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ScienceDirect
Procedia Engineering 69 (2014) 104 – 111
Procedia Engineering
www.elsevier.com/locate/procedia

24th DAAAM International Symposium on Intelligent Manufacturing and Automation, 2013

Micro-Nanometrologically and Topographic Characterization of Nanostructured Surfaces

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Abstract

Micro-nanometrologically and topographic characterization of materials is important to determine features of different surfaces that are used in many areas. During the time, depending on the application, the surfaces deteriorate having a complex deterioration mechanism. The changes that appear on these surfaces and wear particles can be characterized using different equipment and techniques. A mechatronic system made up of an atomic force microscope and a robotic nanomanipulator is used at INCDMTM, in order to characterize steel, titanium alloys, CoCr alloys or polycrystalline surfaces. It is a proper system to study surfaces of different materials from the tribological point of view.

Keywords: atomic force microscopy; nanopositioning; tribological parameters; topographic characterization; nanostructures

1. Introduction

Materials characterization is important to determine features of different surfaces used in many areas. Mechanical properties of materials surfaces or interfaces of thin films deposited on a surface have to be known when we want to use these materials for a special application. When materials are parts of systems used for different periods of time, their surface deteriorates due to the pressures produced by mechanical movements of the systems it is part of. The deterioration process varies from one system to another, in some cases with a complex mechanism. The basic types

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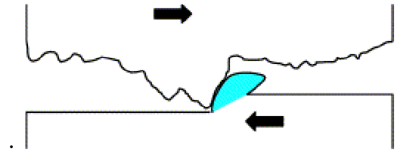
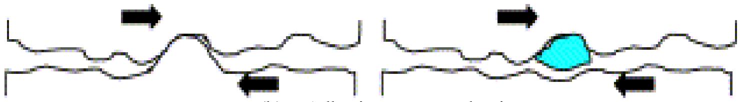
doi:10.1016/j.proeng.2014.02.209

of wear mechanisms (Fig. 1) are abrasive wear, adhesive wear, wear with the third body and fatigue wear [1]. In some systems there is a complex mechanism containing a combination of the basic ones.

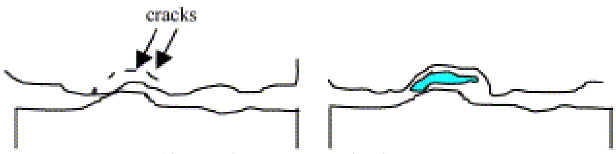


(a) Abrasive wear mechanism



(b) Adhesive wear mechanism

(c) Third body wear mechanism



(d) Fatigue wear mechanism

Fig. 1. Wear mechanisms [1].

Abrasive wear [2] represents the removal of material from one surface by the other. A local high point called "asperity" on the surface of the harder material will produce wear particles into the softer material.

Adhesive wear is produced where the localized bonding of the two surfaces occurs, such that the attachment force is stronger than the yield strength of the material. A small piece of material is removed from one surface and is attached to the other.

Three-body wear appears when metallic, ceramic or polymeric wear particles are trapped between two moving surfaces [3,4].

Fatigue wear can lead to subsurface cracks propagating and flaking off of particles from the surface. High subsurface stresses can also be caused by third bodies between the two articulating surfaces leading to accelerated fatigue wear.

All the wear particles that appear as a result of the wear processes occurring in different systems have different characteristics. Techniques with complex mechanisms were used in order to see these particles and the changes produced on the surfaces areas.

Different methods of investigation were used during the years to establish the surface topography and to obtain tribological parameters. Tribological parameters help to the characterization of the worn surfaces.

Characterization of worn surfaces and determination of its tribological parameters can be achieved by non-contact surface measurements using atomic force microscope, confocal microscope, scanning interferometer or laser. Techniques based on scanning probe microscopy (SPM) were developed to study elastic and inelastic properties of surfaces, interfaces at micrometer and nanometer scales. Scanning acoustic microscopy (SAM) is used to study the material properties at micrometer scale. Atomic force microscopy is a popular method for these types of studies.

2. Experimental studies

At INCDMTM, characterizations and determinations of tribological parameters for different materials surfaces are realized. For example, surface roughness of some femoral heads from hip prostheses [5,6] realized by ceramics, CoCrMo [7], Ti6Al4V, steel coated with TiN [8]; polycrystalline diamond compact surfaces COMPAX 1321; metals – component material of different mechanical parts was determined.

