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Metrology Techniques for Nanometer-Scale Structures, Roughness and Shape

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Surface Science Western has a group of metrology tools including atomic force microscope (AFM), stylus profilometer, confocal scanning laser microscope (CLSM) and the forthcoming optical coordinate measuring machine [CMM, acquired by Dr. Teeter (https://www.teeterlab.com/) through his CFI grant]. Each instrument has its strength and weakness, which is why, collectively, the lab is capable of providing the necessary means to study surface morphology from nanometer scales to centimeter scales. This technique note aims to present an introduction on how to (a) select an instrument suitable for the analysis requirement for a specific surface finish and (b) determine what types of information and what scales are needed to quantify the surface finish.

1. AFM for revealing nanometer-scale structures

Atomic force microscope is a mechanical probe technique and measures surface morphology at nanometer scales due to its sharp probe (the tip radius is on the order of 10 nm). As shown in **Figure 1**, AFM (Park Systems XE-100) provides a real-space three-dimensional (3D) image of a surface through the detection of an interaction between a sharp mechanical probe (containing a cantilever and a tip) and the surface features.

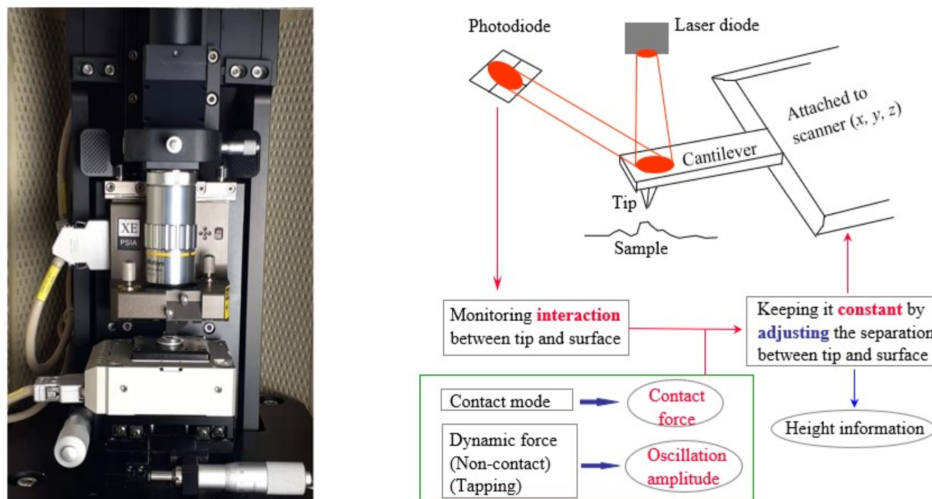

***Figure 1**. Left: An AFM having an x-y scanner separated from the z head. Right: Schematic illustration of AFM principle: while scanning the tip across the sample surface (x, y), the system adjusts the distance (z, which is thus the measure of the height of the sample surface features) between the tip and the sample surface to maintain a constant contact force (contact mode) or oscillation amplitude (dynamic force mode).*

Depending on the operation mode of AFM, the interaction can be contact force (contact mode) or oscillation amplitude of the cantilever (dynamic force mode or tapping mode), which is used as the feedback parameter to adjust the distance between the tip and the sample surface. The tip scans the sample surface in one direction (e.g., x) to measure heights over a number of points (e.g., 256), which is called fast scan direction. Upon completing the first scan, the system moves to the next y position (slow scan direction), which is perpendicular to the fast scan direction, and continues to complete scan lines of the same number of points (256). This way, height information at points evenly distributed over the lateral dimensions (e.g., 256 × 256 pixels) is obtained, which constructs 3D surface morphology with predetermined lateral dimensions (x and y) and measured height (z).

Figure 2 shows 2D AFM images in false color scales (i.e., higher spots are represented with brighter colors), revealing the fiber-like network structure of a biaxially oriented polypropylene (BOPP) film and the mounds formed upon a brief UV/ozone treatment, which is due to the oxidation of the polymer. The scan area is 2 µm × 2 µm and the height range is 25 nm, demonstrating that AFM is a powerful technique in mapping surface morphology of extremely fine structures.

