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NFFA

Nanoscience Foundries and Fine Analysis

D3.4

Design Study of user-oriented Advanced Analysis and Metrology Facilities

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Deliverable D3.4: Design Study of user-oriented Advanced Analysis and Metrology Facilities

1. INTRODUCTION

1.1. Purpose of the document

The purpose of this document is to describe the concept of Advanced Analysis and Metrology facilities for NFFA centres for characterisation of various material properties in synergy with the other NFFA facilities and with Large Scale Facilities (LSFs). A possible technical layout is proposed. In addition, the document analyses the idea of a *common* metrology, to ensure reliability, traceability and, whenever possible, reproducibility of results. In particular, the current situation on standardisation at the nanoscale is reviewed and new suggestions for the development of a NFFA *internal* standardisation are given.

1.2. Application Area

The targets of this document are the members of the NFFA Project, the EC Project Officers, and the general public.

1.3. Sources

Description of Work (DoW). See at web site:

<http://www.nffa.eu/ResearchActivityData.aspx?IdRACT=14&idTypeRACT=1>

The final document is the result of several discussions among NFFA partners and with external experts in the different techniques, from both research and tool maker companies. Scientists specialised in a wide range of distinct fields have been contacted and their needs have been discussed.

To gather information on the present situation and future perspectives concerning standardisation at the nanoscale, members of national and international standard organisations have been personally reached. Furthermore, three of the authors of this text attended the Co-Nanomet Workshop, held in November 2009 at PTB.

1.3.1. Objective of Work Package 3

The Objective of WP3 is the design study of NFFA-RI centres, the technical layout of instrumentation and tools.

1.3.2. Description of work broken down into tasks

The following tasks are defined in WP3:

- T3.1) Design study of the overall infrastructure
- T3.2) Design study of a nanolithography station within the facility
- T3.3) Design study of user-oriented material growth facilities
- T3.4) Design study of user-oriented metrology facilities
- T3.5) Design study of a molecule and nano-particle manipulation lab
- T3.6) Design study of nano-bio labs
- T3.7) Assessment of the possible contribution of existing facilities that could be integrated in NFFA-RI

2. EXECUTIVE SUMMARY

One of the major bottlenecks for the development of new nanoscale science and applications is the limited reproducibility of both sample fabrication protocols, and experimental results. NFFA will try to fill in this gap by putting a strong effort into the development of Advanced Analysis and Metrology facilities offering a clear and full traceability of results, thus potentially opening new perspectives towards the definition of new certification criteria.

A precise and unequivocal sample definition before accessing Large Scale Facilities (LSFs) will strongly impact their outcome, optimising the use of beamtime. To this extent, the Advanced Analysis and Metrology facility of each NFFA centre will have therefore a key role in joint nanoscale synthesis/fabrication and fine analysis experiments, providing the consistency of the results of all facilities.

Besides a strong interaction with the other NFFA facilities for characterisation of a wide range of material properties, the Advanced Analysis and Metrology facilities will be centres for high-level science in advanced analysis experiments, offering access to state of the art tools that are typically only available in few laboratories.

The instrumentation inside the facility will be divided into a *basic* tool set, *common* to all centres, and an *advanced* tool set to be diversified among the centres according to the scientific specialisation of the single location, linked to the other NFFA facilities, the nearby LSF, and the environment. Part of the in-house activity of the metrology facility will be dedicated to the development of new characterisation and metrological tools at the nanoscale. A “Total Characterisation Line” will be developed as a fully automated system providing complementary information for the sample definition standard that will be well connected to the data repository. When operative, the new system will be offered as a fast means for characterisation of basic and well-defined properties, complementary to the standard, human-controlled activity that will be entirely dedicated to address more specific and/or more challenging scientific questions.

In the general effort towards a definition of a common metrology concept, NFFA will adopt an “internal” metrology at the nanoscale, by identifying internal reference materials and protocols and by performing periodically cross-check experiments in all centres for all relevant instrumentations in the *basic* tool set. NFFA Advanced Analysis and Metrology facilities will operate in compliance with the available standards and regulations whenever relevant to the goals of the infrastructure, and will propose new internal protocols and reference materials whenever official documents are missing or are not appropriate. In collaboration with LSF scientists, common protocols will be defined and applied also for the characterisation of LSF beamlines. A well-established use of the internal metrology will ensure transferability of the results within the infrastructure, thus making really feasible subsequent steps of a complex experiment at different centres or at different times in the same centre.

To optimise use and management of the Advanced Analysis and Metrology facility, it is advisable to create sections and, when deemed appropriate, sub-sections. A specific section is designed to be located at the partner LSF, with NFFA staff personnel working day by day in collaboration with LSF scientists. Task of such NFFA personnel will be the support of NFFA users as well as the development of new tools. A first suggestion for the overall structure of the facility could be: Microscopy (Electron Microscopy, Scanning Probe Microscopy, Emerging Microscopes); Structural, Optical & Elemental Analysis (X-Ray Laboratory, Optics Laboratory, Surface Analysis, Composition Analysis); Magnetic Characterisation; Thermal & Mechanical Characterisation; Transport Properties, LSF Located Section.

3. MOTIVATION

The limited reproducibility of both sample fabrication protocols and experimental results when the same experiment is performed in different sites is presently a bottleneck for the development of new nanoscale science and applications. NFFA centres will address this problem by providing a common well-defined metrology. In this context, the main aim of NFFA Advanced Analysis and Metrology facilities will not be to provide a recognised certification but rather a traceable characterisation activity. To the purpose, a facility has to offer a common set of continuously calibrated instrumentation, to ensure constant quality of the results, and common test measurements have to be developed for direct comparison of results obtained in the different NFFA centres, as thoroughly analysed in Section 6 of this document. In this view, NFFA internal protocols will have the capacity of contributing to the definition of new standards for nanoscience. Metrology at the nano-scale itself will be a core research activity, devoted to the development of new instrumentation and new internal calibration/standardization protocols, potentially providing the basis of new certification criteria. With its calibrated instrumentation and the know-how in standardisation issues, Advanced Analysis and Metrology facilities will provide the consistency of all results of the other facilities in each NFFA centre and periodically check their operating conditions.

Besides metrological tasks, the facility will be a centre for advanced analysis, with a “dedicated” scientific activity, offering access to state of the art tools that are typically only available in few laboratories, thus fostering high-level science.

