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APPLICATION OF TRIBOMETRY IN INVESTIGATIONS OF BIOMATERIALS

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Abstract: *Development of materials for manufacturing of the chirurgical implants in medicine - biomaterials (metals, polymers, ceramics and composites) is directly determined by characteristics and nature of the tissue, organs and systems that are being replaced or supplemented.*

Modern material investigations at micro-and nano-level enable introspection into new aspects of material behaviour and offer possibilities to significantly improve systems in use, from different aspects of changes in material production technologies or application of surface technologies modifications or coating technologies.

Biomaterial investigations from a tribology point of view offer contribution to testing realised in this area, especially with use of novel devices for research in area of nanotribology. In this paper, some tribological characteristics of Ti-6Al-4V alloy, aimed for medicine applications, are presented. Investigation is realised at CSM nanotribometer in accordance with testing conditions significant for this type of biomaterial.

Keywords: *Biomaterials, Ti alloys, Nanotribometer*

1. INTRODUCTION

Development of materials for manufacturing of the chirurgical implants in medicine - biomaterials (metals, polymers, ceramics, and composites) is directly determined by characteristics and nature of the tissue, organs and systems that are being replaced or supplemented. Very often, this information is of more qualitative then quantitative character and existence of broader database of material properties is necessary as a condition for development and improvement of existing biomaterials. Inability to precisely determine biomaterial behaviour during the longer period of time and under different living organism states, imposed detailed testing of implants material with recognition of influence of large number of system parameters.

Necessity to know environment parameters including features of living tissue materials, but also general and special biomaterials characteristics, exploitation stability, material selection and its design, as well as many other requested characteristics, is conditioned by complex nature and functioning of biomaterials.

Data from practice in area of biomaterials applications shows that material development for certain types of biomaterials in use, had been done even over a decade, to only be applied for relatively short time and to be replaced by a new or improved ones.

The first funded nanotechnology programme in Europe comprised areas of biomaterials for information technology and nanobiology (Finland, 1997–1999) [1]. From those first programs, a broad variety of investigations in area of biomaterials have been conducted. Mechanical integrity and wear resistance of a biomaterial is vital for long term implantable devices, such as total joint replacements, which need to function effectively over periods of 20 years or more. Laboratory tests are necessary to help optimize the biomaterial performance, among which nanotribometer testing can be very significant for extended wear testing. Allowed wear rates for such a system are very strictly defined and are significantly more sensitive to a whole range of influences. It is important however, to evaluate their durability, what is done through tribological testing.

2. TRIBOMETRY OF BIOMATERIAL

Tribometry, in general, represents an area of tribology that comprises means and methods of measuring: friction forces in contact zones; wear of tribosystem elements; temperature; surface roughness; contact surfaces sizes; contact strain etc. [2]. The measurement of friction force and the calculation of the coefficient of friction are of great importance for many tribosystems and for some it is even especially critical, like for brakes, clutches or similar control system, where the friction force determines the system behaviour. Another major challenge is to anticipate the type of wear to which a components will be subjected and accordingly applying a specific model of testing. Surface modeling, from various aspects like contact type, temperature, lubricating modes and other case based important features, are also crucial for other types of testing.

Very important part of nanosciences, in general, is research work in area of new materials and new production technologies development. There are number of already implemented new materials which greatly improve specific systems under study. Development of medicine related materials is of special importance. Soft matter, like polymers and biomaterials are studied by nanotribometry instruments and testing procedures, in order to validate theoretical findings or to study newly found effects.

It is, however, very important to connect, in more extent, existing theoretical findings with practical data obtained from some tribometry device. Information on, for instance, modeling the wear types for realized tribological experiments is of significant importance for further advancements in understanding mechanisms of wear development on macro-, micro and nanoscale at systems of interest.

Tribo testing of biomaterials are somehow different than those conducted within metal working industries, due to a fact that it is mostly done with soft materials compared to metals. Area of biomaterials investigations is yet different because research here must be done with multidisciplinary approach taking into account series of diverse data. Investigations are currently done to establish more accurate methodologies for predicting wear in complex environments of existing tribo-systems, what would greatly enhance development of more durable biomaterials for application in human related issues. Friction estimation in oscillation nano- and micro-tribometry, experimental results for controlled load, wear tests of polymer film, all can be realized at nanotribometer instrument, thus helping resolving previously mentioned questions.