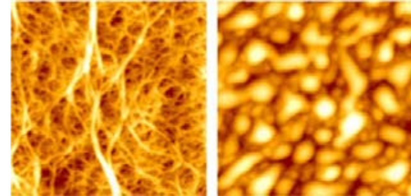

Figure 2*. AFM images of a BOPP film before (left) and after (right) treatment).*

Shown in **Figure 3** is an AFM image of self-assembled monolayers (SAMs) of octadecylphosphonic acid (OPA) formed on a silicon wafer. Also displayed are two profiles isolated from the image, showing that the thickness of the SAMs is around 1.7 nm. The combination of the extremely high height resolution (better than sub-nanometers) and excellent lateral resolution (several nanometers - limited by the tip apex size) makes AFM the best, if not the only technique to investigate the coverage of SAMs.

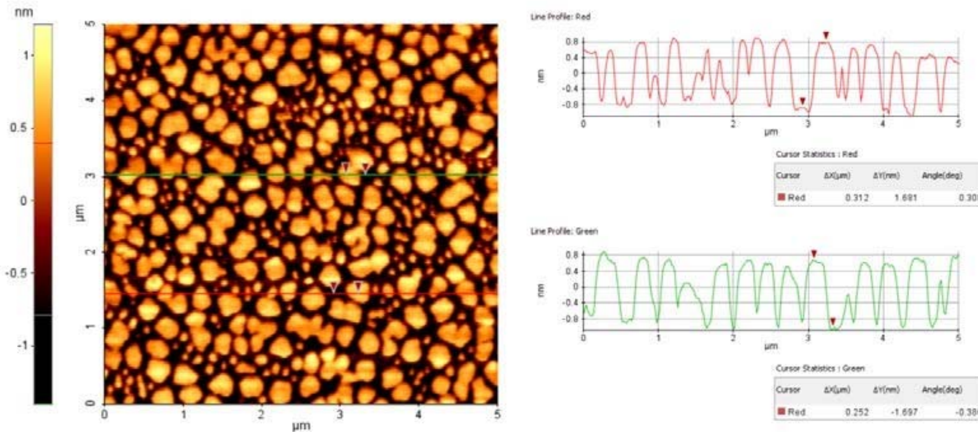

Figure 3*. Left: AFM image of OPA SAMs formed on a silicon wafer. Right: Two profiles isolated from the AFM image showing the thickness of the SAMs.*

As demonstrated above, AFM is powerful in imaging structures at nanometer scales or evaluating the surface morphology of smooth substrates. On the other hand, most AFMs have a maximum scan range of 50-100 µm and height rang of 10-15 µm, thus limiting their applications for samples having larger features and/or rougher surfaces. This limitation of AFM calls for the use of a stylus profilometer to handle larger scale structures.

2. Stylus profilometer for roughness evaluation

Stylus profilometer is the traditional technique used in industry for quantifying surface finishes via roughness measurement. A stylus profilometer (KLA-Tencor P-17) is shown in **Figure 4**, along with an illustration showing the capacitive force sensor. At the end of the stylus there is a diamond tip serving as the probe that scans the surface of a sample with a certain applied force. The most commonly used diamond tip has a radius of 2 µm, which is due to the fact that smaller tips are deficient in mechanical durability and larger tips impact too much on spatial resolutions. The scan range and height range can be as large as 20 cm and 1 mm, respectively. While the height resolution of this technique is a couple of nanometers, the lateral resolution, due to the rather large size of the diamond tip, is a couple of micrometers.

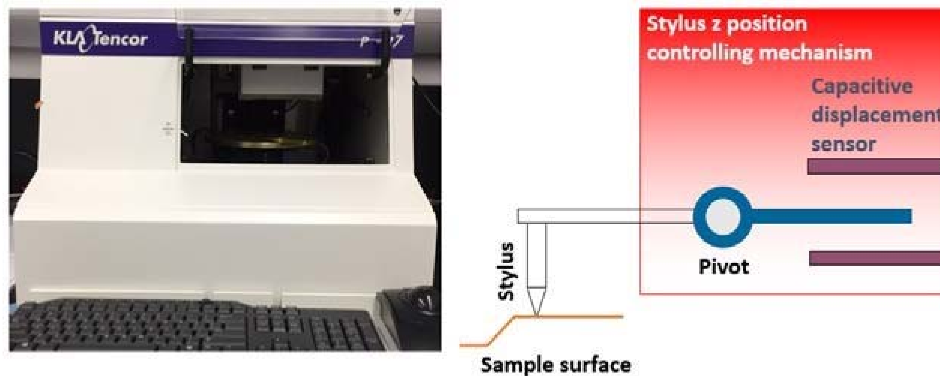

***Figure 4**. Left: A stylus profilometer. Right: Illustration of the capacitive displacement sensor measuring the contact force between the stylus and the sample surface.*

