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RESEARCH

# Tribometry of Materials for Bioengineering Applications

*Development of materials for biomedical implants (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, such as improvement of manufacturing technologies or surface technologies modifications.*
*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.*

***Keywords:*** *Biomaterials, Ti alloys, Nanotribometer*

## 1. INTRODUCTION

Development of materials for manufacturing of the biomedical implants in medicine - biomaterials (metals, polymers, ceramics) 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 human related environment, impose necessity to investigate implants material taking into account the influence of large number of system parameters. Complex nature and application of biomaterials require knowledge about different variables in regards to biocompatibility, corrosion behaviour, mechanical characteristics, stability and durability in aggressive environment, wear mechanisms and wear rates during exploitation and many other.

Data about use of biomaterials shows that material development for certain types of implants, is a process done over years. Then after its application for relatively short time it is replaced by new or improved solutions. Clinical exploitation require constant improvements of biomaterials in use, since some features can be determined only after years of real applications.

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.

According to a new market research report, ‘Global biomaterials Market (2009-2014)’, published by MarketsandMarkets (www.marketsandmarkets.com), the total global biomaterials market is expected to be worth US$58.1 billion by 2014, growing at a CAGR of 15.0% from 2009 to 2014. The biomaterials market today has already crossed $28 billion. The biomaterials market is still in a growing phase, with about 100,000 heart valves, 200,000 pacemakers and 1 million orthopaedic devices implanted worldwide every year. Increase in applications has increased the demand for new biomaterials from 8% to 15%. Improved patient benefits form the most important factor stimulating market growth for biomaterials. The other market drivers are increase in aging population, rising awareness, shorter product approval time, and larger application area.

Additive Manufacturing Technologies, Rapid manufacturing, Rapid prototyping (RP), 3D printing are general terms describing a variety of methods used to directly fabricate physical objects

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(three-dimensional models and end-use products) from electronic CAD data. Today's additive technologies offer advantages in many applications compared to classical subtractive fabrication methods such as milling or turning: Objects can be formed with any geometric complexity without the need for elaborate machine setup or final assembly. Rapid prototyping systems is used to make prototypes of parts through relatively fast process without having to invest the time or resources to develop tooling or other traditional methods. This has resulted in their wide use by engineers as a way to reduce time to market in manufacturing, to better understand and communicate product designs, and to make rapid tooling to manufacture those products. Medical applications are also routinely using the technology. Studies ranging from fundamental research to the latest applications of virtually all classes of materials are of great importance in order to provide detailed information on new modern materials.

The American Society for Materials and Testing (ASTM), in January, 2009, has set up the Technical Committee F42 on Additive Manufacturing Technologies with the scope to promote knowledge, stimulate research and implement technology through the development of international standards in this area. The Society of Manufacturing Engineers as the world's leading professional society advancing manufacturing knowledge and influencing more than half a million manufacturing practitioners annually has members in more than 70 countries and is supported by a network of technical communities and chapters worldwide, emphasized Rapid Prototyping Practitioners and Researchers at RAPID 2009 Conference as Life-Saving Technology and Advancements in Hybrid Metal Manufacturing. Bioinformatics market is expected to exceed $2 billion by 2010, according to Battelle Report, 2006, USA.

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 [2] can be very significant for extended wear testing. Allowed wear rates for such a system are very strictly defined and significantly more sensitive to a whole range of influences. It is important however, to evaluate materials durability, what is partly done through tribological testing.

## 2. TRIBOMETRY AT SMALL SCALES

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. [3]. The measurement of friction force and the calculation of the coefficient of friction are of great importance for tribosystems in use and for some it is even especially critical, like for human joints implants, 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 of contact type, temperature, lubricating modes and environment, are also crucial for valid testing. It is very hard to setup simulation model for laboratory testing of tribological systems in relation to real human systems.

Very important part of nanosciences, in general, is research work in area of new materials and development of new production technologies. 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 laboratory device. For instance, information on modeling wear types is of utmost 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 softer 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 diversified data. Investigations are currently done to establish more accurate methodologies for predicting wear in complex environments of existing biotribo-systems, what would greatly enhance development of more durable biomaterials for application in human related systems. Friction estimation on nano- and micro-scales, experimental results for controlled load, wear tests of polymer film, all can be realized at nanotribometer instrument, thus contributing to previously mentioned issues.

