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Material properties and life time of a high performance alumina matrix composite for use in total joint replacement

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Abstract: **Objective** Describe reinforcing mechanisms, material properties and life - time relevant material performance of a new ceramic composite material which is intended for surgery applications. **Method** Analysis of microstructure, model of reinforcing mechanism. Measurement and discussion of mechanical tests with standardized specimens and artificial joint components. Analysis of a combined life - time test with simulated hydrothermal ageing and cyclic fatigue. **Result** The material provides significant higher strength and toughness than standard alumina due to phase transformation toughening. The high strength of the material provides a higher safety margin for artificial joint components. It is also shown that-in contrast to pure zirconia materials-the material is insensitive against hydrothermal ageing. Even under combined extreme conditions of very long exposure time in water vapour at 134 °C and cyclic loading there is no material degradation.

Key words: Zirconia toughened alumina; Material properties; Life-time; Hydrothermal ageing; Cyclic fatigue

应用于关节置换术的高性能氧化铝基复合陶瓷的材料特性和使用寿命

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摘要: **目的** 研究用于外科手术的一种新型陶瓷复合材料的增加机理,材料特性和使用寿命。**方法** 分析材料的微显微结构和增加机理的模型。测量和讨论有关标准试样和人工关节组件的力学试验。分析在模拟热环境老化和循环疲劳试验条件下的使用寿命试验。**结果** 由于对这种新型陶瓷复合材料相变增韧作用,所以它的强度和韧性都高于标准的氧化铝材料。这种新型材料的高强度特性提高了人工关节组件的可靠性。**结论** 与单纯的氧化锆材料相比,这种陶瓷复合材料进一步解决在热环境下老化的问题。即使在温度高达 134 °C的水蒸气和循环加载的极端复合条件下,它也没有降低材料的性能。

关键词: 氧化锆增韧氧化铝; 材料特性; 寿命; 热老化; 循环疲劳载荷
中图分类号: R318.01 **文献标志码**: A

1 Introduction

Since the year 2000 more than 500.000 artificial hip joints with components of the new high performance ceramic composite BIOLOX® *delta* have been successfully implanted. Due to the unique strength and toughness of this material the risk of fracture has been substantially reduced when compared to conventional ceramic materials.

The outstanding properties of BIOLOX® *delta* rely on complex reinforcing mechanisms. Therefore, it is necessary to assess if reinforcement is maintained throughout the live-time of the artificial joint which is anticipated to exceed more than 20 years.

Like any other material which is intended for surgical applications, the suitability must be evaluated based on multiple approaches, like Intrinsic mechanical material properties, biocompatibility, system compatibility and fi-

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nally in-vivo scoring of the surgical outcome

The basis of all progress in material development for surgical applications are the intrinsic material properties. When the surgeon decides to replace a known material by a new one, there must be sufficient indication for a substantial benefit. The most challenging question is to predict the reliability of the material after many years of service life.

Within the scope of this paper, the intrinsic material properties of the composite ceramic BIOLOX® *delta* are analysed. Live time can be traced back to basic principles, i.e. how can a material be damaged after many years of service. Every material degrades after many years loading in an aggressive environment. It is the challenge to create a material which preserves sufficient residual reliability even under worst case conditions for many years.

Due to the chemical stability ceramics obviously provide an intrinsic advantage in comparison to other materials like metals and polymers. Ceramics are produced in the state of a fully saturated chemical bonding. There is no driving power left for further chemical interaction with the environment. Thus, typical live time limiting problems like corrosion or water adsorption are not relevant for high performance and high purity ceramics.

It must be considered if there are other mechanisms which may limit the live time of ceramics. It is well known that like all other materials also ceramics may suffer degradation from following distinguished events:

- Fatigue　resistance against long time static and alternating load
- Ageing　resistance against hydrothermal or other chemical attack
- Wear　durability under abrasive conditions

In this paper, the live time limiting mechanisms and the relevance for the application as a surgical implant are discussed. It is shown how live time of the ceramic material BIOLOX® *delta* can be described and evaluated. The unique microstructure and reinforcing mechanisms of the material not only support the short term performance like fracture toughness and strength but also improve substantially the long term reliability.

2　Description of BIOLOX® *delta*

BIOLOX® *delta* is an alumina based composite ceramic. 80 vol % of the matrix consist of fine grained high purity alumina which is very similar to the well known material BIOLOX® forte. As it is the case in any other composite material, the basic physical properties like stiffness, hardness, thermal conductivity etc. are mainly predetermined from the dominating phase. It was the basic idea for the development of the new material to preserve all the desirable properties of BIOLOX® forte which has millions of components in service but to increase its strength and toughness.