Using roughness analysis it is possible to determine tribological parameters like minimal, maximal and average height of the surface. Some other important parameters that can be determined are:

- Ten point height (S_z) expresses surface roughness by the selected five maximal heights and hollows, nm
- Surface skewness (S_{sk}) characterizes the non-symmetry of distribution. If the asymmetry is different from zero the distribution is non-symmetrical. The asymmetry equals to zero for the symmetrical distribution.
- Coefficient of kurtosis (S_{ka}) characterizes the distribution spread.

For these measurements we are using an NTEGRA Atomic Force Microscope working in the non-contact mode.

The working principle of an AFM is to measure the interaction force between the tip and sample surface using special measuring heads, made of a cantilever with a pointed end. AFM images are processed using Nova SPM software.

The samples are positioned on the base unit of AFM using a robotic nanomanipulator that has more degrees of freedom including rotation for control of orientation. Thus, it can be used to manipulate in space 0D objects (spherically symmetric) to 3D objects.

A Hexapod positioning system for Micro-Movement F-206 is used in this experiment [9]. 6-axis positioning system F-206 consists of an attachment position system and a control unit. A keyboard and a monitor for the control unit may be used to control F-206 system. System's mechanics uses a parallel – cinematic positioning system. The system provides 6 degrees of freedom and a minimal increase of movement of 0.1 μm. Workspace boundaries are not parallel to the axes, but it cannot overcome a rectangular solid which is given by the limits of movement X, Y and Z. The control unit is equipped with integrated software to define a pivot point anywhere inside or outside the workspace of F-206 system. Rotation around this pivot point may be ordered for any combination of the 3 rotation axes. Digital command system processes complex positioning and elements of movement, including scanning procedures and alignments using optical or analogue response signals from more than 2 meters.

The connection between NTEGRA Atomic Force Microscope and the Hexapod positioning system for Micro-Movement F-206 is done with a gripper that allows nanopositioning of the samples used. A scheme of the obtained complex system used to characterize and analyse the studied surfaces is presented in Fig. 2.

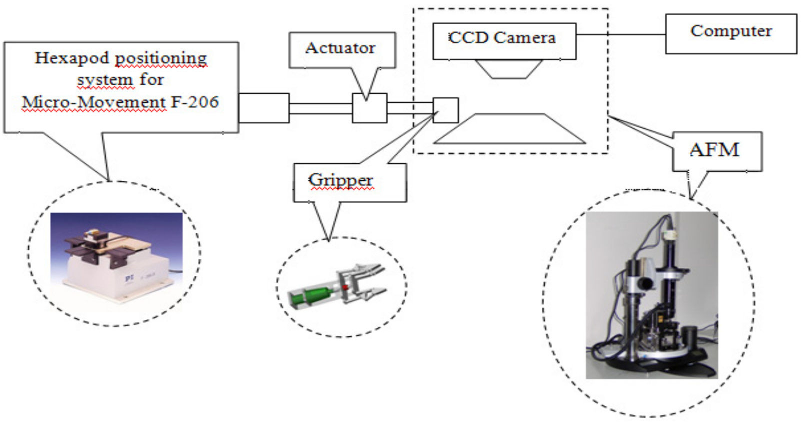


Fig. 2. A complex mechatronic system made up of NTEGRA Atomic Force Microscope and F-206 alignment and positioning system with six axes.

3. Results

This paper presents some of the results of the analysis regarding the topography and tribological parameters of femoral head structures, a polycrystalline compact diamond COMPAX 1321 surface and surface of a microgripper.