Mechanisms by which lubrication and wear reduction within biotribological systems (e.g. joints) act and develop are not fully understand yet and needs to be investigated further. It is usually the case of boundary lubrication in aggressive environment of human fluids. Laboratory simulations as well as in-situ testing are needed.
Identification of wear debris and its acting on system in question have been investigated by many researchers leading to identification of adequate couples of materials (metal on metal, ceramic on metal, etc), as well as improvement of existing contact systems by application of surface engineering technologies (e.g. multilayered coatings). However, there still exists a need to study these systems from aspects of their long term clinical performance.

Low wear rate within biotribological systems are necessary characteristic of a biomaterials in use for hip and knee replacement systems. In case of total hip replacement, for instance, wear debris released in joint system produce highly unwanted consequences, such as osteolysis leading to lowered implant performance and further revision surgeries. Many variables influence biotribological system in relation to system design and material properties.

Many improvements of existing biomaterials have been introduced into clinical practice over the last years. More stable materials have been investigated by many laboratories across the world (low reactivity to oxygen, better biocompatibility, lower density, increased durability, decreased reactions in human fluids environment, etc.).

Models of systems in question are necessary to be able to further investigate different parameters that are interconnected to each other. Application of RP technology in the field of biomedical engineering represents cutting-edge RTD solution. It offers broad possibilities to manufacture and provide end users with ready parts made of extensive range of biomaterials for further investigation. Biomaterials are very expensive and hard to obtain with precisely set up properties, since they are not commercially produced in large series. RP technologies offer rapid and quick production of complex shapes with predefined geometry according to specific cases. Even the most complex live systems (bones, skull) can be precisely replicated in short time, effectively and by relatively low price.

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.

### 2.1 Trends in research of biomaterial

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 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 obligatory requests that determine 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, wear, lubrication, testing methodology, standardization, etc.

Important ceramics which have been used 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 bone ingrowth and other [4].

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 [5].

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 [6]. Today, biomaterials are largely used (Figure 1).

Estimates of the numbers of biomedical devices incorporating biomaterials used in the United States in 2002 include [6]:

- 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.

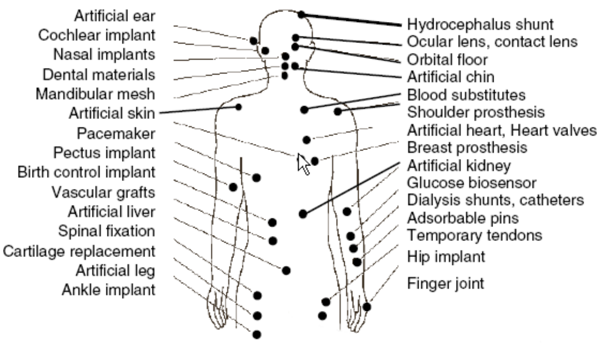


**Figure 1.** Impact of Biomaterials [6]

In the field of biomaterials many novel processing techniques and methods have been developed for the construction of improved material characteristics. Many novel alloys are being investigated for potential applications, including application 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, may be extremely high and of dynamic nature. That 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 surgical wires.

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 [7].

The biocompatibility of the Ti metallic biomaterials is based on a passive oxide layer which is always present on the surface and which will be restored quickly (milliseconds) after damage [7]. These oxide layers 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 high affinity to the gases oxygen, nitrogen and hydrogen, high reactivity to all metals producing intermetallic compounds, low Young's modulus and low thermal conductivity [8]. There are two different crystal structures (hcp and bcc) and the corresponding allotropic transformation temperature of pure titanium (cp Ti). They are the foundation for the large variety of properties achieved by titanium alloys.

Usually titanium alloys are classified as $\alpha$, $\alpha+\beta$, and $\beta$ alloys [8]. According to recent literature, more than 100 titanium alloys are known today, of which, however, only 20 to 30% have reached commercial status. Standard alloy Ti-6Al-4V covers more than 50% of usage. Another 20 to 30% are unalloyed titanium [8].

Among the $\alpha+\beta$, 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 properties 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 Figure 2 [9].