These properties are rigorously improved by implementation of reinforcing elements. Figure 1 shows the microstructure of BIOLOX® *delta*.

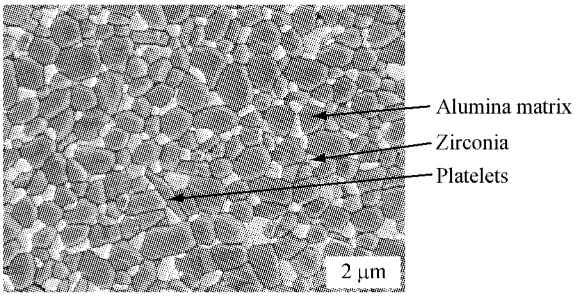

Fig.1　Microstructure of BIOLOX® delta

Two reinforcing components are integrated in BIOLOX® *delta*. 17 vol % of the matrix consist of tetragonal zirconia particles. The average grain size of the zirconia is around 0.2μm. As a further reinforcing element, apprx. 3 vol % of the matrix are built by platelet shaped crystals of the ceramic composition strontiumaluminate. The platelets stretch to a maximum length of apprx. 3 μm with an aspect ratio of 5 - 10. The reinforcing ability of these ingredients is explained below.

Additionally to the reinforcing components, there are also stabilizing elements doped to the material. Chromium is added which is soluble in the alumina matrix and which increases the hardness of the composite. The minor amount of chromium is the reason for the mauve colour of the material. Furthermore, some yttrium is added to the composite which is solved in the zirconia and which supports the stabilization of the tetragonal phase.

The reinforcing elements, in particular the zirconia, substantially increase fracture toughness and strength of the material[1-2]. Fracture toughness (K_{IC}) is a measure for the ability of the material to withstand crack extension. Strength (°C) is defined as the maximum stress within a structure that causes failure of the component. Consequently, when the fracture toughness of the alumina is increased also the strength is directly improved. This basic principle is the concept of the development of BIOLOX® *delta*. The microstructure is designed in order to provide a maximum of resistance against crack extension.

The benefit in crack resistance which is obtained from incorporating zirconia into an alumina matrix are well known in the science of high performance ceramics, as it is shown in figure 2.

The figure represents a realistic part of the microstructure. In the case of severe overloading crack initiation and crack extension will occur. High tensile stresses in the vicinity of the crack tip trigger the tetragonal - monoclinic phase transformation of the zirconia particles. The

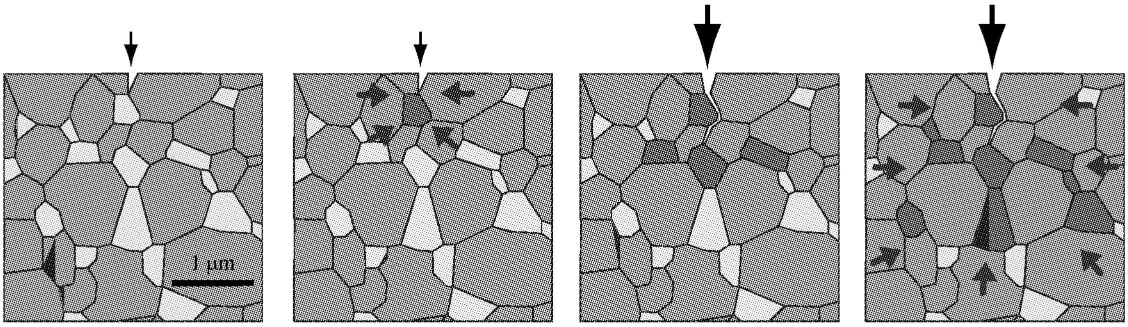

Fig.2 Reinforcing mechanism in BIOLOX® *delta* at crack initiation and propagation

accompanied volume expansion leads to the formation of compressive stresses which are very efficient for blocking the crack extension.

As it is shown this reinforcing mechanism is fully activated within a region of a few micrometers. For the macroscopic performance of the material it is extremely important that immediately at the beginning of crack initiation also the reinforcing mechanisms are activated. Regarding figure 2 one should keep in mind that the average distance between the reinforcing zirconia particles is apprx. 0,2μm, i.e. similar to the grain size. Thus, the reinforcement is activated immediately when any microcrack is initiated.

