the better way to

ceramill zi

Technical Documentation

AMANNGIRRBACH

1 Product description

Ceramill ZI Blanks

Ceramill ZI Blanks are presintered Zirconia-Blanks of Y-TZP ZrO_2 (with yttria partially stabilized Zirconia polycrystals).

Ceramill ZI Blanks are used for the production of frameworks for fixed or removable prosthetics (e.g. crowns and bridges, conus-/telescopic crowns, suprastructures etc.) with CAD/CAM or copy milling (e.g. Ceramill multi-x) devices.

The material is milled in a pre-sintered state and after that it gets densely sintered in a high-temperature furnace (e.g. Ceramill Therm) with a material optimized pre-programmed sintering process.

After this sintering procedure Ceramill ZI, surpasses the requirements of standard ISO 6872 Dental Ceramic, type II, class 1 [1].

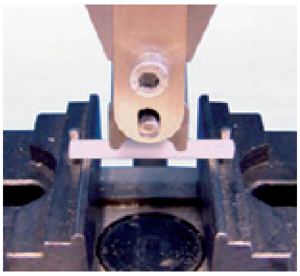
Material properties of the blanks are proofed according to ISO 13356 "Surgical Implants – Ceramic materials of yttria-stabilized tetragonal zirconia (Y-TZP)" [2] and recorded in a test certificate.

Table of contents

2 Properties

2.1 Physical properties

2.1.1 Bending strength



The bending strength of a ceramic material describes the tension at the materials failure.
Bending bars were prepared according to testing standard for technical ceramics EN 843-1 [3]. After sintering the bending bars were proved in a 4-point-bending-test (Fig. 1).

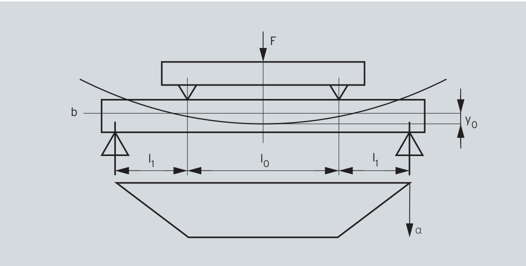


Fig. 1: Bending bar in testing machine (right); schematic representation (top)

The bending strength of Ceramill ZI is compared to the strengths of 6 competitor materials in Fig. 2.

AmannGirrbach, internal manufacturing guidelines for 4-point-bending-strengths (Weibull strength 63.21%) are 1200 ± 200 MPa. In the scope of this investigation Ceramill ZI samples presented a mean initial bending strength of 1358 MPa.

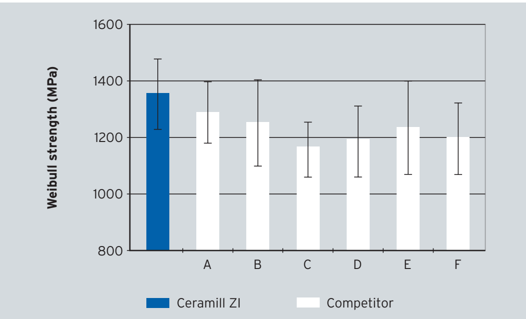


Fig. 2: Comparison of bending strengths of several dental Y-TZP zirconia materials; AmannGirrbach 2008

4-point-bending-strength in comparison to a competitor material (translucent)

The strength of Ceramill ZI was proved in comparison of a competitor material (translucent) at University Clinics of Regensburg, Germany [4]. Results are shown in Fig. 3.

The bending-strength of Ceramill ZI amounted 1566 MPa and was significantly higher than the competitor material (translucent) showing 934 MPa. The comparison of theirs contrast ratios is charted in 2.1.6.