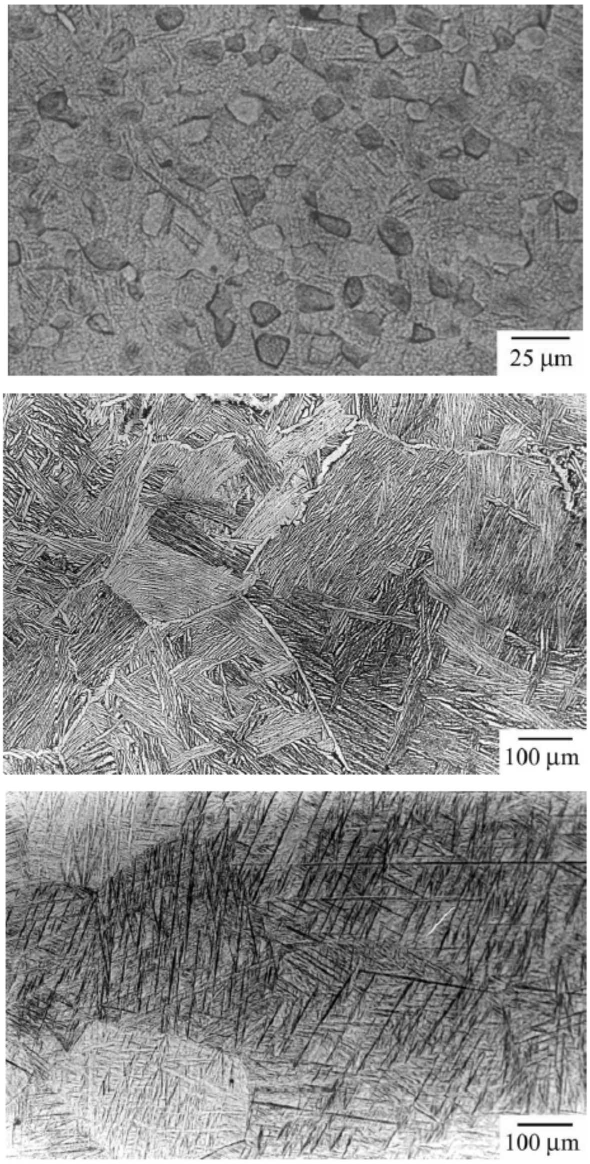


**Figure 2**. Different microstructures of Ti-6Al-4V alloy

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 Ti, Ti-6Al-4V, or recently developed alloys such as Ti-6Al-7Nb.

## 3. TRIBOLOGICAL INVESTIGATIONS OF BIOMATERIALS

Corrosion resistant material may not necessarily be biocompatible and 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 medical purposes. Tribological approach is important in that process.

For biomaterial design, engineers need to consider the physiologic loads (axial rotation, flexion, extension, and lateral bending) to place sufficient structural integrity on the implant. 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 by bone cement. One of the major problems for these devices is purely materials-related: wear of the elements. A review of tribo-biomaterials used in human related areas is given in Table 1.

Materials characterization methods are mechanical testing, chemical analysis, microstructural characterization, coatings evaluation, development of special test techniques and component design and analysis including structural analysis, reliability and probabilistic modeling, engineering design optimization, failure analysis [10]. 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, creep, coatings, etc. Another important area of research activities in relation to biomaterials are investigation of possibilities to continuously measure and monitor wear rate within a system, in laboratory conditions and as well as in-vivo.

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.

**Table 1.** Summary of Tribo-biomaterials [11]

| 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 | Biocompatible coatings | Reduced friction and increased wear resistance |
| Ceramics: $Al_2O_3$, $ZrO_2$, $Si_3N_4$, SiC, $B_4C$, quartz, bioglass($Na_2O$-CaO-$SiO_2$-$P_2O_5$), sintered hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$) | Bone joint coating | Wear and corrosion resistance |
| Polymers: Ultrahigh molecular weight polyethylene, Polytetraflouroethylene (PTFE) Poly(glycolic acid) | Joint socket Interpositional Implant temporomandibular 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 |

Current research trends encompass development of novel and improved material combinations for the implant surface [12]. Such development is in turn dependent on 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 [12]. Linear reciprocating nanotribometer is used for simulation of many real life cases, where typical reciprocating motion is present (total hip replacement). Most contact geometries can be reproduced including Pin-on-Plate and Ball-on-Plate. It is equipped with appropriate software that can provide wear rates or 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. Biomaterials with hard, inert coatings (e.g. DLC coatings) have been suggested as a mean to improve the lifetime of the femoral head in total hip replacement prostheses [13].