3.1 Potential for an Advanced Analysis and Metrology facility in the context of fine analysis at LSFs

Both NFFA centres and LSFs will strongly benefit from a close link between Advanced Analysis and Metrology facilities and LSFs.

From the metrological point of view, the Advanced Analysis and Metrology facilities will offer their calibrated instrumentation and collaborate with LSF scientists in the development of well-defined procedures for the characterisation of beamline performance, in the general NFFA effort towards full traceability of experimental results.

On the scientific side, LSFs will benefit from the proximity of Advanced Analysis and Metrology facilities opening the possibility of joint proposals for complementary studies. Microscopy, for example, turned often out to be fundamental in conjunction with synchrotron based techniques for significant advances in nanoscience, as recently demonstrated by a nice HRTEM/synchrotron XRD and GISAXS investigation of shape changes of supported nanoparticles during reduction/oxidation cycles [1]. To enhance this synergy, in the layout of an NFFA metrology facility a significant role will be given to microscopy techniques.

A more direct advantage for LSFs will result from the use of the facility as a centre for a first characterisation of samples before fine analysis experiments at LSFs. This step would become crucial to avoid wasting time during beamtime with a sample that is not what it should be.

Advanced Analysis and Metrology facilities will also offer the possibility for characterisation of the properties of nano-objects produced at LSFs (see paragraph 3.4 in D3.2). This interaction will give a unique chance for technology transfer in pre-competitive research: potential industrial users will be able to fabricate new materials or devices with the cutting-edge precision of a LSF and to immediately characterise (and eventually maybe also certify) the properties of the obtained prototype within the same proposal, thus speeding up the overall process.

Finally, another possible synergy, to be discussed and agreed with the specific LSF partners, involves the use of LSF side instrumentation that does not require beamtime allocation for the preliminary characterisation activity offered by the Advanced Analysis and Metrology facility (see Section 4.1d of this document), in an LSF-located section with dedicated staff, with combined competences. The

collaboration will extend to the development of new tools by combining typical lab characterisation techniques with X-ray/neutron/laser beams.

3.2 Scientific needs

NFFA centres will provide access to specialised tools capable of designing, producing and characterising new generations of nanotechnology products, thus fostering new research activities leading to nanoscience advancements. In this context, NFFA Advanced Analysis and Metrology facilities have to offer a combination of well-established, state of the art tools and emerging tools to ensure reliable characterisation and advanced science for all the four generations of nanotechnology products recently foreseen by the “Nanosciences, Nanotechnologies, Materials and Production Technologies” (NMP) experts as the future foci of nano research in Europe, namely passive nanosystems, active nanosystems, systems of nanosystems and molecular nanosystems [2]. As suggested by the NMP experts, this requires an effort towards the implementation of accurate and precise three-dimensional characterisation tools, and the analysis has to move from static measurements of quenched samples to dynamic, real-time measurements, to monitor kinetic processes. It’s also fundamental to offer tools capable of functioning in different environments, such as vacuum, air, high pressures, liquids or *in-vivo*. All the above are also the directions LSFs are moving towards: Free Electron Lasers are becoming operative for the investigation of ultra-fast processes, synchrotron based 3D techniques are being implemented as well as well as beamlines, both at synchrotron and neutron facilities, where experiments can be performed under high pressure environment (from ~5-10 mbar for X-Ray Spectroscopy [3], to 10 GPa for neutron diffraction [4], up to more than 100 GPa for XRD in diamond anvil cell [5], just to give a few examples) or specialised in the handling of biological samples. Particular attention has to be paid to hybrid interfaces and to issues related to toxicology and nanomedicine, answering the need to identify and define geometrical properties of nano-particles or other nano-structured systems (see also D3.6).

3.3 Relevant technical criteria

An Advanced Analysis and Metrology facility has to be highly flexible to face extremely different requests from the users. Such an extended target makes it difficult if not impossible to have the most advanced characterisation techniques in all centres. For this reason it is necessary to define a *basic* tool set – intended both as very typical and easily accessible techniques (e.g. profilometry, XRD, optical techniques) and as basic level instrumentation for more complex techniques (e.g. EM, SPM). The basic tool set should be always accessible, flexible in response to very different needs and *common* to all the NFFA centres. In addition, an *advanced* tool set will be part of each NFFA centre. Due to the specificity of advanced instrumentation, the advanced tool set will be much less flexible and/or highly costly, and it will not be possible to duplicate the same instrumentation in all centres, instead it should fit the scientific/technological specialisation of each centre.

It is extremely important to define a common standard for resolution and accuracy of each tool, at least for the basic tool set, to allow direct comparison of measurements carried out in different NFFA centres. In this context, a set of standard measurements and protocols to be accurately performed before every experiment has to be determined. Standard measurements could include test samples, common to all NFFA centres. More details are given in section 6 in this document. Particular attention will be paid to the possibility of contributing to the definition of new official standards, to be established in collaboration with International Metrology Offices (see also D4.8).

In the choice of the instrumentation offered to users, speed of response will be relevant: for preliminary characterisation activities, to be done with the basic tool set, a fast response is necessary, which requires the use of robust instrumentation and the presence of technical personnel, to ensure the maintenance of the instrumentation and to support inexperienced users. However, the actually achievable speed depends on the technique, as reviewed at the end of this document. Slow response is possible and sometimes unavoidable for advanced analysis, which will be mainly carried out as part of

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the ‘dedicated’ scientific activity of the facilities and require mid- or long term proposals for ensuring a successful result.

3.4 Access

Both serviced (“hands off”) and self-serviced (“hands on”) access will be allowed, as described in D4.4, according to the complexity of procedure/instrumentation and to the experience of the users. For very simple and frequently used instrumentation (e.g. basic optical techniques, SEM, AFM in air) “open hands on” should be allowed for all users after a short training. For standard but more sophisticated and critical instrumentation (e.g. simple STM) direct “limited hands on” will be given to specialised and experienced users only. In all other cases (inexperienced users for standard but sensitive instrumentation and all users for advanced instrumentation) access will be provided through a staff member. In case of basic instrumentation, the technical personnel might be the most adequate interface with inexperienced users. For advanced instrumentation, researchers are needed. Training will be always necessary for direct access. The duration of the training should depend on the instrumentation.