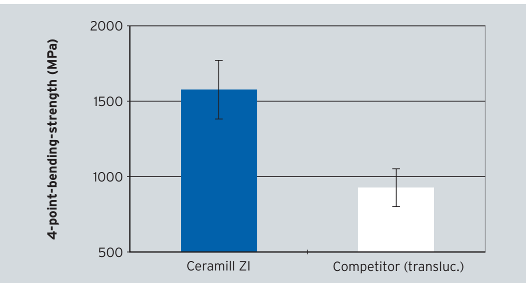


Fig. 3: Bending-strength of Ceramill ZI in comparison to a competitor material (translucent); University Clinics of Regensburg, Germany, 2009 [4]

Influence of coloring

After infiltrating the Ceramill ZI bending bar with Ceramill Liquid (Fig. 4) according to manufacturers instructions for use, the influence of coloring on bending strength was tested.

The results in Fig. 5 verify that the coloring with Ceramill Liquid does not influence the bending-strength of Ceramill ZI negatively.

Fig. 4: Non-colored and colored bending bar

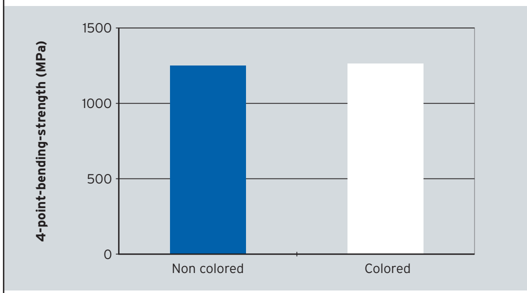


Fig. 5: Bending-strength of non-colored and with Ceramill Liquid colored Ceramill ZI; AmannGirrbach, 2009

2.1.2 Weibull Modulus

The Weibull Modulus represents the scattering of the strength values of a material. Ceramic materials should demonstrate a Weibull-Modulus m ≥ 10. Higher values are representing a higher reliability of the material in its clinical use. Fig. 6 shows Weibull Moduli of several dental Y-TZP zirconia materials according to the bending-strength test measurements of Fig. 2.

Analysis of Ceramill ZI bending-strength results of Fig. 2 resulted in a Weibull Modulus of 16.5.

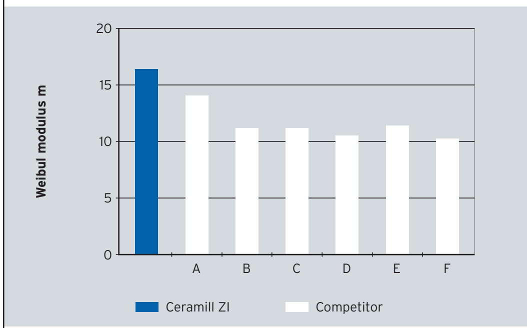


Fig. 6: Comparison of Weibull Moduli of several dental Y-TZP zirconia materials; AmannGirrbach, 2008

2.1.3 Fracture Toughness

The fracture toughness of a material represents the resistance against crack propagation and is a very important ceramics property. At the University Clinics of Regensburg the fracture toughness values of Ceramill ZI and a competitor material (translucent) was determined [4]. The results are pictured in Fig. 7.

Fracture toughness of Ceramill ZI (13.7 MPa*m^{0,5}) is slightly higher than the competitor materials (13.1 MPa*m^{0,5}). Both oxide-ceramics fracture toughness values were on a high level. The comparison of contrast ratio of both materials is shown in Fig. 10.

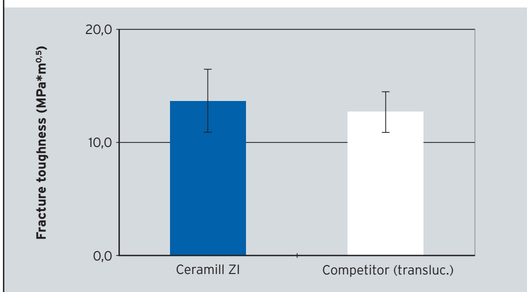


Fig. 7: Fracture toughness of Ceramill ZI in comparison to a competitor material (translucent); University Clinics of Regensburg, Germany, 2009 [4]

2.1.4 Density

The density of several sintered zirconia samples after final sintering was evaluated according to DIN EN 993 part 1. For maximum mechanical properties of the zirconia materials, a void and pore free microstructure and a final density of > 6.07 g/cm³ is necessary.

