Title :

Towards a new analytical method adapted to the nanoscale : elastic electron microscopy (MM-EPEM)

Supervisor : Luc BIDEUX
Co-supervisor : Christine ROBERT-GOUMET, Guillaume MONIER
Lab : Institut Pascal, research team « Surfaces et Interfaces »
University : Clermont Auvergne
Email et phone : luc.bideux@uca.fr, +(33) 4 73 40 76 49

Summary :

For several years now, the field of nanotechnology has attracted increasing interest, particularly in the industrial world (electronics, cosmetics, materials, etc.), which expects precise and reliable tools for better control of manufacturing processes and improvement of quality systems. This involves the development of different types of instrumentation already used, through the introduction of new features.

In order to observe and analyze nano-transformed surfaces (nano-pores, nano-wires, nano-drops, nano-particles, etc.), new characterization techniques must be developed. Indeed, at present, imaging techniques, the most commonly used is scanning electron microscopy (SEM), are confronted with important technological locks limiting the resolution of images obtained during the study of objects of nanometric sizes. New techniques have emerged recently to obtain a 3D reconstruction of very small objects such as Serial Block-Face Scanning Electron Microscopy (SBFSEM), Focused Ion Beam Scanning Electron Microscopy (FIBSEM) or Transmission Electron Microscopy Tomography (TEMT) but they are very expensive and lead to the destruction of the analyzed sample.

It is in this context that the "Surfaces and Interfaces" team of the Pascal Institute has developed for several years an electronic spectroscopy technique called Elastic Peak Electron Spectroscopy (EPES) based on the elastic interaction of electrons with matter. After low energy electron bombardment, the surface of a material returns electrons having the same energy as the incident electrons: these are the electrons backscattered elastically. This spectroscopy proved to be a method of characterization very sensitive to the surface and non-destructive. Associated with a Monte-Carlo simulation, developed by the same team, it has taken off in two areas:

- fundamental: very widely used to determine the mean inelastic free path [NIST Electron Inelastic-Mean-Free-Path Database, Standard Reference Database 71, Version 1.1. Nat. Inst.

Standards Technology, Gaithersburg, MD, 2000.] which is a fundamental parameter in all calculations allowing a quantitative analysis of the results obtained in electron spectroscopies (XPS, AES, etc ...).

- applied: for the surface analyzes, the EPES proved to be a tool complementary to the other spectroscopies for the global analysis of a surface and the highlighting of the surface segregations, diffusions during the manufacture of heterostructures, but also during the analysis of nanoporous materials.

Based on this expertise, we propose a thesis topic on the development of a new imaging method based on these electrons backscattered elastically. Indeed, by scanning the surface using a very small electron beam and analyzing the backscattered elastic electrons, it is possible to reconstruct an image of the surface. This new technique called Multi Elastic-Elastic Peak Electron Microscopy (MM-EPEM) will allow precise and fine localization of the different structures present on the surface and, by modulating the primary energy of the electrons, to probe deeply the matter without having of destructive effect. To access quantitative information, the results obtained experimentally will be coupled with Monte-Carlo simulations describing the path of electrons in matter.

This new microscopy technique can be easily installed in ultrahigh vacuum chambers, or Auger Nano-Probe (SAN) systems with a scanning electron gun and electron analyzer for in-situ analysis. non-destructive and in real time. But the main objective of this project would ultimately be to integrate this new technique into a SEM, a reference instrument for the production of surface images, which would considerably broaden its use in the case of nanoscale objects. This new feature would allow users of electron microscopes to superimpose information on surface morphology to depth information over a few nanometers without damaging the surface. The first results obtained by the team represent a significant evolution in the world of microscopy, since current non-destructive techniques are not able to detect such a small amount of material. Much of the work will be devoted to the optimization of the Monte Carlo program (algorithm, computation time, database) and its adaptation to the MM-EPEM technique. This last point is essential since it will automate the search for an optimum solution by combining the experimental and theoretical results.

Partnership :

This project has significant development potential. In 2017, we submitted a maturing project to SATT Grand Center whose first step allowed us to protect the software associated with Monte Carlo simulation. The second stage involved a market study that led to a first contact with a French company (Orsay Physics) developing new analytical tools. His expertise could allow us to develop our technique for applications directly related to the needs of users of instruments such as SEM and nano-SAN probes.
The different locks related to this subject can be raised by the study of test samples. These will be done at the Pascal Institute or provided by Orsay Physics. Measurements will be carried out in the ultrafide team of the "Surfaces and Interfaces" team equipped with a scanning electron gun and a hemispherical electron analyzer. In addition, studies on an instrument not present on the Clermont place will be carried out in collaboration with the CEA / LETI laboratory of Grenoble or with the Lavoisier Institute of Versailles, using a nanoprobe Auger SAN whose lateral resolution of electron gun (≤50 nm) will achieve a finer resolution, important point for the analysis of nanomaterials. In addition, the MEB microscope of the 2MaTech engineering and expertise company present on the Clermont Ferrand campus can serve as a basis for the integration of the MM-EPEM technique.

Keywords :
- Vacuum techniques
- Surface analysis by:
• XPS, AES, EPES and UPS electronic spectroscopies.
• LEED slow electron diffraction
- Development of model surfaces under ultra-vacuum
- Elastic Imaging, Scanning Electron Microscopy and Nanosonde Auger
- Monte-Carlo simulations for the study of the path of electrons in matter