3.5 In-house research

In-house research will be an important part of the activity of the facility. Scientists working in the Advanced Analysis and Metrology facility must be allowed to use the instrumentation to carry out their scientific projects. This will keep NFFA scientists at the top-level and will bring new ideas for future new applications and developments of the instrumentation. In addition to scientific projects, it would be important and useful, in fact, to carry out also methodological research. New developments, after implementation and testing as part of in-house research activity, will be made available to users, thus establishing a more timely transfer from advanced research and development to users. This will represent a new, outstanding way to keep the offer of NFFA centres at the state of the art level. When a new technique or a new application of an existing technique is successfully tested in one of the NFFA centres, there should be a quick knowledge transfer to the other centres and training of other NFFA staff members, in order to give the possibility to offer the same tool in more centres.

4. TECHNICAL SPECIFICATIONS

An Advanced Analysis and Metrology facility has to offer a wide range of techniques for characterisation of complementary properties. For the organisation and management of such a diversified structure, it is useful to divide it into sections: Microscopy, Structural Optical and Compositional Analysis, Magnetic Characterisation, Thermal and Mechanical Characterisation, and Transport Properties. The sections can in some cases overlap, sharing instrumentation.

In the following, a suggested structure for the above sections of the Advanced Analysis and Metrology facility is presented. Each technique included in this structure is briefly reviewed in Appendix A with a rationale on its role in a NFFA facility. An analysis of the distribution and operation of the instrumentation in NFFA centres will be presented in Sections 5 and 8 of this document, together with a cost estimate. It has to be noted that not all the techniques discussed below provide information on sample properties at the nanoscale (see Figure 1). Nevertheless, these techniques allow complementary measurements, necessary for a

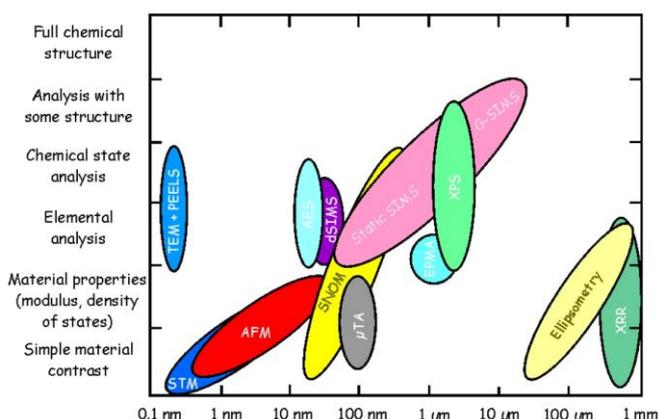


Fig. 1: Some typical characterisation techniques giving chemical information, plotted vs. typical measurement spot sizes [Source: National Physical Laboratory (UK)]

comprehensive understanding of the sample properties, e.g. chemical properties of nanostructured materials.

4.1 Microscopy Section

Microscopy should be one of the main resources for an Advanced Analysis and Metrology facility. The Microscopy section has not to be intended only for morphological characterisation of nanostructures but all most advanced applications should be allowed both as advanced offer to external users and as part of in-house activity. Suggested sub-sections (and included tools) are:

4.1a Electron Microscopy

- *Scanning electron microscopy (SEM) with Tools for Compositional and Crystallographic Analysis*
- *Transmission Electron Microscopy (TEM) and Scanning Transmission Electron Microscopy (STEM)*
- *Sample Preparation Lab equipped with standard tools, and in particular:*
 - *Focused Ion Beam (FIB) Scanning Electron Microscope with a Dual Beam source*
 - *Microtome*
- *Three-dimensional Atom Probe (3DAP)*

4.1b Scanning Probe Microscopy

- *Scanning Tunneling Microscopy (STM)*
- *Atomic Force Microscopy (AFM)*
- *AFM related techniques (LFM, EFM, SKPFM, MFM)*
- *Scanning Near Field Microscopy (SNOM)*

4.1c Emerging Microscopes

NFFA will also be an access point for emerging microscopy techniques. Some of these techniques have been introduced in the scientific panorama only recently, others are still under development but all of them will potentially be of great impact for nanoscience in mid- or long term and their presence in the NFFA centres is therefore crucial to constantly provide a technical offer at the state of the art level. At the start-up of the centres, these techniques will be limited to an advanced analysis activity. The initial equipment could include:

- *He-ion Microscopy (HeIM)*
- *Tip-Enhanced Raman Spectro-microscopy (TERS)*
- *Brillouin Microscopy*

4.2 Structural, Optical and Elemental analysis

In nanotechnology, knowledge of the structure and composition of the materials studied is a key requirement for understanding material properties on a nanometre scale. Suggested sub-sections (and included tools) are:

4.2a X-Ray Laboratory

- *X-Ray Diffraction (XRD)*
- *Small-angle X-ray Scattering (SAXS) and Grazing Incidence X-ray Scattering (GISAXS)*

4.2b Optics Laboratory

- *Ellipsometry and Spectroscopic Ellipsometry (SE)*
- *Dynamic Light Scattering (DLS)*
- *Profilometry*
- *Fluorescence Spectroscopy*
- *Fourier Transform Infrared Spectroscopy (FTIR)*

- *μ-Raman Spectroscopy*
- *Ultraviolet and visible absorption spectroscopy (UV-Vis)*
- *Photo- and Electro-luminescence*

In addition, the facility will provide state-of-the-art tools for Ultra-Fast Laser Spectroscopy (see Appendix A):

- *Ultra-Fast Transient Absorption (UFTA)*
- *Time-Correlated Single Photon Counting (TCSPC)*
- *Time-Resolved Photo-Electron Spectroscopy (TRPES)*

4.2c Surface Analysis

- *Low Energy Electron Microscopy (LEEM) / Photoemission Electron Microscopy (PEEM)*
- *X-Ray Photoelectron Spectroscopy (XPS)*
- *Auger Electron Spectroscopy (AES)*

4.2d Composition Analysis

- *Secondary Ion Mass Spectrometry (SIMS)*
- *Solid State Nuclear Magnetic Resonance (NMR)*
- *Gas Chromatography (GC) and Microreactors*

4.3 Magnetic characterisation

- *Magneto-optic Kerr effect magnetometry (MOKE)*
- *Superconducting quantum interference design (SQUID)*
- *Vibrating sample magnetometer (VSM)*
- *Ac-susceptibility*