Extremely complex issues of biomaterials and their application at medicine in order to improve lives of millions of people are subject to investigations at several R&D areas and can be successfully addressed only through joint acting of experts from different areas of research, where tribology is one necessary part of it. Nanotribometry is powerful tool that can offer answers to existing questions.

2.1 Biomaterial properties

Biomaterials as an exciting area of science exist for half a century now. Many companies have been investing large amounts of money into the development of new products, therefore making this field of science as one with constant growth and improvement. Biomaterial science comprises elements of medicine, biology, chemistry, tissue engineering and maybe above all, of materials science. Research activities are aimed at understanding of the fundamental relationships between material properties and the biomedical applications of materials, taking into account specific applications of materials and also rules that determined the choice of those materials.

In general, all biomedical materials belong to one of following categories: biological materials, polymers, ceramics, metals or composites. Important aspects of research activities in this area encompasses principles of materials science and mechanical properties, some of which are fatigue, creep, mathematical models of materials, tribology, wear, lubrication, testing methodology etc.

Important ceramics which have use in medicine are bioceramics that are inert ($Al2O3$, zirconias, silicon nitride). Its applications in medicine are mainly aimed at artificial joints, coatings, heart valves. Some problems with use of ceramics are wear particles and poor union to bone.

Composites are used for their mechanical behaviour and their use to optimize mechanical properties and behaviour. Applications of composites are mainly in dentistry and medicine for fillings, artificial skin, bone plates, bone substitutes and devices. Problems with composite materials in medicine are matrix failure and delamination.

Group of the major metals in use in medicine comprises stainless steel, cobalt chrome, titanium and titanium alloys. Applications are mainly in orthopaedics (e.g. joints, plates, screws, rods, bars), dentistry (dental implants, braces, fillings) neurological implants (e.g. cochlear, pacemaker) and general surgery (tools). Problems with these metals that have been reported over the years of applications are corrosion, heavy metal ion release, wear, ductile failure etc.

The beginning of the metal material use in medicine is in relation to the first metal devices to fix bone fractures in the late eighteenth century; the first total hip replacement prosthesis was implanted in 1938; and in the 1950s and 1960s, polymers were introduced for cornea replacements and as blood vessel replacements [4]. Today, biomaterials are largely used (Figure 1).

Estimates of the numbers of biomedical devices incorporating biomaterials used in the United States in 2002 include [4]:

- Total hip joint replacements: 448,000
- Knee joint replacements: 452,000
- Shoulder joint replacements: 24,000
- Dental implants: 854,000
- Coronary stents: 1,204,000
- Coronary catheters: 1,328,000.

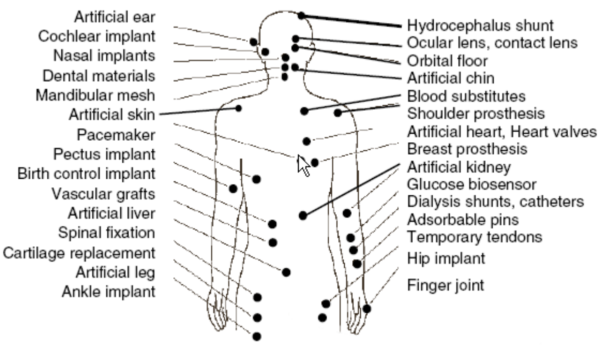

Figure 1. Impact of Biomaterials [4]

In the field of biomaterials many novel processing techniques and methods have been developed for the construction of improved material characteristics. Many more materials are being investigated for potential applications, including novel uses of natural materials, combinations of natural and synthetic materials and new structures designed to be closer to the in vivo cellular environment.

2.2 Titanium alloys

In comparison with other biomaterials the metallic biomaterials possess the outstanding property of being able to endure tensile stresses, which, in the case of alloys, may be extremely high and also of dynamic nature. This is the reason why alloys are widely used as structural materials for skeletal reconstructions if high acting loads are expected to occur. Typical examples for such highly loaded implants are hip and knee endoprostheses, plates, screws, nails, dental implants, etc. However, their application is not limited exclusively to these highly loaded elements, but also for functional devices such as various conducting wires, pumps etc.

The main requirements that must be satisfied by all biomaterials are corrosion resistance, biocompatibility, bioadhesion (bone ingrowth), biofunctionality (adequate mechanical properties, especially fatigue strength and a Young's modulus as close to that of the bone as possible), processability and availability [3].