The feedback parameter for the instrument is a contact force (normally 2 mg or 19.6 µN) between the tip and sample, which is sensed by a capacitive displacement sensor (that is, the contact force is detected via the position of a plate inserted between the two electrodes of the capacitor). A surface profile is obtained by scanning the tip across the surface of a sample while maintaining a selected contact force so that the tip needs to go up and down according to the surface contour. The calibration of the instrument can be verified (or recalibrated if needed) using step height standards (e.g., a NIST-traceable VLSI standard having a mean step height of 970.0 nm and an expanded uncertainty of 5.8 nm at the 95% confidence level).

One of the main applications of stylus profilometer is to evaluate surface finishes of parts via roughness measurement. The profiles shown in **Figure 5** were collected on the smooth and rough portions of a combination wrench and a 14-gauge needle with a scan length of 4.8 mm, a scan speed of 200 µm/s and a sampling rate of 100 Hz (resulting in a data interval of 2 µm). Arithmetic mean roughness Ra (the averaged deviation in height for the measured points against their mean value) is a common parameter to quantify surface finish. In order to calculate Ra, a measured profile is filtered using an industrial standard with the cut-off wavelength of 0.8 mm (under ISO 4287 for profiles obtained using a stylus profilometer), removing waviness and form error from the profile. The first and the last 0.4 mm portions of the profile are excluded from roughness estimation. The remaining 4.0 mm portion of the profile is divided to five 0.8-mm sampling segments, each of which is used to calculate a roughness value. The average of these five roughness values is reported as the final roughness. The roughness estimation was performed using Apex 3D Advanced, a third-party software associated with the instrument. As shown in **Figure 5**, the Ra for the smooth portion of the combination wrench is 0.19 µm, while for the rough portion it increases to 3.80 µm. The Ra for the 14-gauge needle is 0.17 µm.

The limiting factor of stylus profilometer in resolving height and lateral dimensions of a sample is the rather large tip apex, which is 2 µm in radius. This impacts height measurement results for surface features having higher aspect ratios. For example, if there are two 1-µm radius spheres that are 10 µm apart, their height of 2 µm will be correctly measured. However, when they are 0.5 µm apart, the tip cannot touch the substrate between the two spheres, resulting in a measured height that is significantly less than 2 µm. Underestimation of height for narrowly aligned features are more severe when aspect ratios of surface features are greater, which is perhaps the only artifact an operator needs to be aware of (for samples that are not extremely soft).

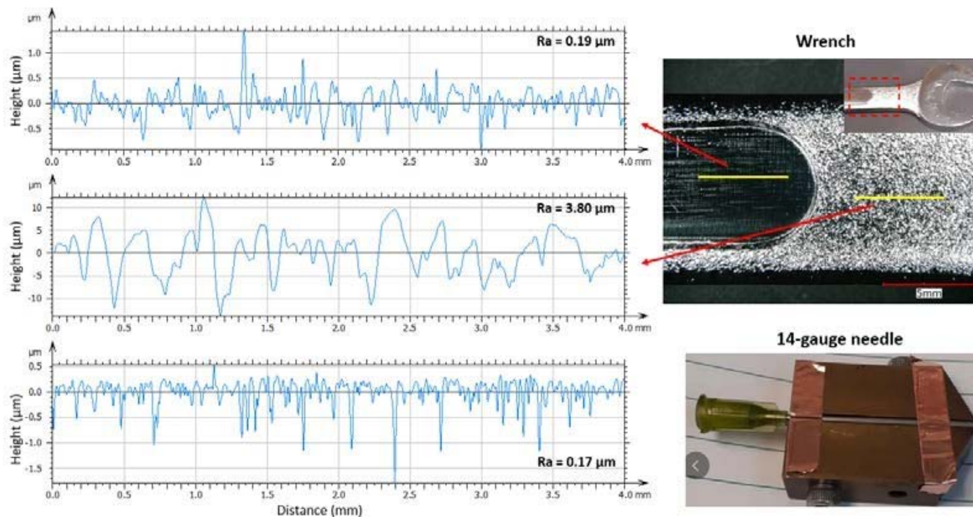

***Figure 5**. Profiles filtered under ISO 4287 with a cut-off wavelength of 0.8 mm for 4.8-mm profiles for Ra measurement.*