The reinforcing ability of zirconia particles is a consequence of the phase transformation, i.e. the spontaneous change from the tetragonal to the monoclinic phase. The phase transformation is accompanied by a volume change of 4 % of the zirconia particle, i.e. a linear expansion of 1,3%. Spontaneous phase transformation is a well known principle in material science. For example, the properties of high performance steels also rely on spontaneous phase transformation from austenite to martensite.

It should be emphasized that the ability of phase transformation is the precondition for any benefit of the zirconia within the material. The composite is designed such that phase transformation occurs when it is needed, i.e. in the case of microcrack initiation. In contrast to pure zirconia (which draws its high strength from the same principle) the main source of the stability of the tetragonal phase is the embedding of the zirconia particles in the alumina matrix. In contrast, the stability of pure zirconia only relies on the chemical stabilisation (i.e. doping with yttria) and the grain size, which should not exceed a certain range. This is the most important distinction of the composite material BIOLOX® delta to pure zirconia. In particular, the mechanical stabilization of the stiff alumina matrix is not sensitive to any ageing effect.

3 Comparison of component and material testing

As described above, it is the objective of this paper to show the intrinsic stability of the material BIOLOX® *delta* against any live-time limiting effects. This is mainly accomplished by using well defined specimens according to the requirements of international standards for surgical materials (e.g. ISO 6474 or ASTM F 603).

However, it may be useful to compare the data obtained from test specimens like bending bars to the properties of hip components. For this purpose, in figure 3 the results of ball head fracture tests and of 4-point bending tests of several powder batches are presented.

The burst tests on BIOLOX® *delta* ball heads (figure 3 left) refers to a standard design diameter 28mm, taper 12/14. Each individual data point in the figure represents the average value of a test series of at least 7 ball heads. The strength (figure 3 right) refers to 4-point bending tests according to ASTM F 603. The strength as it is derived from bending tests represents the maximum stress in the specimen at the moment of fracture. Each individual data point represents the average of 30 specimens. As it is shown, plenty of data is available for either ball head burst tests and strength. The larger scatter in the burst tests is a consequence of the smaller number of specimens used in this test.

From these data, one is able to compare the strength of the material to the performance of the components. The average burst load is 83 kN and the average strength 1400 MPa. Usually the load acting on an artificial hip joint is expressed as multiples of the body weight (BW). A reasonable value for 1 BW is 1 kN (apprx. 100 kg). From various experiments and calculations it is derived that the maximum load which can occur in-vivo in an extreme situation (e.g. one leg balancing of a stumble) is apprx. 9 (BW. This result gives an impressive indication of the large safety margin which is provided from the use of the material BIOLOX® *delta* as a surgical material.

On this basis, the live time experiments were designed. The long term stress on the specimens was chosen such that a reasonable margin in comparison to maximum in-vivo loading is provided. Thus, for the cyclic loading tests two stress levels of 300 MPa and 600 MPa were chosen. From the comparison discussed under figure 3 the stress level of 300 MPa is equivalent to a component loading of 18 × BW, i.e. double the maximum in-vivo load. (300 MPa / 1400 MPa ≈ 18 BW / 83 BW). Analogous, 600 MPa correspond to 4-fold maximum in-vivo load. Using these stress levels it is analysed whether the material is able to resist extreme conditions over a live-time relevant period.