4.4 Thermal and Mechanical characterisation

- *Thermogravimetry (TG)*
- *Differential scanning calorimetry: Nano (DSC)*
- *Nanoindentation*
- *Acoustic measurements of elastic constants (Pulse-echo technique)*

4.5 Transport properties

- *Transport properties vs. temperature*
- *Transport properties in magnetic field*
- *Single molecule transport (with instrumentation of the Scanning Probe Microscopy sub-section)*

4.6 LSF Located Section

A section of the Advanced Analysis and Metrology facility will be located inside the neighbouring LSF. NFFA staff personnel will work on specific beamlines together with LSF scientists as a bridge between NFFA and LSF competences. The collaboration will extend to the development of new instrumentation combining typical LSF techniques with classic lab characterisation tools. First steps in this direction are already leading to the development, for example, of coupled scanning probe/synchrotron instruments for the investigation of topographic and spectroscopic [6] as well as mechanical and structural [7] properties at the same time at the nanoscale.

Additionally, LSF beamlines often offer side-instrumentation working (only or also) with conventional sources that might be partially accessed by NFFA users under the supervision/assistance of the LSF located NFFA staff, upon specific agreements with the LSF. These tools, as well as the new

instrumentation to be developed, will be evaluated case by case after the final choice on the partner LSFs.

The LSF Located Section, when activated, will represent a new, visible, example of direct synergy between nanoscience centres and LSF.

5. REQUIRED DISTRIBUTION OF FACILITIES

5.1 Distribution of the instrumentation among the NFFA-Centres – basic and advanced characterisation

In the choice of the specific instrumentation in the basic tool set, several characteristics have to be taken into consideration: instruments should have generally medium/high flexibility, being not too fragile, and providing fast/medium times for measuring material properties in an as much reproducible as possible way. For these techniques it is therefore also important to define common internal procedures and standard measurements to ensure the reproducibility of experiments in different NFFA centres (see Section 6).

The choice of the instrumentation for the advanced tool set should conversely take into account not only technical criteria but also the advantage of offering to general users the possibility of accessing specialised and/or state of the art characterisation tools, therefore including high-performance but high-cost instrumentation. The use of advanced instrumentation might require the allocation of a longer time period for each single experiment, due to the complexity of the tools. This should not be considered as a handicap for their inclusion in NFFA centres, but rather they should be related to mid- and long-term proposals. Such kind of proposals could also foster active collaborations between the scientific staff of the facility and external users on joint specific scientific projects to be carried out as part of in-house research.

For the distribution of the advanced tool set, the characteristics of the other facilities of the same NFFA centre and of the nearby LSF have to be taken into account in order to optimise the scientific overlap and the chances for high-impact common proposals. It is therefore difficult to establish now a precise scheme, which requires knowing exactly which LSFs will contribute to NFFA, but some general considerations can be made. In an NFFA centre specialised in nano-bio, for example, it will be useful to expand the capabilities of the metrology facility in this direction, including among the advanced instrumentation more tools for bio-related studies, including biophysics. In a NFFA centre particularly focused on energy, specially built instruments for application in liquid cells, e.g. for *in-situ* studies of electrodes in fuel cells, should also be taken into consideration, as well as instruments for application in high pressure environment in connection with LSF offering HP versions of FA tools. The possibility of specialising the Advanced Analysis and Metrology facility of a NFFA centre in research at extremely low temperature and/or in high magnetic fields could also be considered where this meets the scientific target of a specific location.

A suggestion on the choice and the distribution of basic and advanced tools for NFFA metrology facilities is given in Table 1. Due to the present uncertainty on the partner LSFs, and therefore the local specialization and competences, tools suggested as part of the advanced tool set have to be considered as possible options based on the considerations above. Their actual presence will be decided at the start-up of the infrastructure.

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Table 1: Distribution of tools for Advanced Analysis and Metrology Facilities

(Sub)-Sections		Basic Tool Set	Advanced Tool Set
Microscopy Section	SPM	RT STM in UHV VT STM in UHV AFM* in air – large sample AFM* in air – High res AFM* in UHV	LT-STM, SP-STM HP-STM ECSTM aperture SNOM Cryo-SNOM
	EM	SEM** TEM/STEM***	SEM# TEM Cs corrected 3DAP
	Emerging Microscopes		HeIM TERS Brillouin Microscope
Structural, Optical & Elemental Analysis	X-Ray Lab	XRD (4-circle and powder) SAXS/GISAXS (basic)	specialised X-ray set-ups (to be specified in a later stage) WAXS/SAXS/GISAXS
	Optics Lab	Ellipsometry/SE DLS Profilometry FTIR μ -Raman Fluorescence UV-Vis EL PL TCSPC	 UFTA TRPES
	Surface Analysis	LEEM/PEEM XPS+AES	AC LEEM/PEEM HP-XPS, XPS in liquids
	Composition Analysis		NMR SIMS GC Microreactors
Magnetic Characterisation	basic MOKE SQUID VSM	MOKE Ac-susceptibility	
Thermal & Mechanical Characterisation	TG DSC	Nanoindentation Acoustic meas.	
Transport Properties	Transport vs. Temperature Set-up Transport in Magnetic Field Set-up	LT setup for opto-electronic properties	

* equipped for LFM, EFM, SThM, SKPFM, MFM and with a liquid cell

** equipped with EDS

*** equipped with EDS, EELS, HAADF

equipped with EDS, WDS, EBSD

5.2 Synergies with other NFFA-facilities in each NFFA-Centre

The Advanced Analysis and Metrology facility will be the fulcrum of the effort towards standardisation and comparability of experimental results that will be one of the main goals of the overall NFFA infrastructure. In this context, its calibrated instrumentation will ensure the consistency of the results of the other facilities and will be used to periodically check the operating conditions of all NFFA apparatuses.