Nanotribometer investigations offer valuable information about behaviour of observed materials [2]. Tests conducted using linear reciprocating module of the nanotribometer give friction coefficient curves, such as a diagram shown in Figure 3.

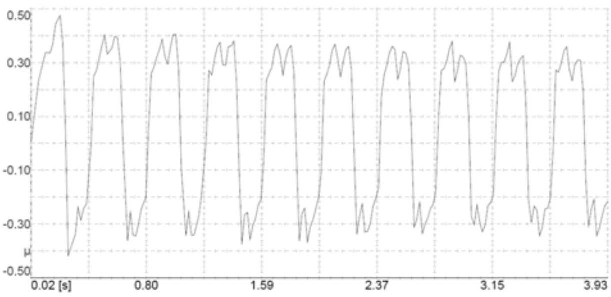


**Figure 3.** Friction coefficient vs. time curve, part of a report generated by CSM nanotribometer

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.

Wear in biotribological systems is characterized by ultra low wear rates of order of nanometers per hour. Sophisticated equipment, such as nanotribometer offer possibility to record and monitor such small levels of wear rate.

Extremely complex issues of biomaterials and their application at medicine in order to improve lives of millions of people are subject of investigations 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 supporting it.

### 3.2 Microscopy techniques for biomaterials characterization

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 [14]. Microscopic imaging is essential for complete understanding of the effects of tribological processes on processes within a contact of two articulating surfaces.

Scanning electron microscope has the following principle of work. Inside electron microscope casing there is an electron gun and several coils, the condensing lenses, the scan coil and the objective lens. Finally there is the target - the sample that is observed. The condensing lenses focus the electrons into a tight beam. The scan coil makes the beam play over the target and the objective lens helps to focus the image. As the beam plays over the target, secondary electrons are not loose. These are gathered by the detector, amplified, then fed to the display where the final image is created.

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 advantage is the equipment 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.

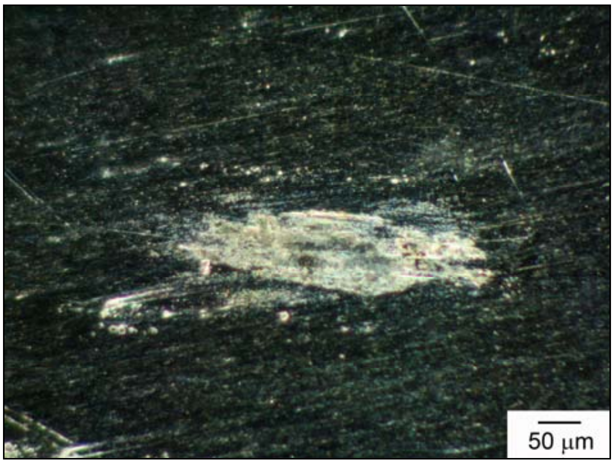


**Figure 4.** Wear track on Ti-6Al-4V alloy sample at 200x magnification

Good quality optical microscope usually has Brightfield/Darkfield illumination with Incident & Transmitted illuminators and simple polarization observation that enable various modes of sample manipulation. 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 offer significant advancements for researchers in area of biomaterials development.

Important task in scope of these investigations is extensive tribological testing of biomaterials in order to understand and explain acting friction and wear mechanisms during their life cycle. Many issues in relation with behaviour of human related systems have not yet been fully understood.

## 4. CONCLUSION

Integration of technologies for industrial applications with focus 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.

Multidisciplinary approach to the research in area of Material Science, Rapid prototyping (RP) technologies, Reverse engineering, 3D visualization and Bioengineering, from both aspects of theory and practice, will surely significantly contribute to improvement of many existing human related biotribological systems.

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 those existing materials or to develop completely new ones with superior characteristics.

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