ATLANTIS PRESS
Advances in Health Sciences Research, volume 16
International Conference on Health and Well-Being in Modern Society (ICHW 2019)

Change of Microhardness of Zirconium Dioxide During Mechanical and Heat Treatment

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Abstract **– The paper studies the hardness of zirconium dioxide used in dentistry for the production of dental implants. The purpose of the study was to analyze the change of material microhardness after mechanical treatment and machining with subsequent firing. In total 30 zirconium dioxide samples were made, which were exposed to machining: diamond drill of high and low granularity, sandblasting and processing with zirconium dioxide disk. It was found that after machining without firing the hardness of material decreases by at least 26 %, and in some cases, even more in comparison with situations when regenerating firing is performed after processing. As a result of the study we came to a conclusion that additional processing of zirconium dioxide implants leads to heating of a material thus changing the structure of its surface, which can potentially cause changes of dental implants. If any clinical case requires additional processing it is preferable to use diamond grinding points with subsequent regenerating firing and sandblasting without additional firing.**

Key words – dental implants, heat treatment, zirconium, microhardness

I. Introduction

These days the dentistry has taken a new quantum leap due to new structural materials for the production of dental implants and technologies of their processing [1, 2].

One of such revolutionary technologies, which radically changed the dentistry is computer-aided design technology and automated production of dental implants – CAD/CAM technology [3].

CAD/CAM technologies improve quality and save time of production of dental implants. Now dentists have an opportunity of indirect restoration in the chair side mode within 1–2 hours, which substantially reduced time for the production of implants and increased their quality.

CAD/CAM technologies increased the amount of new materials with high esthetic and physical characteristics, which are currently applied in dental laboratories [4, 5].

Metal-free ceramics based on zirconium dioxide has been widely used in dentistry. This material has high strength, hardness, biological compatibility and good esthetics [6, 7].

Despite certain progress in the development of dental prosthetics, new technological processes, techniques and materials, frequency of complications and percentage of orthopedic recure due to various complications are still high. The fracture of dental implants (tooth crowns and bridges) is the most frequent reason for orthopedic recure. Mechanical treatment of the surface of synthesized frame from zirconium oxide such as grinding with diamond tools and sandblasting can cause the release of excessive amount of energy and lead to volume damage of a crystal lattice or even to change of ZrO_2 phase from tetragonal to monocline [8, 9]. In a frame this may be expressed by tension across boundary surfaces, which can break an implant [10].

According to some authors, the hardness of ceramics on the basis of ZrO_2 increases after machining if the material is subjected to secondary temperature regenerating firing [10, 11].

The above poses the following question: how will mechanical properties of this material change at its processing after firing and mechanical treatment [12].

II. Problem statement

To study the change of microhardness and surface structure of ceramics based on zirconium dioxide after mechanical and heat treatment.

III. Materials and methods

For the study we used ceramics on the basis of zirconium dioxide – Superfect Zir SHT. At the 1st stage using 3D modeling and milling 30 square examples with a 10 mm size of the side and 4 mm thickness were made. The material was subjected to firing in a furnace for zirconium sintering (MIHM-VOGT GmbH & Co.) at a temperature of 1500 °C within 12 hours, according to the manufacturer's instruction. The samples were divided into 5 groups and their surface was mechanically processed through certain intervals within 20 seconds with air-to-water cooling. The 1st group – control. For the 2nd groups standard diamond drills with granularity of 120-135 microns were used as processing tools.

The surface of materials of the 3rd group was processed with a grinding stone from zirconium dioxide. The samples of the 4th group were subjected to sandblasting.

In the 5th group the samples were ground with a diamond drill and an air-turbine handpiece as it is done in clinical conditions, the granularity of a diamond drill made 50-60 microns. Then the samples of the 2, 3, 4, 5 groups were divided into subgroups depending on heat treatment: materials of the 1st subgroup were subjected to mechanical treatment only. In the 2nd subgroup after tool processing the material was exposed to regenerating firing in a ceramic furnace at a temperature of 1000 C within 15 minutes. At the 2nd stage we studied the microhardness of a material surface on LOMO PMT-3 microhardness tester. The microhardness tester is a microscope to measure microhardness of various materials. The principle of operation of the device is based on a diamond pyramid hardness test under a certain loading and subsequent measurement of linear diagonal of the obtained result. Loading was applied in 500 g for 15 seconds so that the diamond contacts with a surface of the studied subject. The hardness number was calculated according to the following formula:

$$H = \frac{1854P}{D^2} \tag{1}$$

where N – hardness number in kg/mm^2, P – loading in grams, C – diagonal in microns. At the final stage we studied the change of the structure of a material surface after processing with optical multiplication of x300.

Statistical data was processed via Statistica 6.0 software.

IV. Results and discussion

During the study we received the following results. The microhardness of zirconium dioxide surface in the control group made on average 1882.96 kg/mm^2 (Table 1).