A final density value of 6.73 g/cm³ was evaluated for Ceramill ZI.

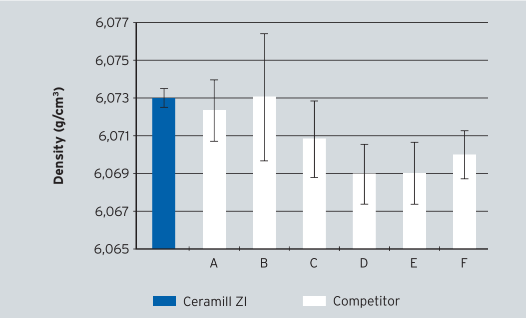


Fig. 8: Comparison of densities of several dental Y-TZP zirconia materials;
AmannGirrbach, 2008

2.1.5 Vickers Hardness

The hardness of a material presents the resistance against the penetration of another material, e.g. a diamond pyramid. In dental application the hardness is relevant for milling and polishing. The harder a material the more difficult it is to grind and mill but the better it is to polish. The Vickers hardness of densely sintered zirconia materials is significantly higher than that of alloys. The Vickers hardness of several dental zirconia materials was determined according to EN 843-4 (Fig. 9).

The differences of Vickers hardness values of investigated zirconia materials were just marginal.

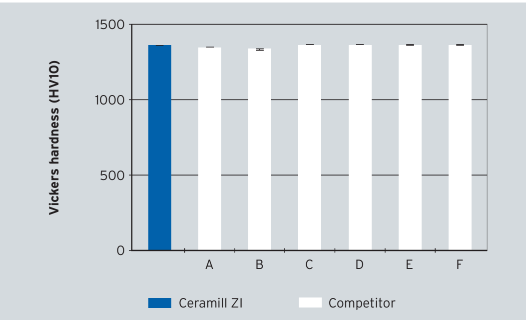


Fig. 9: Comparison of Vickers hardness of several dental Y-TZP zirconia materials;
AmannGirrbach, 2008

2.1.6 Translucency / Contrast ratio

For a translucency resp. opacity comparison of Ceramill ZI and a competitor material (translucent), the contrast ratios according to British Standard BS 5612:1978 [6] were determined at the University Clinics of Regensburg [4]. Contrast ratio (opacity) comparison values were 6.4 % for glass and 100% for metal.

Fig. 10 makes clear that there is just a marginal difference of contrast ratios of both materials and that the differences in light transmittance are visually not differentiable. Bending-strength-comparison of both materials is illustrated in 2.1.1.

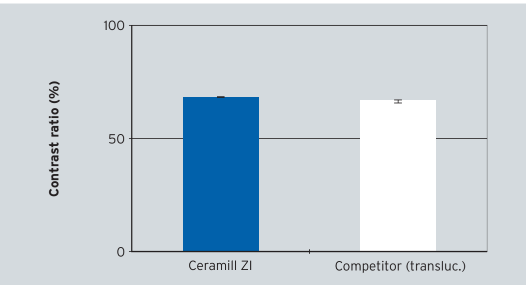


Fig. 10: Contrast ratio (opacity) of Ceramill ZI in comparison to a competitor material (translucent); University Clinics of Regensburg, Germany, 2009 [4]

2.1.7 Microstructure

To represent the Ceramill ZI microstructure after sintering procedure at 1450 °C (2 hrs.), SEM-investigations were carried out. Samples were grinded, polished, and after that thermally etched. Fig. 11 and Fig. 12 represent the homogeneous, fine-grained and pore-free microstructure of Ceramill ZI after sintering. SEM-pictures show neither macro- nor micro-pores which stands for a void-free microstructure.