The biocompatibility of most metallic biomaterials is based on a passive oxide layer which is always present on the metal surface and which will be restored quickly (milliseconds) after damage [3]. These oxide layers, similar to alumina, show an inert behaviour towards the surrounding tissue. Metallic materials exhibiting such a passive and highly inert oxide layer that belongs to a group of stainless steel and a cobalt-chromium base alloy have been available for about 60 years.

About 25 years ago, due to the favorable properties of the special metals niobium, tantalum and titanium, their application as biomaterials became of interest. Especially titanium and its alloys began to be a subject of research in area of biomaterials.

Titanium and its alloys stand out primarily due to their high specific strength and excellent corrosion resistance. Their first application was primarily in the aerospace and the chemical industries. But increased application of titanium appeared in last decades also in areas such as architecture, chemical processing, medicine, power generation and transportation.

The main characteristics of titanium are as follows [5]:

1. high affinity to the gases oxygen, nitrogen and hydrogen
2. high reactivity to all metals producing intermetallic compounds
3. relatively low Young's modulus
4. relatively low thermal conductivity
5. tendency to stick to the tools

Two different crystal structures (hcp and bcc) and the corresponding allotropic transformation temperature exist for pure titanium. This is of central importance since they are the basis for the large variety of properties achieved by titanium alloys.

Usually titanium alloys are classified as α, $\alpha+\beta$, and β alloys [5]. According to recent literature, today more than 100 titanium alloys are known, of which, however, only 20 to 30% have reached commercial status. Of these, the classic alloy Ti-6Al-4V covers more than 50% of usage. Another 20 to 30% are unalloyed titanium [5].

Among the α+β, alloys, Ti-6Al-4V is by far the most popular titanium alloy. These alloys can be heat-treated to develop a variety of microstructures and mechanical property combinations. The very reason that titanium alloys can exhibit a wide variety of is because titanium and its alloys exhibit a broad range of phase transformations. Three different microstructures of Ti-6Al-4V alloy depending on a manufacturing technology are shown in a Figure 2 [6].

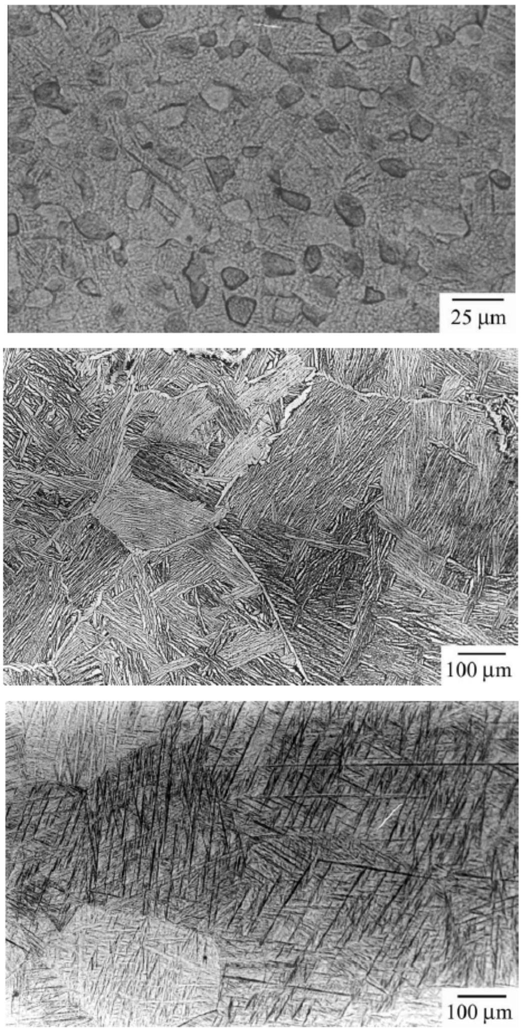

Figure 2. Different microstructures of Ti-6Al-4V alloy

In the field of biomedical applications, titanium is used for prosthetic devices for bone and joint implants, heart valves, and dental implants. These are made from CP titanium, Ti-6Al-4V, or recently developed alloys such as Ti-6Al-7Nb.

3. TRIBOLOGICAL INVESTIGATIONS OF BIOMATERIALS

In comparison the different materials show a different behaviour according to the demands. A corrosion resistant material may not necessarily be biocompatible and, contrarily, a more biocompatible material may be less corrosion resistant. Especially fretting corrosion may represent a problem in articulating devices like knee joints or plate/screw systems. It is therefore necessary to investigate, in number of details, materials aimed for medicine purposes. Tribological approach, though being an important one, is just one step in that process.