TABLE I. MICROHARDNESS OF ZIRCONIUM DIOXIDE MATERIALS IN THE CONTROL GROUP (KG/MM^2)

Samples	Hardness (kg/mm^2)
1	1932,82
2	1932,82
3	1783,24
Average indicator	1882,96

Through processing with a diamond drill with the granularity of 120–135 microns (group No. 2) the hardness of the material of the 1st subgroup, which was subjected to mechanical treatment only, decreased up to 1436.99 kg/mm^2, and in the 2nd subgroup, which was exposed to regenerating firing after processing, it increased up to 2163.41 kg/mm^2 (Table 2).

TABLE II. MICROHARDNESS OF ZIRCONIUM DIOXIDE MATERIALS IN GROUP 2 (KG/MM^2)

Samples	Hardness of subgroup 1 (without regenerating firing)	Hardness of subgroup 2 (regenerating firing)
1	1425,61	2165,52
2	1425,61	2161,11
3	1459,75	2163,41
Average indicator	1436,99	2163,41

In the 3rd group the surface of samples was processed with a special grinding stone for zirconium dioxide. The microhardness of zirconium dioxide surface after mechanical treatment made 958.48 kg/mm^2 and after regenerating firing – 1392.64 kg/mm^2 (Table 3).

TABLE III. MICROHARDNESS OF ZIRCONIUM DIOXIDE MATERIALS IN GROUP 3 (KG/MM^2)

Samples	Hardness of subgroup 1 (without regenerating firing)	Hardness of subgroup 2 (regenerating firing)
1	970,87	1392,64
2	952,29	1391,53
3	952,29	1393,75
Average indicator	958,48	1392,64

After sandblasting (group 4) we received different results. Without regenerating firing the surface hardness increased and made 2102.4 kg/mm^2. After firing it decreased up to 1650.37 kg/mm^2 (Table 4).

TABLE IV. MICROHARDNESS OF ZIRCONIUM DIOXIDE MATERIALS IN GROUP 4 (KG/MM^2)

Samples	Hardness of subgroup 1 (without regenerating firing)	Hardness of subgroup 2 (regenerating firing)
1	2102,04	1650,97
2	2101,03	1649,07
3	2103,05	1651,07
Average indicator	2102,04	1650,37

The processing of a material in clinical conditions with an air-turbine handpiece with the granularity of 50–60 microns decreased up to 1531.83 kg/mm^2 (Table 5).

TABLE V. MICROHARDNESS OF ZIRCONIUM DIOXIDE MATERIALS IN GROUP 5 (KG/MM^2)

Samples	Hardness (kg/mm^2)
1	1530,53
2	1533,13
3	1531,83
Average indicator	1531,83

The study of zirconium dioxide ceramic surface with optical multiplication of x300 resulted in the following. The samples of the control group had smooth corrugated surface. In the 2nd group insignificant amount of cavities was noted on the surface of samples. In the 3rd group after processing with a grinding stone from zirconium dioxide we observed some changes of a surface in the form of cavities and expressed material stippling. The surface of a material in the 4th group after sandblasting became fine-grained. The processing of a material in clinical conditions in the 4th group with a diamond drill led to multiple cavities and expressed lobing on the surface of material.

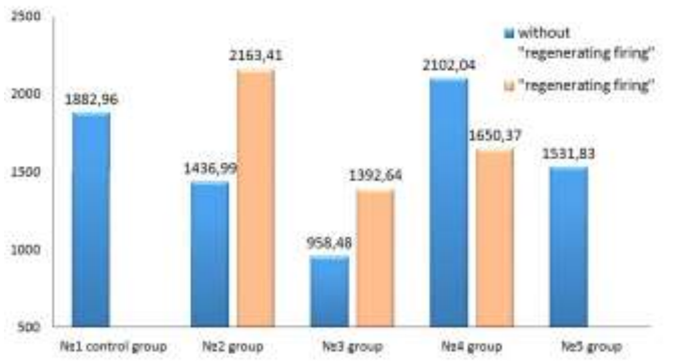

Fig. 1. Microhardness of a material within the studied groups

The analysis of results revealed that the roughest changes of hardness and structure of zirconium dioxide ceramic surface were received after processing with a grinding disk from zirconium dioxide and an air-turbine handpiece with a diamond drill in clinical conditions. The samples of the 2nd and 4th groups gave the most satisfactory results, which is show in Figure 1.

Thus, the obtained data show certain repeatability and regularity: as a rule, the hardness of a material after mechanical treatment considerably decreases, which is caused by microcracks and internal stress in the material. Regenerative firing after mechanical treatment increases hardness, except sandblasting. We believe that this is caused by the fact that the surface of a material is contaminated with sand particles during sandblasting, and during firing the purity and structure of a material decreases due to these inclusions.

V. CONCLUSION

The study made it possible to conclude that additional processing of dental implants from zirconium dioxide leads to heating of a material thus changing the structure of its surface, which may potentially change the dental implant. If any clinical case requires additional processing it is preferable to use diamond grinding points with subsequent regenerating firing and sandblasting without additional firing.

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