Besides metrological issues, the synergy with other NFFA facilities will extend to a direct collaboration in joint proposals. On the one hand, the Advanced Analysis and Metrology facility, through its basic tool set, will provide a *fast* characterisation as an *intermediate* step between separate fabrication and preparation procedures, and before fine analysis at LSFs. In order to facilitate the synergy, part of the instrumentation of the facility might be located inside the cleanroom, to ensure close proximity, speed and controlled environment for sample transfer, and to avoid contamination (see scheme in Fig. 2). On the other hand, scientific projects mainly focussed on *advanced analysis* within the

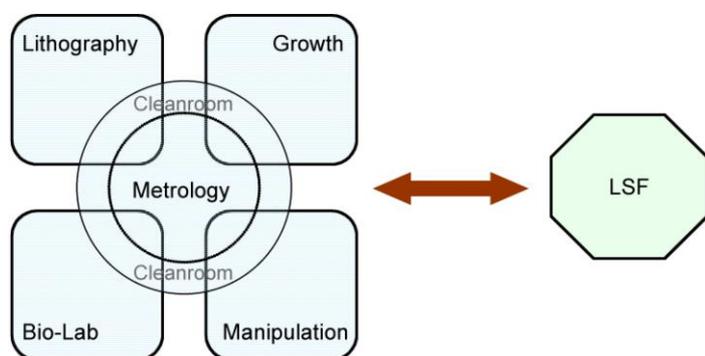


Fig 2: Scheme of the synergies between Metrology Facility and other facilities in an NFFA centre and the nearby LSF

facility, to be performed also with the advanced tool set, may involve nanosystems synthesised with the help of other NFFA facilities. Analogously, projects aiming at the fabrication of nano-objects in other facilities may be complemented by a thorough analysis of their properties within the Advanced Analysis and Metrology facility.

Finally, a third field of synergy might stem from the development of new tools: new techniques initially developed as part of the activity of other NFFA facilities might, in fact, at a later stage extend their applications to a more general purpose and contribute to the activity of the Advanced

Analysis and Metrology facility, as, for instance, Photonic Force Microscopy (see D3.5).

5.3 Synergy with LSFs

Besides providing a preliminary characterisation for joint NF and FA projects, the activity of the Advanced Analysis and Metrology facility will also support and complement experiments conducted at LSFs and give the possibility of advanced characterisation for nano-objects fabricated at LSFs (see Section 3.4 in D3.2). In this context, proximity plays a fundamental role, speeding up the overall experiment. Furthermore, as described in Section 4.6 of this document, a section of the facility might be located inside the partner LSF, with NFFA staff personnel working day by day in collaboration with LSF scientists. Task of such NFFA personnel will be the support of NFFA users as well as the development of new tools. The continuous interaction with, on the one hand, the nanoscience and nanotechnology communities, often unaware of the advantages of accessing fine analysis at synchrotron/ neutron/laser sources, and, on the other hand, the LSF staff will give to the LSF located NFFA personnel a key role to enhance the use of LSFs, spreading the potential of Fine Analysis experiments within the academic/technological communities and developing new application areas for LSF, thus attracting new users.

5.4 Required infrastructure, staff, and environment

The Advanced Analysis and Metrology facility will need dedicated space well connected to the other NFFA facilities. This includes sharing the clean environment for relevant measures.

To speed up the overall process in case of multi-technique characterisation experiments with

frequently used tools requiring UHV environment, an UHV system with multiple chambers interconnected by transfer lines and separated by valves is recommended. Particular attention has to be paid to technical solutions for electromagnetic and vibration isolation, especially when simple scanning probe instruments are included. A possible layout for such UHV system includes: XRD, PEEM/LEEM, XPS, AES, RT-STM/AFM. Other more complicated instrumentation, often requiring particular working conditions and longer experimental time, will be host in separate UHV chambers, each one equipped with standard sample preparation tools.

In order to ensure reliability and speed in operation of the basic tool set, a suitable amount of specialised technical personnel in addition to scientific personnel should be hired. In particular, technical personnel will be responsible for the maintenance and operation of the instrumentation available for direct access, under the supervision of a scientific staff member who will be in charge of discussing with the user the best experimental solution for the specific scientific project. Scientific personnel will be in charge of the advanced instrumentation, both for user-oriented activity and for in-house scientific and methodological research.

An overview of required staff and environment is summarised in Section 8 together with a cost estimate.

6. COMMON METROLOGY

When matter is approached at the nanoscale, different physical phenomena come into play, and traditional materials can show completely new properties and functionalities depending on the specific size of the particle or object. Besides advanced experimental tools that are developed in research laboratories with the main goal of discovering new properties of matter, it has become more and more important to have reliable quantitative measurements of materials properties at the nanoscale to evaluate different particle configurations, compositions, etc. Mechanical properties like friction have been addressed with scanning probes creating the field of nano-tribology with relevant applications to industrial products. Micro and nano-fluidics are also born out of nanotechnology and of reliable probes that can be successfully employed to compare directly objects nanostructured with different geometries, made of different materials, assembled in different ways.

The birth and development of nanoscience is entirely due to the capability of measuring in real space with atomic resolution. This has generated nanotechnology along with a great development of novel as well as empowered systems and methods for high spatial resolution. A new generation of instruments have been developed that have moved, over the last decade, from the advanced research laboratory to a wide spread application in many multidisciplinary domains of science and technology, specially scanning real-space probes and few-molecule probes.

One key aspect of NFFA is to create a common technological platform for advanced nanofabrication and characterisation of nanostructures, with a well-defined metrology. Standardisation is the frontier to be reached for an upscale of nanodevice production and quality control.

To date, the most relevant activity for standardisation for nanotechnology is carried out by the International Standards Organisation (ISO), which in June 2005 has formally established the ISO-TC229-Nanotechnology (not including electrical/electronics field) and the International Electrotechnical Commission (IEC), which in June 2006 has established the IEC-TC-113-Nanotechnology for electrical and electronic products and systems, for the development of technical standards in these specific fields. There are currently more than 30 standards documents under development, but it will take some time before the related protocols will be delivered.

It is clear that, although a number of International and National standards bodies are presently engaged in a considerable effort in nanotechnology standardisation (a list is shown in Appendix B), normative and regulations in matter of standards for nanotechnology are not yet thoroughly addressed. Standards are generally developed with the maturity of a technology while nanotechnology is still in early stages of development, which means that established standards for the macroscopic and

microscopic world cannot be easily scaled down to the nanoscopic world. As addressed by the scope of ISO TC229, it is crucial:

- to understand and control processes, as a rule, on the nanometer scale (including well below 100 nm) in one or more measurements;
- to use the properties of objects and materials on the nanometer scale to produce more perfect materials, instruments and systems which realise these new properties.

