron micro-scanning probe (Comebax SX 50) and the Visilog image processing system, the elements Si, Al, Ca, La, Zr and K were determined (Fig.5-10). The scanned area with a size of 1 mm x 1 mm was evaluated in the stage modus. The step size used was 2 µm in the x-y-direction of the diagram, so that all together 262144 single analyses have been made. As to the colour-codes, red stands for the highest, black for the lowest x-ray intensity.

Results

The results of the XPS linescans show a comparatively flat increase resp. decrease of the concentration curves at the borderline between ceramic and Al2O3. This might indicate the existence of an interface area, where ionic exchange processes take place.

This hypothesis was not confirmed by the results of the x-ray micro-scanning analysis. The distribution of the elements Si, Al, Ca, La, Zr and K in the interface area shows a very distinct borderline between the infiltered Al2O3 and the ceramic. The entire area of infiltered Al2O3 shows an insular, though homogenous distribution of Al and La (Fig.5 and 6). Ionic exchange or diffusion processes could not be verified.

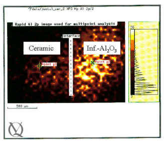

Fig.2: Localization of the measuring spots for the qualitative and quantitative analysis

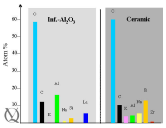

Fig.3: Quantitative analysis of the elements in the Inf.-Al2O3 and the ceramic

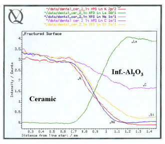

Fig.4: XPS linescans of the surface of the adjoining area Inf.-Al2O3 / Ceramic

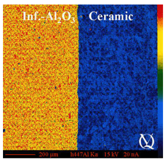

Fig.5: Extensive distribution of Al in the interface area between ceramic and infiltered Al2O3

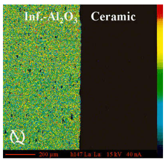

Fig.6: Extensive distribution of La in the interface area between ceramic and infiltered Al2O3

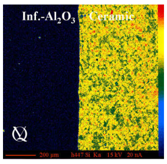

Fig.7: Extensive distribution of Si in the interface area between ceramic and infiltered Al2O3

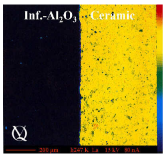

Fig.8: Extensive distribution of K in the interface area between ceramic and infiltered Al2O3

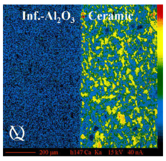

Fig.9: Extensive distribution of Ca in the interface area between ceramic and infiltered Al2O3

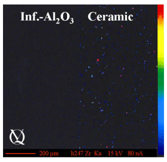

Fig.10: Extensive distribution of Zr in the interface area between ceramic and infiltered Al2O3

This Poster was submitted on 03.02.00 by PD Dr. A.J. Patyk.

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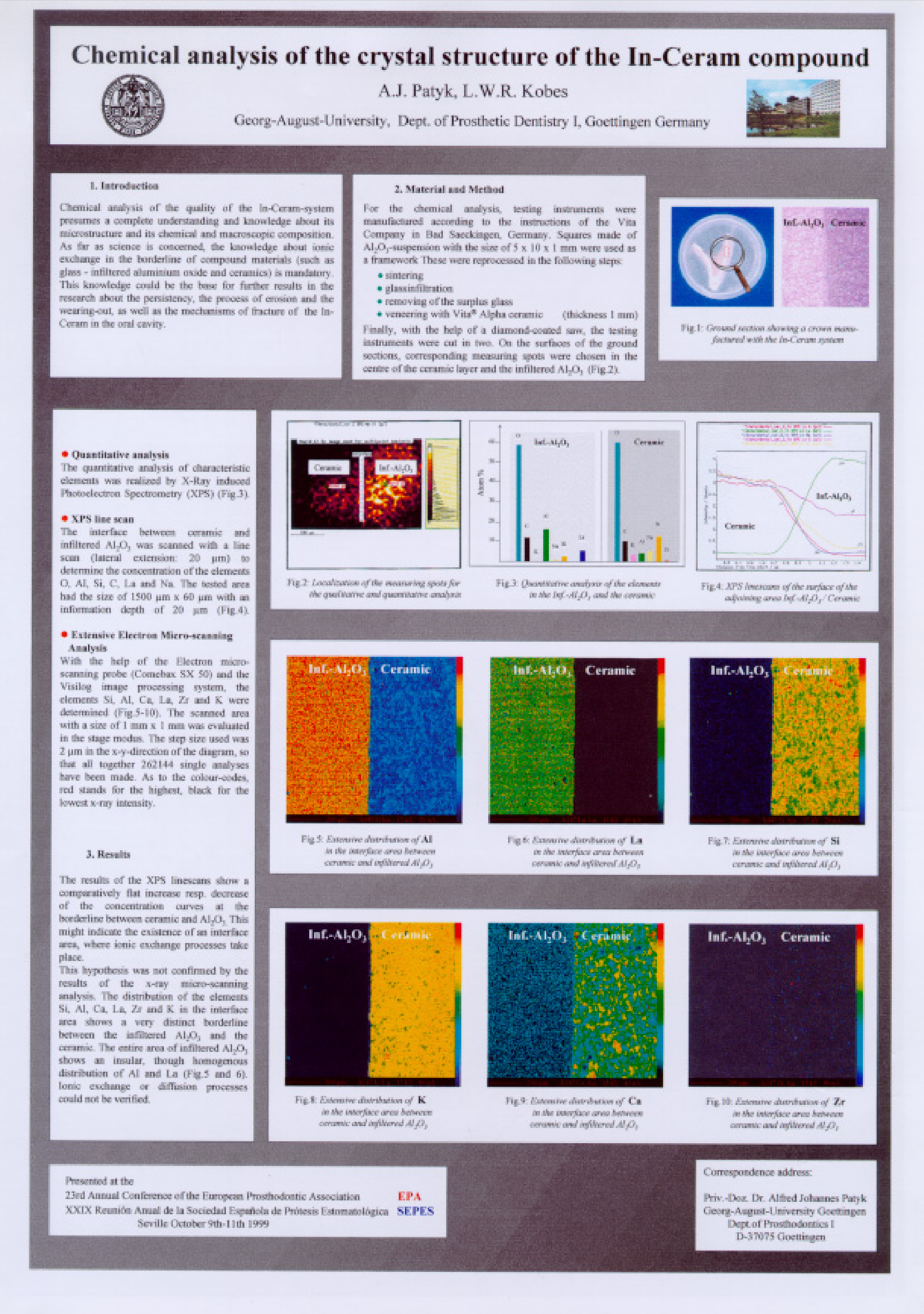