By collecting multiple profiles (e.g., 100) one can reconstruct a 3D image for surface morphology. Unlike AFM images, where height is measured at points evenly distributed in both the fast and slow scan directions (e.g., 256 × 256 pixels), stylus profilometer images usually have a much larger number of points in the fast scan direction (e.g., 25000 points) than in the slow scan direction (e.g., 100 lines). This is due to the fact that the scan length is usually rather large and enough data points are needed to evaluate the height distribution, while completing 100 scan lines takes about an hour under a reasonable scan speed. It is thus unrealistic to have as many scan lines as the number of points in each scan line because it will take too much time. Nevertheless, an image having 25000 × 100 pixels makes a decent visualization of the surface morphology for most structures. For example, **Figure 6** shows profilometry images obtained on two areas of a steel part, with one area worn significantly. Values of Ra are indicated in the images, with the worn area being >11 times rougher than the pristine area.

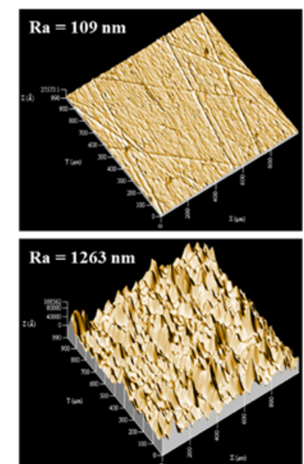

***Figure 6**. Images for a pristine area (upper) and a worn area (lower) of a steel part*

Stylus profilometer is often used to measure the thickness of a film coated on a substrate if a step of the film can be prepared. Measurements of step heights as small as 3 nm have been confirmed. However, it is worth pointing out that for measuring ultra-thin films on a substrate, both a step of the film and a sufficiently smooth substrate are required. The instrument is designed for a flat substrate, requiring careful sample preparation or positioning for irregular samples. This is because the clearance between the sample and the profilometer head is only a couple of millimeters. For smaller samples or for measurement close to the edge of the sample, adding materials (e.g., soft clays or the like) around the sample or its edge is necessary for avoiding tip/sensor crushes.

3. *CLSM for imaging and evaluation of roughness*

CLSM is an optical technique to measure surface morphology. Shown in **Figure 7** are a CLSM (Zeiss LSM 800 for Materials), its working principle and two examples of reconstructed surface morphology. A laser beam scans an area at a specified pixel density (e.g., 512 x 512 pixels) of a sample placed at a pre-set distance from the objective lens. The reflected laser beam from each pixel passes through the objective lens, the splitter and another lens and finally arrives at the plate with a pinhole, which is placed at the front of the detector. Only the laser beams (blue lines in **Figure 7**) reflected from the pixels falling to the fixed focal plane of the objective lens pass through the pinhole and are detected, while those from the pixels deviated (red and green lines) from the focal plane are largely blocked by the plate. This is because the pinhole is placed at a position that is conjugated with the focal plane of the objective lens, which is the reason why the word confocal is coined. By mechanically moving the sample to different distances with a fixed step height (down to 10 nm) in the optical axis (coined as optical sectioning) that cover the height difference of its surface morphology, all pixels are eventually brought to the focal plane. The height for each pixel is calculated by looking for the optical sectioning (i.e., distance from the objective) corresponding to the largest intensity of the reflection of the laser beam, through which the surface morphology is reconstructed.

The 3D images of surface morphology shown in **Figure 7** are for a roughness standard and a corroded metal substrate. The sinewave-like structure is reconstructed, with its roughness estimated (Ra=0.97 µm), which serves as a standard to verify CLSM. The image of a corroded metal substrate shows that the left corner is largely uncorroded and the corroded areas are higher than the uncorroded.