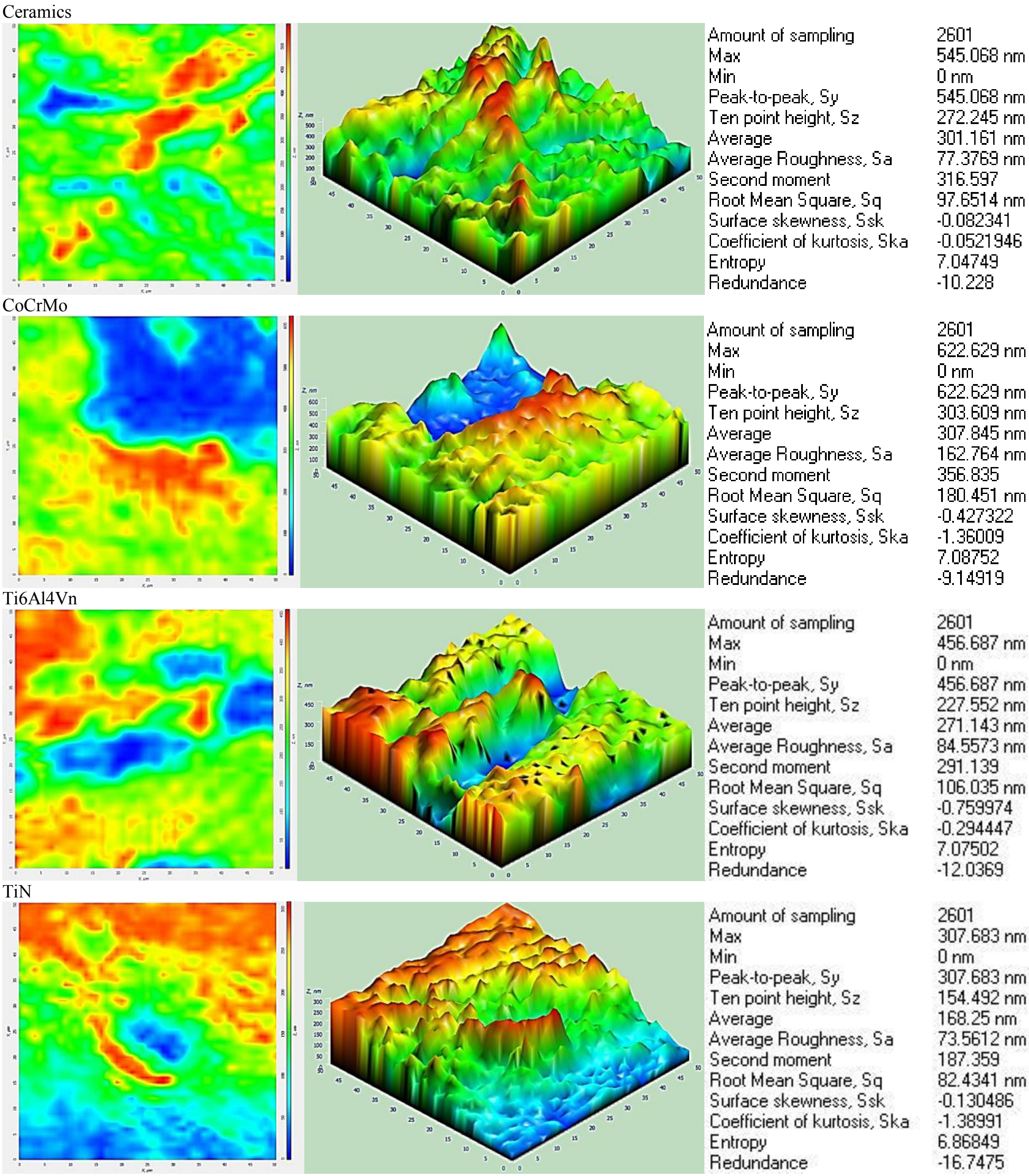
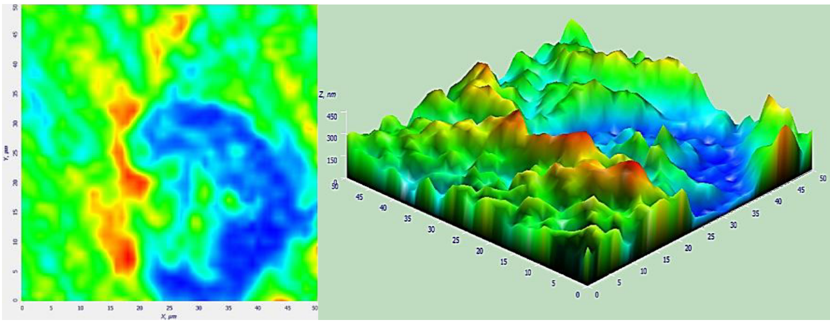


Fig. 3. Topographic characterizations and tribological determinations of femoral heads surfaces using the complex mechatronic system with NTEGRA atomic force microscope.