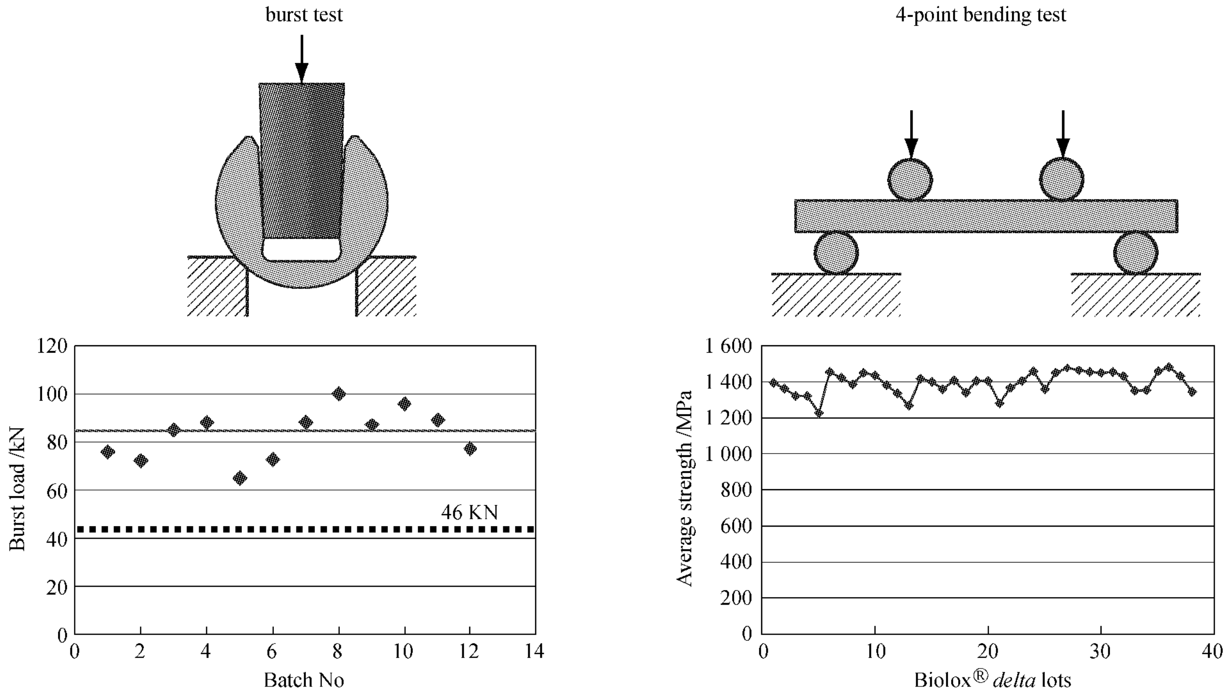

Fig.3 Burst load of BIOLOX® *delta* ball heads (28 +3,5) and strength of bending tests

4 Discussion of live time limiting effects

The analysis discussed in this paper refers to a combination of ageing and fatigue experiments. Any degradation of the material after long term treatment is evaluated by comparison of residual strength to the as-received state.

Ageing is a relevant issue for all zirconia containing materials. The transformation from the tetragonal to the monoclinic phase can be triggered by the so-called hydrothermal attack[3-5]. "Hydrothermal" means that this particular ageing effect only takes place in aqueous environment at elevated temperatures. It has been shown that a critical temperature range for hydrothermal ageing is around 134 - 150°C. Obviously, this temperature is not realistic for human body environment. However, today it is well accepted that the ageing in the human body environment can be simulated in an accelerated test using autoclaving conditions of 200 KPa water steam and 134 °C. Various authors claim that 1 hour autoclaving conditions are equivalent to 2 - 4 years in the human body[1-2]. Consequently, accelerated ageing is also required as a standard test for pure zirconia as a material for surgical implants. Usually, it is investigated whether the residual strength of the material deteriorates after ageing. The concept which is presented here does not only rely on the residual strength but also to the performance of the material at cyclic loading.

Fatigue is defined as the material sensitivity against cyclic loading. Limited fatigue resistance is usually observed when the materials ability of crack resistance is continuously deteriorating during the cycling. Even materials which offer plastic deformation and high crack resistance like metals can substantially loose their strength during cyclic loading and exhibit brittle fracture. In general, ceramics show higher fatigue resistance in comparison to metals. However, the fatigue effects of ceramics also depend on their specific crack resistance mechanisms. As it was shown under figure 2, the crack resistance of BIOLOX® *delta* is rather complex. Thus, it is necessary to demonstrate whether this material may show any degradation at cyclic loading.

As a special feature of this investigation, hydrothermal ageing and fatigue are combined. According to the theoretical background one should consider if any ageing effect may also impair the fatigue resistance or vice versa.

5 Result of live-time experiments

The experiments were designed to simulate a combination of worst case conditions on BIOLOX® *delta*. The specimens were prepared according to the 4-point bending configuration as it is shown in figure 3 (right). As discussed above, the live time limiting effects ageing and cyclic fatigue were combined in these tests.

Two stress levels (300 MPa and 600 MPa) are chosen for the cyclic loading tests. The lower stress level was applied for 20 Mio cycles, the higher stress level for 5 Mio cycles. All tests were performed in Ringer's solution. The accelerated ageing was simulated by 5 h and 100 h treatment in autoclaving conditions which is equivalent to 10 years and 200 years in vivo. All specimens used for cyclic loading where proof tested prior to the cycling. Table 1 shows the test matrix including the number of specimens used.