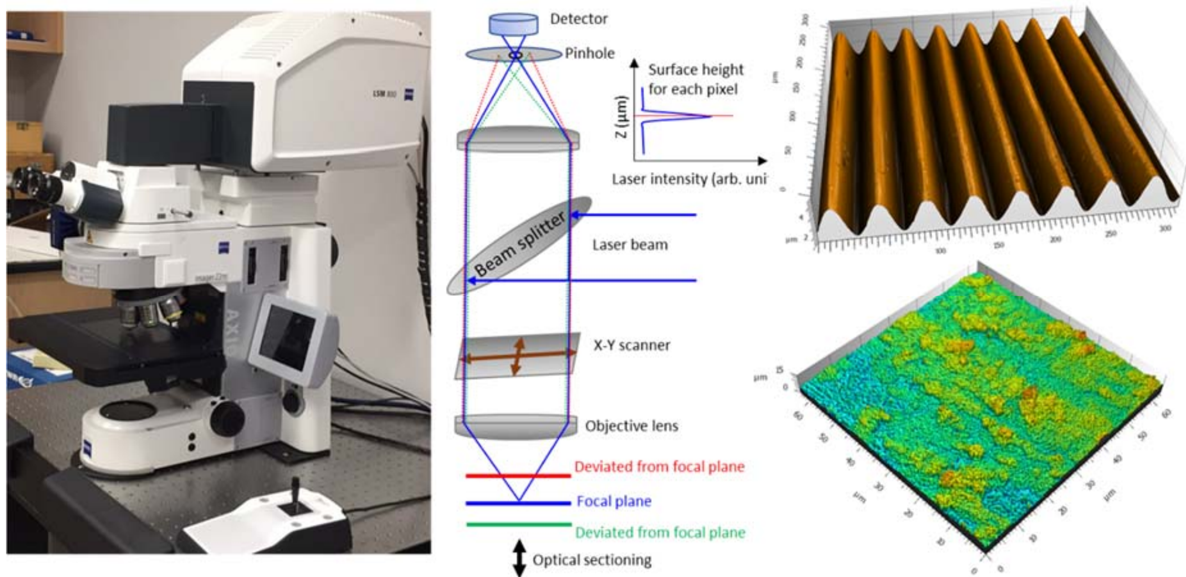

Figure 7*. A CLSM (left), its working principle (centre) and surface morphology of a roughness standard and a corroded metal substrate.*

The size of surface features of the sample and the numerical aperture of the objective lens are the two most important factors to consider when using CLSM. In comparison with stylus profilometer that requires physical contact between the tip and the sample, CLSM is advantageous in measuring (1) fragile surfaces that can be easily altered by scanning of the mechanical probe in a stylus profilometer and (2) samples that a mechanical stylus is hard to reach.

The lateral resolution of CLSM is dependent on the pixel density, the numerical aperture of the objective lens, the wavelength of the laser beam and the pinhole size. Practically, lateral resolutions of 0.2 µm are readily achieved. Therefore, CLSM has a much better lateral resolution than stylus profilometer. The height resolution of CLSM can be as good as several nanometers.

In addition to surface morphology measurement, CLSM can also be used in measuring the thickness of transparent films through determining the separation of the two surfaces, where the intensity of the reflected laser beam is greatly enhanced, assuming that their refractive indices are known. For example, shown in **Figure 8** is the result for a microscope glass slide. The distance separating the two planes having the strongest intensity is 667 µm, which determines the position of the two surfaces of the slide. Because glass has a refractive index of n_{glass} = 1.5, which is different from that of air (n_{air}=1.0), the physical thickness of the glass slide needs to be calculated as 1.5 x 667 µm = 1000.5 µm.