Femoral heads of total hip prostheses made of ceramics, CoCrMo, Ti6Al4V, TiN were the main surfaces characterized in our institute (Fig. 3). This has been done in order to find out more information about the wear rate in the case of some prostheses used for 10 – 15 years in the human body. Having this information, a procedure and a material to improve these surfaces was possible to be determined, in such a way to have a smaller wear rate.

As it is shown in the examples presented in Fig. 3, the surfaces of all the femoral heads, made of different materials, have a high deterioration degree. Roughness, an important tribological parameter, has different values, in different parts of the same femoral head. These values depend on the movements of the body that used the prostheses and on the material femoral heads are made of. After a big number of experimental results it was observed that the average roughness is the lowest for TiN surfaces. So, it was concluded that this material can be used in the femoral heads structures for a longer period of time with a lower degree of damage compared with the other materials studied by us and presented in literature.

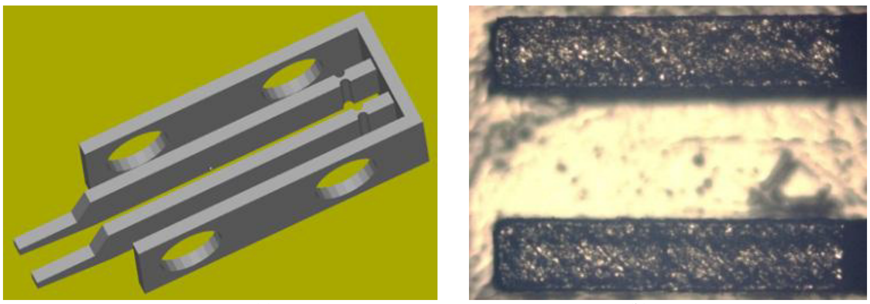
Besides the hip prostheses surfaces, polycrystalline compact diamond COMPAX 1321 surfaces were also characterized. This material can be used as an active part of a lathe tool for processing metallic/non-metallic materials. Before using polycrystalline compact diamond COMPAX 1321 in such an application, characterization with the complex mechatronic system with NTEGRA atomic force microscope had been done. Tribological parameters obtained are presented in Fig. 4, together with the 3D image of the surface. This is just one example from all measurements that shows that the polycrystal that we studied has a non-uniform surface.



Amount of sampling	2601
Max	461.982 nm
Min	0 nm
Peak-to-peak, Sy	461.982 nm
Ten point height, Sz	225.415 nm
Average	193.397 nm
Average Roughness, Sa	81.0254 nm
Second moment	217.169
Root Mean Square, Sq	98.7934 nm
Surface skewness, Ssk	0.0662898
Coefficient of kurtosis, Ska	-0.65042
Entropy	7.12918
Redundance	-12.0112

Fig. 4. Topographic characterizations and tribological determinations of polycrystalline compact diamond COMPAX 1321 surfaces using the complex mechatronic system with NTEGRA atomic force microscope.

The surface of a microgripper made by the laser sintering method in Rapid Prototyping Laboratory of INCDMTM has also been characterized using the mechatronic system described before (Fig. 5). The laboratory is based on High-tech EOS M 270 - Laser sintering system for metal powders.



(a) (b)

Fig. 5. Microgripper produced using EOS M 270 Titanium Version laser sintering system. (a) General view; (b) Microgripper arms seen using AFM.

Selective Laser Sintering (SSL) is a family of technological methods that can build a solid body of various types of materials (plastic, metal, ceramic, including rare metals or with special physico-mechanical and biocompatibility properties) by solidification of powder material following the successive exposure of powders layers to laser beam of different variable powers [10].