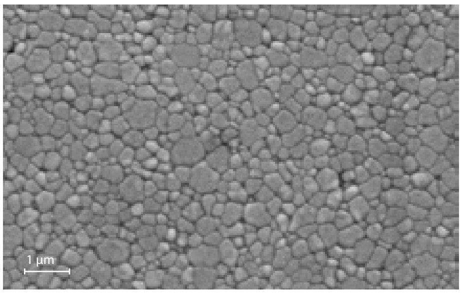


Fig. 11: Ceramill ZI microstructure; sintering temperature 1450 °C; magn. 10.000x; AmannGirrbach, 2008

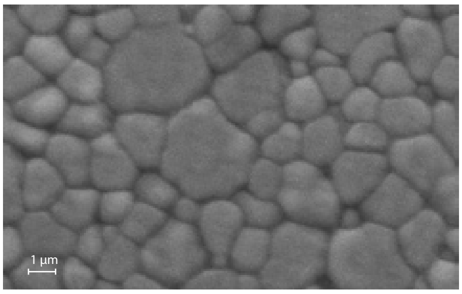


Fig. 12: Ceramill ZI microstructure; sintering temperature 1450 °C; magn. 30.000x; AmannGirrbach, 2008

Mean grain-size

To evaluate the mean grain-size of Ceramill ZI crystals in comparison to a competitor material (translucent) after final sintering, SEM-pictures (Fig. 13 and 14) were taken and a grain-size analysis (Fig. 15) was carried out at the University Clinics of Regensburg [4].

For Ceramill ZI a mean grain-size of 577 +/- 90 nm was determined and requirement of ISO 13356 regarding maximum mean grain-size of 600 nm was fulfilled while 803 +/- 145 nm were measured for the competitor material. A mean grain-size of 600 nm represents the critical grain-size, i.e. for larger grains the tetragonal-monocline phase transformation is not reversible and the mechanism of transformation toughening is not given.

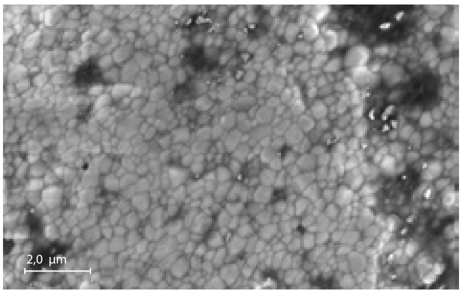


Fig. 13: SEM-picture Ceramill ZI; magn. 20.000x; University Clinics of Regensburg, 2009 [4]

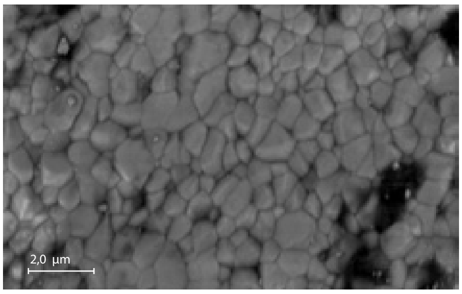


Fig. 14: SEM-picture competitor material (translucent); magnification. 20.000x; University Clinics of Regensburg, 2009 [4]

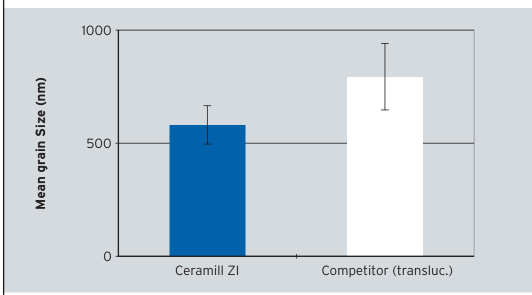


Fig. 15: Mean grain size of Ceramill ZI in comparison to a competitor material (translucent); University Clinics of Regensburg, 2009 [4]

2.2 Biological Properties

2.2.1 Zytotoxicity

For the analysis of biocompatibility Ceramill ZI material was tested at the accredited testing laboratory Bioserv Analytik und Medizinprodukte GmbH (Rostock, Germany). Under the test conditions of DIN EN ISO 10993-5 [7] the extract of the Ceramill ZI material caused no toxicological or biological critical damages to subconfluent monolayer of L929 cells at any of the concentrations tested. Results of negative and positive controls confirm the sensitivity and accuracy of the test system. There was a good correlation between the colorimetric results and microscopically detectable cell appearance. The test can be considered homogeneous and valid.

