ECOLE DOCTORALE DES SCIENCES POUR L'INGENIEUR

Title of the thesis:

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Summary :

For several years, the field of nanotechnology has been attracting increasing interest, particularly in the industrial world (electronics, cosmetics, materials, etc.), which requires precise and reliable tools for better control of manufacturing processes and improvement of system quality. Nanotechnology involves developing different types of facility already used, through the implementation of new features.
In order to observe and analyze nano-transformed surfaces (nano-pores, nano-wires, nano-dots, nano-particles, etc.), new characterization techniques must be developed. Indeed, nowadays, imaging techniques, of which the most commonly used is scanning electron microscopy (SEM), are confronted with crucial technological 'road-blocks', which limit the resolution of the images obtained during the study of nano-metric dimensions of objects. New techniques have emerged recently to obtain 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.

In this context, the "Surfaces and Interfaces" team of the Pascal Institute has developed over a number of years an electronic spectroscopy technique known as Elastic Peak Electron Spectroscopy (EPES) based on the elastic interaction of electrons with matter. Consequently to a low energy electron bombardment, elastically electrons are backscattered by the surface of the material (i.e. electrons having the same energy as the incident electrons). This spectroscopy has proven to be a method of characterization which is very sensitive to the surface and non-destructive. Associated with a Monte-Carlo simulation, developed by the same team, it has advanced spectacularly in two areas:
- fundamental aspect: it is very widely used to determine the inelastic mean free path [NIST Electron Inelastic-Mean-Free-Path Database, Standard Reference Database 71, Version 1.1. Nat. Inst. Standards Technology, Gaithersburg, MD, 2000.] This is a fundamental parameter in all calculations allowing a quantitative analysis of the results obtained with electron spectroscopies (XPS, AES, etc ...).
- applied aspect: for the surface analyzes, the EPES proved to be a complementary tool to the other spectroscopies for the overall analysis of a surface and the demonstration of

surface segregations, diffusion during the manufacture of hetero-structures 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 backscattered elastically electrons. Indeed, by scanning the surface using a very small electron beam and an electron analyzer, it is possible to reconstruct an image of the surface. This new technique called Multi-Mode -Elastic Peak Electron Microscopy (MM-EPEM) will allow specify and refine localization of the different nano-structures present on the surface and, by modulating the primary energy of the electrons, to in depth probe the material deeply without having any destructive effect. To gain access to quantitative information, the results obtained experimentally will be coupled with Monte-Carlo simulations describing the path of electrons in the matter.

This new microscopy technique can be easily installed in ultrahigh vacuum systems, or Scanning Auger Nano-Probe (SAN) systems equipped with a scanning electron gun and an electron analyzer for in-situ non-destructive and real time analysis. However, the main objective of this project would ultimately be to incorporate this new technique into a SEM, the standard instrument for surface imaging, which should considerably expand 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 advance in the world of microscopy, since current non-destructive techniques are not able to detect such a small amount of matter.

Partnership :

This project has significant development potential. In 2017, we submitted a maturing project to SATT (Grand Center Region) 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 Franco-Czech company developing new analytical tools. Their expertise should allow us to develop our technique for applications directly related to the needs of users of instruments such as SEM and nano-SAN probes.

In order to proceed with this development, we plan to carry out studies on an instrument not present on the Clermont site. In collaboration with CEA Grenoble, analyses will be carried out in a SAN. One of the advantages of this equipment is the lateral resolution of the electron gun (≈50 nm) that will achieve a better resolution, important point for the analysis of nanomaterials. The results of elastic microscopy associated with those obtained by SAN will show the potential of these two techniques for the study of nanometric objects.

As we would like to eventually implement this new technology in a SEM, we also plan to

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carry out tests within our partner 2Matech Clermont, an engineering and expertise company mainly in the fields of advanced mechanics and characterization of materials.

Keywords:

- Ultra High Vacuum Techniques.
- Surface analysis : Electronic spectroscopies XPS, AES and UPS and LEED
- Surface elaboration in UHV conditions
- Elastic imagery and scanning electron microscopy
- Monte-Carlo simulations to study the path of the electrons inside the matter.

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