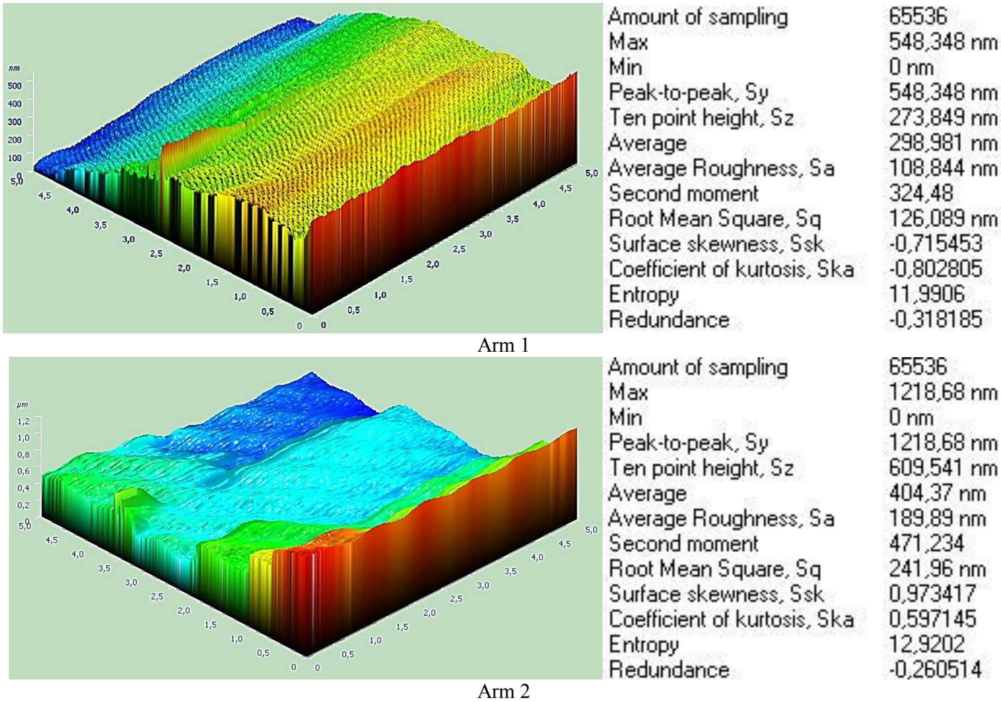


Fig. 6. Roughness of microgripper arms surfaces measured using NTEGRA Probe NanoLaboratory.

The microgripper, made of EOS CobaltChrome MP1, is a piece realized in order to be used as a part of a positioning system from our institute. EOS CobaltChrome MP1 is a fine powders (Co, Cr, Mo, Si, Mn, Fe, Ni) mixture for processing on EOSINT M 270 systems, which produces parts in a cobalt-chrome-molybdenum-based superalloy. This class of superalloys is generally characterized by excellent mechanical properties (strength, hardness a.s.o.), corrosion resistance and temperature resistance.

The surface of this piece was characterized to evaluate the uniformity obtained after selective laser sintering process. For a complete characterization, the microgripper's surface was divided into three sections whereon the micro-nanometrological and topographic characterizations were performed. Fragments of 5 x 5 μm were characterized. The surfaces of both arms were also topographic and metrological characterized.

Using the mechatronic system containing the NTEGRA AFM all surface protrusions were observed. Topographic characterization of microgripper arms is presented in Fig. 6. For these, values of average roughness of 137.041 nm (arm 1) and 169.751 nm (arm 2) were obtained. As is shown in Fig. 7, the average roughness for other fragments of the piece has different values, in different parts of the microgripper. Average roughness value of 123.54 nm was obtained. These values and images presented demonstrate that following the sintering process, surfaces with a medium uniformity were obtained. These are not completely uniform surfaces, but not totally non-uniform surfaces.