For biomaterial design, engineers need to consider the physiologic loads (axial rotation, flexion, extension, and lateral bending) to be placed on the implants sufficient structural integrity. Material choices also must take into account biocompatibility with surrounding tissues, the environment and corrosion issues, friction and wear of the articulating surfaces, and implant fixation either through osseointegration (the degree to which bone will grow next to or integrate into the implant) or bone cement. One of the major problems for these devices is purely materials-related: wear of the implants' elements. A review of tribo-biomaterials used in human related areas is given in Table 1.

Table 1. Summary of Tribo-biomaterials [7]

Material	Application	Major Properties Description
Alloy: Titanium Alloys, Titanium Aluminum Vanadium Alloy, Cobalt Chromium Alloy, Cobalt Chromium Molybdenum Alloy	Total joint replacement	Wear and corrosion resistance
Inorganic: diamond-like carbon	Biocompati ble coatings	Reduced friction and increased wear resistance
Ceramics: Al2O3, ZrO2, Si3N4, SiC, B4C, quartz, bioglass(Na2O-CaO-SiO2-P2O5), sintered hydroxyapatite (Ca10(PO4)6(OH)2)	Bone joint coating	Wear and corrosion resistance
Polymers: Ultrahigh molecular weight polyethylene, Polytetraflouroethylene (PTFE) Poly(glycolic acid)	Joint socket Interpositio nal Implant temporoman dibular joint(Jaw) Joint bone	Wear and corrosion resistance Low coefficient of friction Elastic with less wear
Composites: Specialized silicone polymers	Bone joint	Wear, corrosion, and fatigue resistance

Materials characterization methods are mechanical testing, chemical analysis, microstructural characterization, coatings evaluation, development of special test techniques and component design and analysis include structural analysis, reliability and probabilistic modeling, engineering design optimization, failure analysis. Characterization is particularly difficult when the materials are used for human body that needs *in vivo* test conditions.

Considering tribological testing, wear test is a basic method to evaluate the durability of biomaterials. Other aspects of tribological investigations can encompass cyclic fatigue testing.

The properties of biomaterials to date still do not meet the application requirements. The part of many potential causes of failure for implants elements, among other are material fracture, wear, and corrosion. Wear of the articulating surfaces within implant system produce wear particles of sizes of a submicron and larger. The negative biological effects of these wear particles are considered to be one important factor that limits the long term clinical performance.

Current research trends, therefore, encompasses development of novel and improved material combinations for the surfaces of implants. Such development is in turn dependent on an improved understanding of the wear processes involved and how these are influenced by different material properties and conditions. Equally important is it to develop reliable and predictive methods for stimulating the wear processes under *in vitro* conditions, preferentially in an accelerated way.

The long term engineering objective is to develop a material (surface) combination which does not produce biologically harmful wear particles under the physiological conditions that occur within human related systems.

3.1 Application of nanotribometer device

Nanotribometer device offers simple and efficient way of measurements of friction or adhesion at nano-scale. It belongs to a group of instruments for nano-scale investigations that require contact with a sample, so contact mode operation is used [8].

Linear reciprocating nanotribometer is used for simulation of many real life cases, where typical reciprocating motion is present. Most contact geometries can be reproduced including Pin-on-Plate, Ball-on-Plate and Flat-on-Plate. It is equipped with appropriate software that can generate wear rates or do calculation of the Hertzian stress.

Mechanical integrity and wear resistance of a biomaterial is vital for long term implantable devices, such as total joint replacements, which need to function effectively over periods of 20 years or more. Laboratory tests are necessary to help optimize the biomaterial performance, among which nanotribometer testing can be very significant for extended wear testing. Allowed wear rates for such a system are very strictly defined and are significantly more sensitive to a whole range of influences. Coating biomaterials with hard, inert coatings of, for instance, diamond-like-carbon has been suggested as a mean to improve the lifetime of the femoral head in total hip replacement prostheses [9].

New testing methods using nanotribometer device gives valuable information on behaviour of observed materials. Investigation conducted using linear reciprocating module of nanotribometer as one result provides friction coefficient curves, such as a diagram shown in Figure 3.

The wear behaviour of a sliding system depends on many factors, including the properties of the specimen and counterface materials, their interaction with the environment and the experimental conditions. A careful characterization of the sliding wear mechanisms of metal alloys is particularly significant due to a fact that many human related systems belongs to this type of motion.