2.2.2 Chemical Solubility

For the determination of the chemical solubility Ceramill ZI material was tested at the accredited testing laboratory Bioserv Analytik und Medizinprodukte GmbH (Rostock, Germany) according to DIN EN ISO 9693:2000 [8]. After cleaning and drying three samples were weighed. Subsequently the samples were exposed to 4% acetic acid at 80 °C for 16 hours. Afterwards they were cleaned with water of quality 3 according ISO 9693 and dried at 150 °C. After cooling down the samples were weighed again. The loss mass was adducted as measurement of chemical solubility. The chemical solubility of the Ceramill ZI material was examined according DIN EN ISO 9693:2000 [11]. No mass loss was determined. The material fulfils the requirements of DIN EN ISO 9693:2000.

2.2.3 Biocompatibility

The assessment of biocompatibility, was carried out by the accredited testing laboratory Bioserv Analytik und Medizinprodukte GmbH (Rostock, Germany) on the basis of results mentioned in 2.2.1 and 2.2.2 [12]. Based on the above mentioned, test results and according to DIN EN ISO 10993-1 „Biological evaluation of medical devices – Part 1: Evaluation and testing“ the Ceramill ZI material can be rated as biologically compatible in scope of its intended use.

2.2.4 Radioactivity

While the Dental Ceramic ISO 6872 standard requires a maximum U-238 activity concentration of 1 Bq/g, the limiting value is 0,2 Bq/g in the ISO 13356, the standard for Y-TZP implant materials. At the Research Center Jülich (Germany) a gamma-spectroscopical investigation with Ceramill ZI was carried out to evaluate the gamma-activity concentrations of the isotopes U-238, Th-232, Ra-226 and Pb-214. The single activities of the isotopes U-238, Th-232 and Ra-226 were lower than the detection limit of the testing method and the activity of PB-214 (< 1 Bq/kg) meets the quantity of natural radiation [13]. The Ceramill ZI zirconia powder contains impurities of UO_2 und ThO_2 only in ppm-quantities (parts per million = 0.0001%).

3 In vitro investigations

3.1 Chewing simulation, bridge strengths and marginal gap

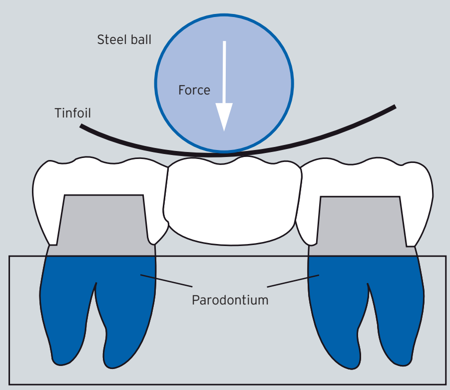


Fig. 17: Schematical representation of bridge-strength-test [5]

Within this investigation 8 adhesive (group 1) and 8 conventional (group 2) - on natural teeth - fixed 3-unit-bridges with ceramic covering were assessed for their bridge strength and margin quality. [5]. The bridge frameworks were prepared by copy-milling process by AmannGirrbach. The ceramic veneering of the frameworks was carried out by the University Clinics of Regensburg, with the veneering ceramic Zi-F Creation (Willi Geller, Baar, Switzerland). The restorations of group 1 were fixed adhesively with the dual-cure system Syntac classic/Variolink II (Ivoclar-Vivadent, Schaan, Liechtenstein) on prepared natural tooth dies. The inside surfaces of the crowns were treated as recommended by the manufacturer with Al_2O_3 110µm (<2 bar). Group 2 will be conventionally fixed (study results to be published). A five year chewing test was simulated in the "Regensburger Chewing Simulator".