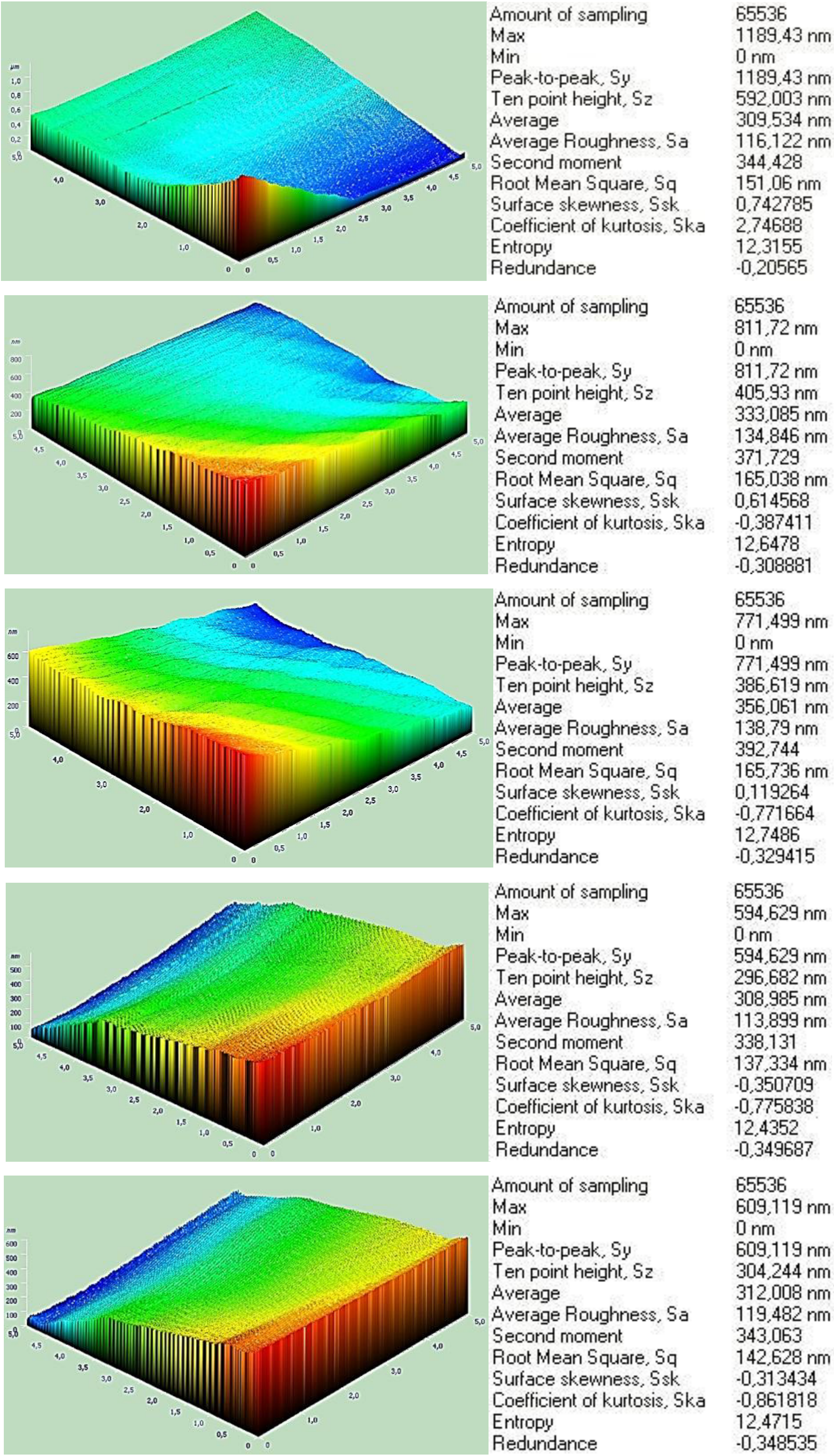


Fig. 7. Roughness of microgripper's surface measured using the complex mechatronic system with NTEGRA atomic force microscope.

After these studies, we observed that it is possible to achieve a high uniformity surface after processing the surface obtained by selective laser sintering. Pieces obtained by this method can further be processed by any known mechanical process: milling, turning, drilling, boring, grinding, broaching, super-finishing.

Further researches and micro-nanometrologically and topographic studies will be conducted after processing the surface obtained by sintering. Some other measurements of femoral heads already used or with improved surfaces will also be made in the future.

4. Conclusion

Characteristics of materials surfaces that will be used in different applications are important to know their quality. Another method to see the quality of a material is to characterize the destroyed surfaces of it in order to determine the hardness of the final surface. This is the reason why is necessary to choose a technique and to have a system for these studies.

In our institute, a clear micromechanical characterization of the femoral heads surfaces of hip prostheses has been done after their use in the human body and after an attempt of improving. Beside this, we also studied the surface of some polycrystals and the surface of some pieces obtained by Selective Laser Sintering (SSL).

At INCDMTM, these studies were made using AFM because its images display high quality and dense nanocrystalline structure of studied surfaces. An NTEGRA Probe Atomic Force Microscope has been connected with a Hexapod positioning system for Micro-Movement F-206 in order to obtain a complex positioning of the sample, followed by surface characterization and determination of few tribological parameters. In this way was possible to determine the roughness and the hardness of some materials used for implants or polycrystals used in processing equipment. An important application was the fact that with this mechatronic system we had the possibility to determine the quality of some pieces just obtained and to see if some other processing method is necessary before using these pieces for other different applications.

Taking into account all the results obtained, we shall continue the research using the described mechatronic system. In this way, the topographic characterization of the pieces and materials surfaces that we are often using can be improved.

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