Tab.1 Test matrix with number of tested samples

Autoclaving /h	no cyclic load	300MPa, 20×10^6 cycles	600MPa, 5×10^6 cycles
0	30	6	6
5	30	6	6
100	30	6	6

Using 30 specimens is usually required for determination of strength. However, due to the time consuming experiments applying the cyclic loading it was decided to use only 6 specimens for each cyclic loading test. After the treatment, the residual strength of the specimens was determined and compared to the initial strength. Furthermore, the monoclinic phase content was measured for each treatment.

As the most amazing result the yield of specimens surviving all the tests was 100 % in all cases. Even most severe conditions (i.e. 100 h autoclaving, 600 MPa cyclic load) did not reveal any premature failure. It should be recalled that this stress level represents 4 times the highest load level at worst case conditions in-vivo. We can thus conclude that the reliability of BIOLOX® *delta* exceeds by far the necessary requirements for reliable surgical components.

Table 2 shows the results of the post - test analysis including residual strength and monoclinic phase content. There is a marginal natural scatter in residual strength which is always expected for ceramic materials. However, statistical analysis using Student's t-test did not reveal any significant deviation of all strength results.

Tab.2 Residual strength and monoclinic phase content after diverse treatments

Autoclaving/h		no cyclic load	300MPa, 20×10^6 cycles	600MPa, 5×10^6 cycles
0	Strength/MPa	1346	1433	1284
	Monoclinic phase content/%	18	33	43
5	Strength/MPa	1332	1248	1361
	Monoclinic phase content/%	22	35	42
100	Strength/MPa	1234	1308	1300
	Monoclinic phase content/%	30	33	47

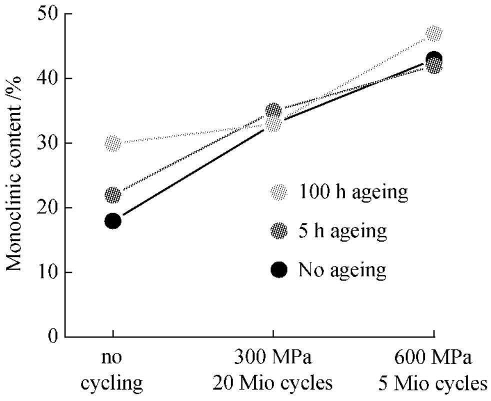

Fig.4 Inrease of monoclinic phase content at cyclic loading

In contrast, there is a clear tendency of an increase in monoclinic phase content both, after autoclaving and after cyclic loading, which is illustrated in figure 4. For example, the test series without autoclaving shows an increase of monoclinic phase content from 18 % in the initial state to 43 % after 5 Mio cycles at 600 MPa. It must be concluded that the cyclic mechanical loading at a high stress level (600 MPa) of almost half the strength (1.4 GPa) activated the reinforcing ability of the material. As discussed under figure 2, a high mechanical stress triggers localized phase transformation which prevents any further crack propagation. Obviously the increased amount of monoclinic phase content does not deteriorate the strength of the material. This important conclusion is independent from the source of the phase transformation. In other words, when the phase transformation is activated either by accelerated ageing, cyclic fatigue or a combination of both, the residual strength remains on the initial level.

The reported monoclinic phase content should be discussed with respect to the composition of the material. The monoclinic phase content shown in figure 4 is related to the total zirconia. As described above, the total volume content of zirconia in the alumina matrix is 17 %. In order to assess the effect of the zirconia content one should refer the amount of monoclinic phase relative to the total volume of the material. For example, the highest amount of monoclinic phase in a region close to the surface measured in this study is 47%. This equals a total monoclinic content of only 8% (= 47% × 17%). Obviously, even under extreme conditions the amount of monoclinic phase in this material is well under control. In this context it is elucidative to remind that in pure zirconia an amount of 20 % monoclinic phase is allowed according to the standard ISO 13356 already in the initial state before accelerated ageing. It is thus concluded that the specific composition of BIOLOX® *delta* provides inherent protection against improper phase transformation.

6 Conclusions

The material BIOLOX® *delta* has been exposed to extreme conditions (accelerated ageing and cyclic loading in Ringer's solution). It has been shown that even a combination of worst case conditions does not reveal any premature failure. Furthermore, it was shown that the residual strength remains on the initial level. A certain amount of phase transformation was observed during the tests. The highest amount of monoclinic phase relative to the total volume of the specimen was 47 %. The residual strength was not affected by the phase transformation.

In other studies it was shown that BIOLOX® *delta* performs extremely well in severe wear tests[6]. These results are also attributed to the reinforcing mechanism in the material. These exciting results promote the confidence that BIOLOX® *delta* offers the highest probability of long term durability in well designed artificial joint systems.

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