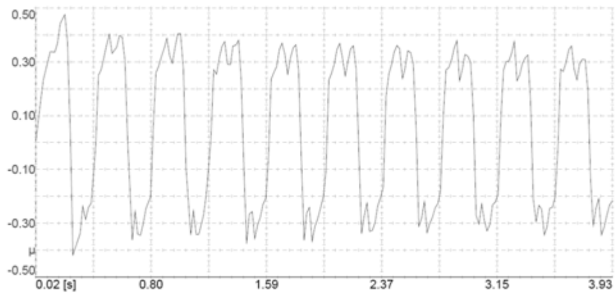

Figure 3. Friction coefficient vs. time curve, part of a report generated by CSM nanotribometer

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3.2 Microscopy techniques for biomaterials

Available knowledge on imaging techniques applied to biomaterials, especially in the orthopaedics field enabled significant advances in this area. The advances involve characterization of surfaces and cell-material interactions.

An atomic force microscope (AFM) and Scanning Electron Microscopy (SEM) are widely used as an effective tool for analysis in area of material investigations. Microscopic imaging is essential for complete understanding of the effects of tribological processes on developmental processes at the contact of two moving surfaces.

Confocal laser-scanning microscopy (CLSM) is a rapidly advancing technique used to produce crisp and precise images of thick specimens in fluorescent and reflective light modes and as a technique is especially useful in biomaterial investigations.

In comparison with AFM and SEM techniques traditional optical microscopy is maybe less powerful, but still have its advantages. The first one naturally, is the price.

Classical metallurgical microscope has been developed in such a way during last years that they can be effectively used for characterisation of metallic surfaces. They are equipped with good quality digital camera and appropriate software for various manipulation of the sample picture captured. Magnification of modern metallurgical microscope can be up to 1000x, what means that the photograph of the sample surface of order of 1-10 microns can be effectively displayed for analysis. Photography of the wear track of Ti-6Al-4V alloy (Ra=0.14μm) at 1000x magnification, taken by MEIJI digital camera (MEIJI MT8530 metallurgical microscope at Tribology Center at Faculty of Mechanical Engineering, Kragujevac) is shown in Figure 4.

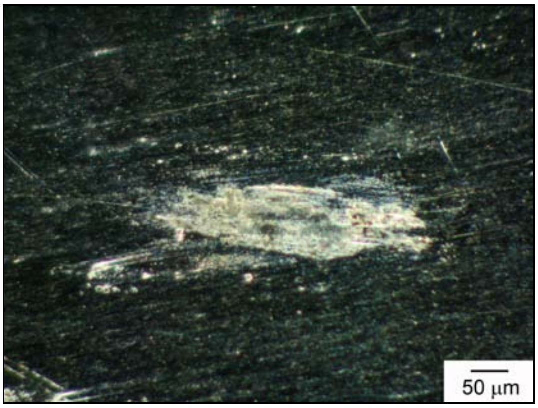

Figure 4. Wear track on Ti-6Al-4V alloy sample at 200x magnification

Good quality optical microscope usually has Brightfield/Darkfield illumination with Incident & Transmitted illuminators and simple polarization observation that enable various modes of sample manipulation. Beside that, new infinity corrected optical system is developed and widely applied for more precise view. Various types of filters are available for obtaining better surface photography (blue/green clear filter, ND50 neutral density filter, polarizing filter). Different eyepiece micrometers and metal stage micrometer are also possible for easier sample manipulation and measuring.

Combination of various imaging techniques and different instruments makes possible significant advancements for researchers in area of biomaterials development.

As one important task in scope of these investigations is extensive tribological testing of biomaterials in order to explain and understand in more details acting friction and wear mechanisms during the life cycle of biomaterials in use such as titanium alloys. Many issues in relation with behaviour of human related systems have not yet been carefully addressed.

4. CONCLUSION

Integration of technologies for industrial applications with focusing on new technologies, materials and applications to address the identified needs by the different areas of human life has become a priority for researchers, among which, nanosciences have a distinguished role. Nanotribometry has become a powerful tool for helping resolving diverse issues in multidisciplinary approach.

New testing methods, with application of nanotribometer instrument, give valuable information on behaviour of materials. Tribological approach is very important in research activities of biomaterials in order to improve already existing ones or to develop completely new materials with superior characteristics. Also, available knowledge on imaging techniques applied to biomaterials enable significant advances in this area.

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