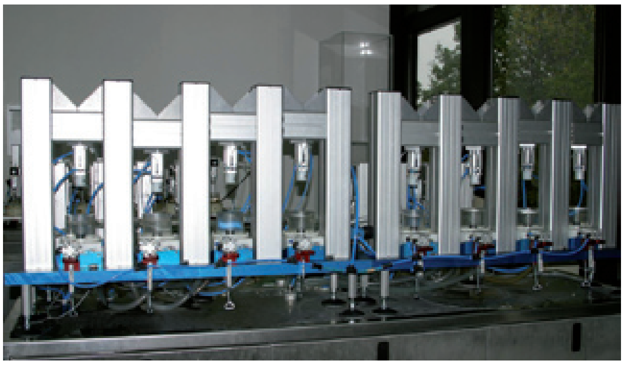


Fig. 16: Regensburger chewing simulator

The Bridges were exposed a thermo-mechanical load of 6000 thermal cycles of 5°C/55°C (2 min per cycle) and 1.2 million chewing-force cycles of 50N according to a 5-year intraoral application. Afterwards a bridge strength test was performed.

Results

In the scope of the chewing device investigation with the Ceramill ZI bridges, no distinctive features were observed. Neither relative failures (cracks or chippings of the veneering ceramics) nor absolute failures (framework fractions) were documented.
The results of the bridges strength tests are shown in Fig. 18.

For group 1 (adhesive cementation) a mean bridge-strength of 1293 N was reported, while the lowest strength was 1204 N. Compared to maximum intraoral chewing forces of 500 N, Ceramill ZI bridges provide a sufficient safety for clinical application.

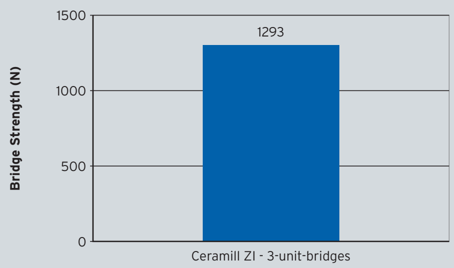


Fig. 18: Results of bridge-strength-test after 5-year chewing simulation; University Clinics of Regensburg, 2008 [5]

Margin quality analysis

For a semiquantitative assessment of margin adaptation in secondary electron microscope (SEM), impressions were taken from each eight restorations before and after chewing simulation (Permadyne double mixing technique, 3M Espe, Seefeld, Germany) as well as Repoxyreplicas (Rencast CW2215, Renshape Solutions). Criteria of SEM assessment were „perfect margin" and „margin gap" at the tooth/cement respectively cement/restoration interface. As „perfect margin" areas were rated without visible interruptions at the interface between tooth and cement resp. cement and restoration. Areas were rated as „margin gap" with recognizable adhesive and cohesive failure of interfaces in combination with detached connections. In the result part only data for the criteria „perfect margin" will be listed.

The results of the margin quality analysis are not recorded yet.

3.2 Abutment study

At the University Clinics of Tübingen (Germany) in-vitro investigations for the assessment of the clinical probation of copy-milled ceramic abutments on Titanium bases and copy-milled all-ceramic abutments are running (Prof. Geis-Gerstorfer, 2008/2009). Titanium abutments are being used as a control group.

For each group 8 abutments have to undergo a chewing-machine-load of (1.2 mill. cycles of 50 N) in an angle of 30° in aqueous environment. After the chewing simulation the abutments will be loaded in a universal testing machine up to their failure.

The overall results are not present yet.

Customized Ceramill ZI abumtments

3.3 Bridge-strength study

At the University clinics of Hannover a chewing machine test with 4-unit-bridges of Ceramill ZI and competitor materials is running (Dr. L. Borchers, 2008/2009).

The results have not been presented yet.