Chemical analysis of the crystal structure of the In-Ceram compound

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1. Introduction

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2. Material and Method

For the chemical analysis, testing instruments were manufactured according to the instructions of the Vita Company in Bad Saeckingen, Germany. Squares made of Al_2O_3-suspension with the size of 5 x 10 x 1 mm were used as a framework These were reprocessed in the following steps:

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Finally, with the help of a diamond-coated saw, the testing instruments were cut in two. On the surfaces of the ground sections, corresponding measuring spots were chosen in the centre of the ceramic layer and the infiltered Al_2O_3 (Fig.2).

Inf.-Al_2O_3 Ceramic

Fig.1: Ground section showing a crown manufactured with the In-Ceram system

- **Quantitative analysis**
  The quantitative analysis of characteristic elements was realized by X-Ray induced Photoelectron Spectrometry (XPS) (Fig.3).

- **XPS line scan**
  The interface between ceramic and infiltered Al_2O_3 was scanned with a line scan (lateral extension: 20 µm) to determine the concentration of the elements O, Al, Si, C, La and Na. The tested area had the size of 1500 µm x 60 µm with an information depth of 20 µm (Fig.4).

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  With the help of the Electron micro-scanning probe (Comebax SX 50) and the Visilog image processing system, the elements Si, Al, Ca, La, Zr and K were determined (Fig.5-10). The scanned area with a size of 1 mm x 1 mm was evaluated in the stage modus. The step size used was 2 µm in the x-y-direction of the diagram, so that all together 262144 single analyses have been made. As to the colour-codes, red stands for the highest, black for the lowest x-ray intensity.

Ceramic Inf.-Al_2O_3

Fig.2: Localization of the measuring spots for the qualitative and quantitative analysis

Atom % Inf.-Al_2O_3 Ceramic

Fig.3: Quantitative analysis of the elements in the Inf.-Al_2O_3 and the ceramic

Ceramic Inf.-Al_2O_3

Fig.4: XPS linescans of the surface of the adjoining area Inf.-Al_2O_3 / Ceramic

Inf.-Al_2O_3 Ceramic

Fig.5: Extensive distribution of **Al** *in the interface area between ceramic and infiltered Al_2O_3*

Inf.-Al_2O_3 Ceramic

Fig.6: Extensive distribution of **La** *in the interface area between ceramic and infiltered Al_2O_3*

Inf.-Al_2O_3 Ceramic

Fig.7: Extensive distribution of **Si** *in the interface area between ceramic and infiltered Al_2O_3*

3. Results

The results of the XPS linescans show a comparatively flat increase resp. decrease of the concentration curves at the borderline between ceramic and Al_2O_3. This might indicate the existence of an interface area, where ionic exchange processes take place.

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Inf.-Al_2O_3 Ceramic

Fig.8: Extensive distribution of **K** *in the interface area between ceramic and infiltered Al_2O_3*

Inf.-Al_2O_3 Ceramic

Fig.9: Extensive distribution of **Ca** *in the interface area between ceramic and infiltered Al_2O_3*

Inf.-Al_2O_3 Ceramic

Fig.10: Extensive distribution of **Zr** *in the interface area between ceramic and infiltered Al_2O_3*

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