Therefore nanotechnology put enormous challenges to the possibility of defining standard procedures to describe, specify and measure nano-objects in a reproducible and certifiable way.

NFFA addresses the key question of developing and adopting in first place an “internal” metrology and protocol of materials characterisation at the nanoscale, and at the atomic scale, in all its centres such to provide well described methods and data for external users as well as guaranteeing the reproducibility at any NFFA centres of such calibration and data. For such a purpose, the NFFA Technical Liaison (TL) – a kernel structure made of personnel, procedures, and data format (cfr. Deliverable 4.8) - aims at managing the technical competences in developing the common metrology and the protocols shared among the NFFA centres. NFFA aims at developing and defining appropriate measurement techniques, instruments and calibration procedures and protocols references materials. This will be done ensuring accuracy and reproducibility and full traceability of results with reference to the International System of Units (SI), according to guidelines and standards provided by National Metrology Institutes, where available, and moving beyond wherever it turns out to be needed.

Internal reference materials and procedures will be identified so that users accessing to laboratories located in the different NFFA centres can be ensured that they are investigating precise replica objects with the same resolution. For this purpose, all NFFA instrumentation will be calibrated and continuously monitored by periodical calibration and cross-check measurements within the whole NFFA infrastructure (all centres), with complete relevant documentation available to users for a high-level of traceability and reproducibility of the experiments. Such a continuous calibration and refinement of experimental reference procedures will also contribute to the development of new standard protocols providing fundamental insights for the possible definition of additional official standardisation and certification procedures.

The interaction between the NFFA staff and academic/industrial users will be at the base of the development of state of the art as well as novel instrumentation in all the NFFA centres. The NFFA personnel (senior scientists and highly-specialised technicians) will identify both the optimal investigation technique and the technical equipments to meet the needs of both the academic and industrial world, cross checking any development in all centres. Basing on the high level of internal competences and state of the art instrumentations which will always be pushed at their best resolution limit, cross-linked experiments will be performed in strong synergy with large scale facilities to cover an as broad as possible resolution range, according to the specific requests of the users. This dynamic and multidisciplinary approach will enhance the capability of NFFA to fulfil the requests of users and will turn into an increased overall return also in terms of implementation of the experimental environment and up-grade and development of the experimental tools set.

We expect that such definition of internal standard protocols, further improved by the users science, will create a reference for other users and qualify the data repository for advanced internal metrology at the nanoscale.

NFFA will operate in compliance with the available normative and regulations in matter of standards, wherever relevant to its goals. NFFA will therefore need to stay constantly updated with newly agreed and newly delivered protocols and standards. To facilitate this purpose, it will be helpful if NFFA scientists or technologists will take part of the activities of standardisation bodies.

The majority of methods for investigating and measuring the properties of and nano objects included in the previous section require calibration of the measuring instruments using standard samples of known composition, structure and properties. In the following the current status for the main techniques included in the basic tool set is reviewed and new possible internal solutions are proposed when needed.

6.1 Microscopy Section

6.1a Electron Microscopy

In electron microscopy the reliability of the information obtained from microscope image measurements is strongly related to the accuracy of the microscope's magnification calibration. Depending on the typology of electron microscope being used (SEM or TEM) and, as a consequence, on the technical requirements of the samples to be analysed (below 10 nm thickness for TEM samples), different calibration procedures are needed. In

general, the magnification scale M of the microscope can be defined as $M=L'/L$, where L is the size of the test object (measurement reference) used for the magnification calibration; L' is the size of the same test object as seen in the magnified image provided by the microscope. For SEM's, calibration magnification procedures are usually performed by using a line-width standard (magnification reference) as the test object. The nominal size L of the magnification reference is assumed to be known with adequate accuracy. However, obtaining the exact

pitch value L' from the microscope's image of the test object pattern is the crucial part of the calibration. The pitch reference contains several or many repeatable identical features (lines or stripes), which appear to be identical to each other regardless of the type of the microscope. This strongly facilitates evaluation of the pitch value of such structures present in the microscope image: the distance between any equivalent points of adjacent stripe pattern features in the image can be considered as the pitch value. Such points are established or noted by using the maxima or minima of brightness in the video signal, any repeated characteristic features on the videosignal slopes, etc. In particular, pitch magnification reference SRM-2090a provided by NIST (see Fig. 3) contains 8 separate parallel line structures, the pitch value between adjacent line features being about 200 nanometres [8]. Considerable efforts have been made by other National Metrology Institutes to fabricate special 1D grating standards (100 nm, 60 nm and 50 nm pitches). More recently, the National Metrology Institute of Japan NMIJ developed 'nanometric lateral scales' for SEM, AFM and optical diffractometer calibrations [9]. For EDS, WDS and EBSD analytical techniques in SEM, there are no certified materials and/or procedures. Calibration procedures can be usually performed on reference pure materials, which allow identification of the optimal initial conditions for reliable and traceable experiments. Calibration of the EDS detector can be made by acquiring a reference spectrum of a sample of known composition (usually Co). At the operating conditions of the instrument the counts number of Co is about 1,2 kcps. Calibration of the WDS spectrometer can be made by calibrating the detectors on a pure certified reference sample (usually Fe). At the operating conditions of the microscope for this analysis, the Fe counts number should be about 8 kcps. Finally, The calibration of the EBSD detector can be obtained by the acquisition of the diffraction pattern of a certified reference sample (usually Ge). At the operating condition for this analysis, the pattern quality has to be greater than 50%.

In the case of TEM's, the magnification calibration can be obtained by a cross-sectional TEM sample made of a semiconductor multilayer consisting of four sets of five nominally 10 nm thick $\text{Si}_{0.81}\text{Ge}_{0.19}$ alloy layers, alternating with nominally 13 nm thick pure silicon layers (MAG*I*CAL) [10]. The test object is grown by Molecular Beam Epitaxy (MBE) on a single crystal silicon <001> substrate. All calibrated values incorporate the strain effects. The calibration structure examined by TEM consists of four sets of bright and dark contrast alternating layers (superlattices) whose thicknesses are accurately known (see Fig.4). The calibration thickness measurements of the light (silicon) and dark (SiGe alloy) layers are obtained by high resolution TEM measurements performed with reference to the {111} lattice spacing of silicon, as measured on the single crystal silicon substrate. This spacing is known to be 0,313 560 136 (8) nm [11].