4 unit Ceramill ZI brigde

4 Technical Data Sheet

Manufacturer/ Supplier	Amann Girrbach AG Herrschaftswiesen 1 6842 Koblach/Austria	
Product	Ceramill ZI	
Product type	ZrO_2 Y-TZP blank for manufacturing of fixed prosthetics. The material gets milled in a pre-sintered state and afterwards sintered in a high-temperature furnace with a material-specific sintering program (1450 °C/2 hrs; heating rate 5-10 K/min).	
Product shape	Blocks, discs: e.g. 40x20x16 mm; 65x30x20 mm; 98x X (X= 14,18,20,25)	
Material type	ZrO_2 Y-TZP (with yttria stabilized tetragonal zirconia polycrystals)	
CE-labeling	CE 1275	
Veneering ceramics	Veneerable with conventional zirconia ceramics (e.g. GC Initial Zr, Creation Zi, VITA VM9)	
Composition (Wt-%)	$ZrO_2 + HfO_2 + Y_2O_3$	≥ 99,0
	Y_2O_3	4,5 - 5,4
	HfO_2	< 5
	Al_2O_3	< 0,5
	other oxides	< 0,5
Mechanical, physical and chemical properties after sintering (2 hrs. at 1450 °C; heating rate: 5-10 K/min)	4-point-bending-strength	1200 ± 200 MPa
	Mean grain-size	≤ 0,6 µm
	Modulus of elasticity	> 200 GPa
	Density	≥ 6,05 g/cm^3
	Vickers hardness	1300 ± 200 HV10
	Open porosity	0 %
	Lin. therm. expansion (25-500 °C)	10,0 ± 0,5 $*10^{-6}/K$
	Chem. Solubility	< 5µg/cm^2
	Radioactivity	< 0,2 Bq/g

Ceramill ZI fulfills the requirements of DIN EN ISO 6872[1)] and after appropriate processing (Sintering; 2 hrs. at 1450 °C; heating rate: 5-10 K/min) the requirements of ISO 13356[2)].

1) DIN EN ISO 6872:1999 Dental Ceramic (ISO 6872:1995 including amendment 1:1997); English version of DIN EN ISO 6872.
2) ISO 13356:1997 "Implants for surgery - Ceramic materials based on yttria-stabilized tetragonal zirconia (Y-TZP)".

Koblach, 09/02/19

Research & Development

AMANN GIRRBACH AG

Dr. Sascha Cramer von Clausbruch

5 Declaration of conformity

Konformitätserklärung / Declaration of Conformity / Declaration de Conformite
Dichiarazione di Conformita / Declaratión de Conformidad CE

Amann Girrbach AG
Herrschaftswiesen 1
A-6842 Koblach

erklärt in alleiniger Verantwortung, dass
declares under sole responsibility that
déclare sous propre responsabilité que
dichiar sotto propria responsabilità che
declama bajo propria responsabilidad que

das Medizinprodukt	Zirkonoxidrohling	**Ceramill ZI**
the medical device	Zirconium oxide blank	Artikel Nr:
le dispositif médical	Pièce brute d'oxyde de zircone	LOT: ....
il dispositivo medico	Crezzo in ossido di zirconio	Made in Austria
el producto medicinal	Pieza btuta de oxido de zirkonio	

der Klasse	**IIa**	Nach Anhang IX der Richtlinie 93/42/EWG
of class		according to annex IX of direct. 93/42/EEC
de la classe		selon l'annexe IX de la directive 93/42/CEE
della classe		secondo l'allegato IX della direttiva 93/42/CEE
de la clase		según apéndice IX del 93/42/CEE

allen Anforderungen der Medizinprodukte-Richtlinie 93/42/EWG entspricht, die anwendbar sind.
meets all the provisions of the directive 93/42/EEC which apply to it.
remplit toutes les exigences de la directive sur les dispositifs médicaux 93/42/CEE qui le concernent.
soddisfa tutte le disposizioni della direttiva 93/42/CEE che lo riguardano.
Corrisponde a todas las normas del 93/42/CEE, que son aplicables.