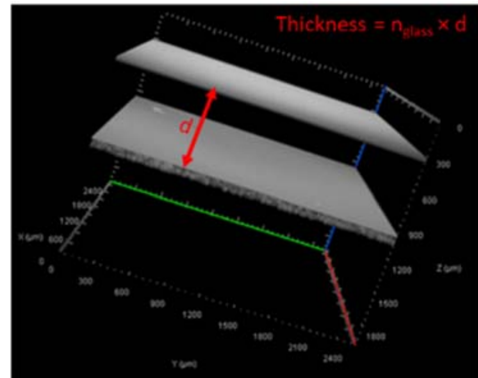

***Figure 8**. Thickness measurement of a glass slide.*

4. Optical CMM for evaluation of roughness and geometry

The three techniques described above are all designed to measure a flat surface, rather than to evaluate the shape of a sample. To also capture the shape of a sample, a CMM is required. Instead of the optical sectioning (i.e., moving the sample in the optical axis of the objective lens) in CLSM, in CMM as illustrated in **Figure 9**, the height of a point of the sample is measured without mechanical movement by taking advantage of chromatic aberration of a white light source. When dispersed via a specially designed objective lens, a white light source is dispersed into its component lights having different wavelengths, each of which renders a specific focal length on the back of the lens. This happens because the refractive index of lens glass is wavelength dependent. As shown in **Figure 9**, a light having a shorter wavelength (e.g., blue light) results in a shallower focal length. If a point of the sample is located at the focal length of a monochromatic light (e.g., green line), its reflection passes through a (confocal) pinhole and is detected by a spectrometer measuring its wavelength, which is related to the height information of the point. CMM is a single point measurement technique, with every point of the sample brought to the measuring position of the optical sensor so as to determine both the shape of the sample and the surface morphology.

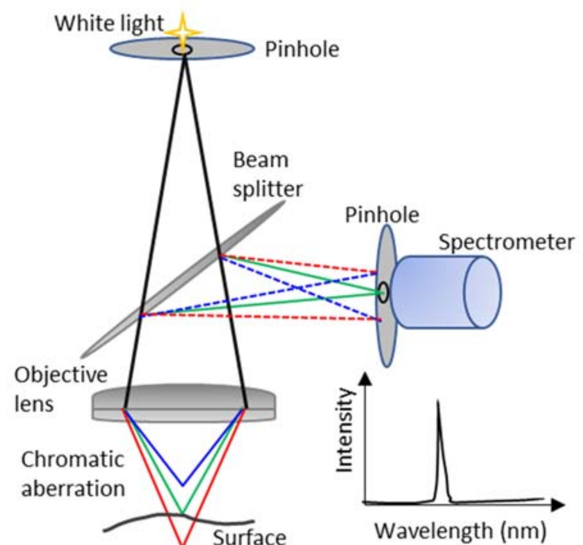

***Figure 9**. Principle of chromatic confocal microscope, making use of the axial chromatic aberration to measure the distance between the objective lens and the sample surface.*

Shown in **Figure 10** are a RedLux optical CMM, the optical sensor making use of axial chromatic aberration (i.e., lenses designed to convert a white light to its monochromatic lights) and a mounted femoral head, as well as a 3D rendering of a cup and a head measured. CMM is a technique capable of measuring surface roughness and mapping the shape of a sample beyond centimeter scales. The measurement time is

normally several minutes, which is extremely quick considering the number of points measured and the fact that both surface morphology and sample shape are measured. When the instrument is installed at Surface Science Western, experiments will be carried out to develop methodologies on exploring surface roughness and geometry of different types of implants and parts.

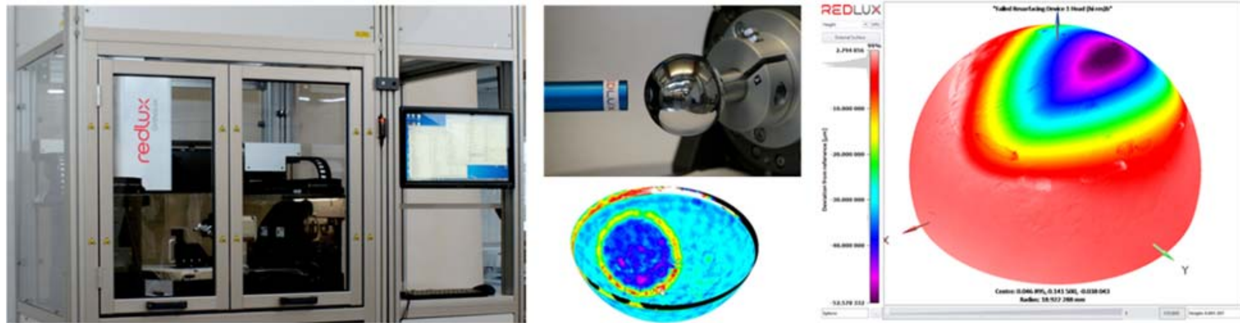

***Figure 10**. A RedLux optical CMM and a mounted hip replacement femoral head, as well as measured head and cap (all from RedLux web site https://redlux.net/).*

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