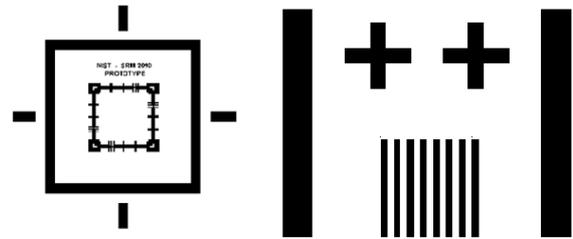


Fig 3: Diagram of the SRM 2090a prototype SEM magnification standard

The uncertainty in measurement across each of the full superlattices is less than one atomic layer at both the top and bottom interfaces: Δt (superlattice) < 0,5%. The MAG*I*CAL calibration sample is directly traceable to the lattice constant of silicon <1 1 1> (0,3135428 nm). This constant can be measured directly on the MAG*I*CAL sample, providing traceability to a fundamental constant of nature. One single calibration sample can be used to provide all three of the major TEM instrument calibrations at all magnifications and all cameras lengths.

NFFA will operate in compliance with the above mentioned standards and calibration procedures. By taking advantage of highly specialised scientists, NFFA will also contribute to identifying new experimental procedures, which will push ahead the achievable resolution limit of the instrumentation, providing fundamental insights for the implementation of the existing calibration procedures. Advanced software will be used for image simulation and deconvolution of the beam broadening to ascertain the nanostructures of the investigated samples down to the atomic scale. Image simulation codes will also be used in ultimate analysis to determine the sensitivity of instrumentation: by using well-characterised TEM standards, high resolution TEM images will be compared with processed and simulated images obtained at different experimental parameters (e.g. beam broadening, defocus values, specimen thickness) thus obtaining a bottom-up approach to check the instrument resolution status.

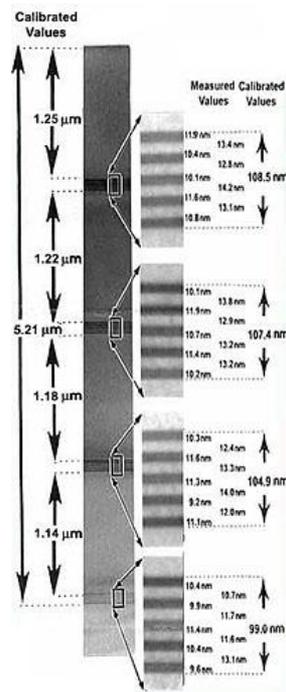


Fig 4: Calibration structure of the MAG*I*CAL TEM standard

6.1b Scanning Probe Microscopy

In Scanning Probe Microscopy the reliability of obtained images is affected by two main factors: the calibration of the scanner and the tip influence. In the effort towards a unified metrology, NFFA will address both problems, relying on existing calibration reference materials for the calibration of the scanner, and proposing new solutions to disentangle the tip influence from the real sample structure.

The first step will be the definition of common standards for the scanner calibration. In particular, for AFM and related techniques certified test gratings exist both for vertical and for in-plane calibration. For vertical calibration several gratings are commercially available in the 20-500 nm range while only a few solutions are presently offered on smaller scale, mainly based on nano-islands (see e.g. the PELCO AFM Gold Standard Kit [12] or the PELCO AFM Tip and Resolution Test Specimen [13]). Such test samples are indicated also by a recent US Patent on “Nanometrology device standards for SPMs and processes for their fabrication and use” [14]. Another nice example for an even higher resolution in vertical calibration for AFM is the STEPP (Silicon Test Echeloned Pattern) proposed by NT-MDT [15], which is designed on the base of a silicon (111) surface with a verified distribution of monoatomic steps (0,314 nm) with an average inter-step distance of ~0,5-2 μm and wider terraces between step bunches (see Fig. 5).

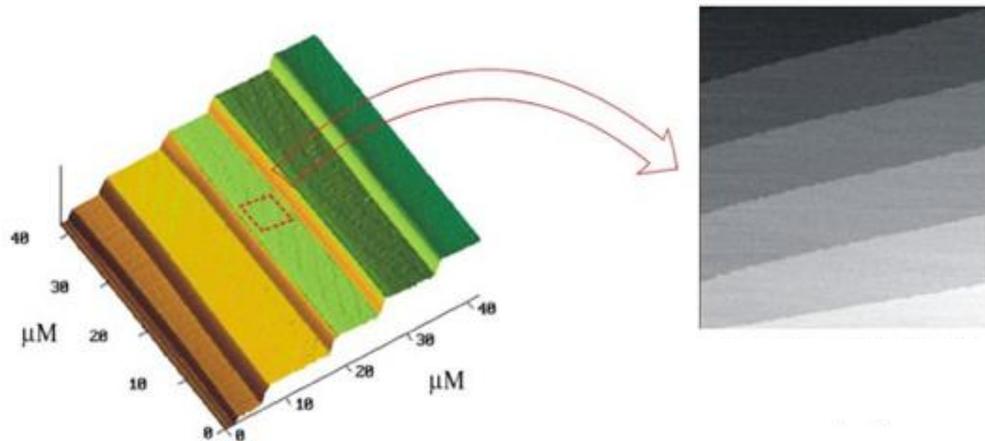


Fig 5: STEPP - Silicon Test Echeloned Pattern for AFM Probes by NT-MDT [Errore. Il segnalibro]

For in-plane calibration, chess board-like patterns of pitches/columns with certified distances in the μm -scale (see Fig. 6, left [16]) and linear (see Fig. 6, right [17]) or triangular gratings with certified intra-distances of a few hundred nm are available.

Scanner calibration for STM is usually performed on the base of a Highly-Ordered Pyrolytic Graphite (HOPG) sample.