Bei einer nicht mit uns abgestimmten Änderung des Produkts verliert diese Erklärung ihre Gültigkeit.
This declaration shall cease to be valid if modifications are made to the product without our approval.
En cas de modification effectuée sur le produit sans notre accord, cette déclaration sera caduque.
Le misure interne adottate garantiscono che le apparecchiature di serie sono sempre conformi ai requisiti delle attuali direttive CE e alle normative applicate.
A través medidas internas se asegura que los productos siempre satisfacen las exigencias de las normas actuales de la CE así como las normativas aplicables.

Konformitätsbewertungsverfahren gemäß Anhang II
Conformity assessment procedure according to annex II
Procédure d'évaluation de la conformité selon l'annexe II
Procedimento di valutazione della conformità secondo l'allegato II
Procedimento de valoración de la conformidadsegún apéndice

Konformitätsbewertungsstelle	CE 1275
Notified Body	LGA InterCert GmbH,
Organe resp. de l'évaluat. de la conformité	Tillystraße 2,
Organo incaric. della valutaz. della conform	D-90431 Nürnberg
Organo resp. De la evaluación de la conformidad	

Ort, Datum	
Place, date	
Lieu, date	
Luogo, data	
Lugar, fecha	Koblach, 12.03.2008

Name und Funktion	
Name and function	
Nom et fonction	
Nome e funzione	
Nombre y función	Dr. Sascha Cramer von Clausbruch - Sicherheitsbeauftragter für Medizinprodukte

6 References

[1] DIN EN ISO 6872:1999 Dental Ceramic (ISO 6872:1995 including amendment 1:1997); English version of DIN EN ISO 6872.

[2] ISO 13356:1997 "Implants for surgery – Ceramic materials based on yttria-stabilized tetragonal zirconia (Y-TZP)".

[3] DIN EN 843-1:2006; Advanced technical ceramics – Mechanical properties of monolithic ceramics at room temperature – Part 1: Determination of flexurals strength; German version EN 843-1:2006.

[4] Test report: „Comparative in vitro investigation of two zirconia ceramics", Prof. Dr. G. Handel, Dr. M. Rosentritt, Dr. C. Kolbeck , Regensburg, Januar 2009.

[5] Advance test report: „In vitro investigation of margin quality and coolor penetration of adhesive inserted Ceramill ZI 3-unit bridges",
Prof. Dr. G Handel, Dr. M. Rosentritt, Prof. Dr. M. Behr, Regensburg, 2008.

[6] British Standard BS 5612:1978 „Specification for dental porcelains for jacketcrowns".

[7] DIN EN ISO 10993-5 „Biological evaluation of medical devices – Part 5 Tests for in vitro cytotoxicity (ISO/DIS 10993-5:2007);
German version prEN ISO 10993-5:2007.

[8] DIN EN ISO 9693:2000 „Metal-ceramic dental restorative systems (ISO 9693:1999); German version EN ISO 96993:2000.

[10] Test report of zytotoxicity according to ISO 10993-5, Bioserv Analytik und Medizinprodukte GmbH, Dr. U. Meyer, 2007.

[11] Test report of chemical solubility according to EN ISO 9693,
Bioserv Analytik und Medizinprodukte GmbH, Dr. U. Meyer, 2007.

[12] Report: Assessment of biocompatibility; Bioserv Analytik und Medizinprodukte GmbH, Dr. U. Meyer, 2008.

[13] Test report of gamma-spectroscopical determination in ZrO_2 powder Y-TZP, Forschungszentrum Jülich, Sicherheit und Strahlenschutz,
Dr. P. Ostapczuk, 19.12.2007.

Amann Girrbach AG
Herrschaftswiesen 1
6842 Koblach | Austria
Fon +43 5523 62333-399
Fax +43 5523 55990
austria@amanngirrbach.com
www.amanngirrbach.com

AMANNGIRRBACH

www.hellblau.com 31365-FB 2009-03-13