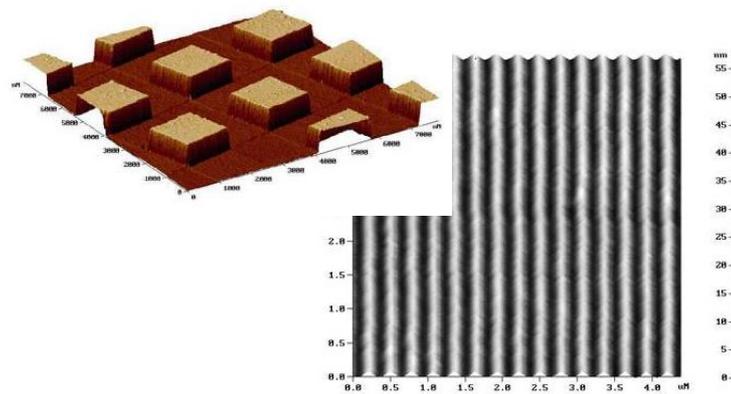
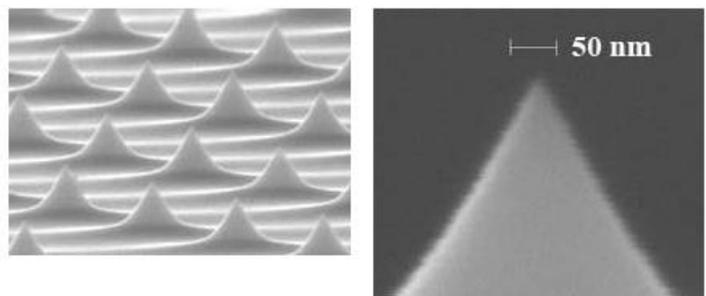


Fig 6: Test patterns for in-plane calibration of AFM. Left: TGQ1 from NT-MDT [16]. Right: TDG01 from NT-MDT [17]

As stated above, scanner calibration is not enough for ensuring reliability of SPM images. Even if distances are correct, the visualized information is in fact strongly influenced by the tip. In order to improve the degree of traceability of SPM measurements it is therefore necessary to address the problem of tip artefacts. NFFA will develop technical solutions for offering the possibility of determining the tip shape (see below) in combination with software for deconvolving ex-post artefacts related to tip shape from actual structural sample features.

The shape of the AFM tip can be visualized in 3D by scanning on a certified grating consisting in a regular array of sharp tips, such as the TGT1

Fig 7:
Example of special grating for 3D visualisation of AFM tips: TGT1 by NT-MDT [18].



produced by NT-MDT [18] in Fig. 7.

For STM, certified tip evaluation procedures are not presently available. NFFA will try to fill this gap by developing new reliable solutions. For example, short (~1 nm long) carbon nanotubes (CNT) could be used for tip visualization. As a substrate for CNT growth, Au or graphite or any other substrate passivated with gold could be considered. The reference sample should be mounted close to the unknown sample, ideally in the same sample holder in a modular way, with the possibility of adjusting the tilt in order to have the same tip-surface angle when measuring on the two samples. This and other possible solutions will be considered and tested during the start-up phase of NFFA.

Particular attention has finally to be paid to the evaluation of noise level, both electrical and mechanical. Specific limits have to be established and each system has to be periodically tuned to fit the limits.

After calibration, the performance of both AFM and STM will be finally checked on common test structures whose lateral dimension has to be related to the scope of the specific instrument, spanning from atomic resolution to 100 nm scale.

Test samples for specific capabilities of AFM related techniques (Magnetic Force Microscopy, Force Friction Microscopy, Scanning Thermal Microscopy, etc.) are also commercially available (see e.g. the Bruker website [19]) and will be used in all NFFA centres for periodic calibration of the instrumentation.

SNOM performances can be evaluated on the base of test gratings such as SNG-01, commercialised by both NT-MDT [20] and AIST-NT [21]. SNG01 Test grating for Scanning Near field Optical Microscope is a test object made of a quartz substrate partly coated with a metal (within an active area). The metal coating is made in the form of array of rhomboids that reflect an optical signal well.

6.2 Nanoscale test object to measure the lateral resolution in surface science

Calibration of the lateral resolution in surface analysis is a key point to ensure traceability and reproducibility of the measurements at the nanoscale. This is a crucial issue to be addressed in the NFFA cluster whose prominent scope is to offer advanced metrology approaches based on common principles of traceability and consistency thanks to the strong synergy between the NFFA centres and LSF. Periodic structures such as gratings are commercially available. They are useful for direct estimation of lateral resolution during the adjustment of instruments but no samples with regular structures below 100 nm are currently available. A great example of certified reference material which narrows the gap between the dimensions of lithographic patterns (>30 nm) and the crystal lattice <1 nm, has been developed by the Bundesanstalt für Materialforschung und -prüfung (BAM). The certified reference material BAM-L200 [22], second evolution of the previously introduced BAM-L002 [23], is a cross section of a AlGaAs-InGaAs-GaAs layer stack consisting of 142 layers composing a complex pattern in the sample surface. The strip widths, ranging from 1 to 600 nm, enable the estimation of the lateral resolution and the determination of specific functions characterising the lateral resolution. Moreover this test object can be used for the calibration of a length scale, the determination of instrument parameters, the optimisation of instrument settings and the continuous quality control of measurement methods for surface analysis. It is applicable to all measurement methods that are able to differentiate between the materials forming the semiconductor strip pattern. Such methods include analysis using incident electrons (SEM, AES), ions (SIMS) or photons (XPS, Photoelectron Emission Microscopy, PEEM). NFFA will definitely employ the commercially available BAM standard for calibration purposes. Moreover, by exploiting the strong interaction between nanocentres and LSF, NFFA will explore the possibility of developing new standard device/procedure to be applied to as many as possible synchrotron-based techniques in order to offer the possibility of making experiments at different light sources really comparable.

6.3 Calibration for other techniques

For the other previously mentioned techniques, NFFA will employ the commonly used calibration procedures. For instance, the calibration of SAXS and WAXS measurements is usually done by calculating the sample/detector distance from Diffraction rings of different reference materials depending on the specific instruments. The reference materials typically used for SAXS experiments are BSA (bovine serum

albumine), Collagen, Ag-behenate powder and Glassy carbon. For WAXS Al_2O_3 and Silicon powder are typically employed. An ISO-standard for SAXS is currently under development and will be introduced in all the NFFA centres when available.

Test measurements on magnetometers are usually performed on specific reference materials. More specifically, the calibration of a SQUID (consisting in the magnetic moment calibration) is usually determined by measuring a palladium standard over a range of magnetic fields and then by adjusting to obtain the correct moment for the standard. The palladium standard samples are effectively point sources with an accuracy of approximately 0,1%. Vibrating sample magnetometers (VSM) are traditionally calibrated by using a small high purity nickel sphere as a standard. For MOKE set-up, the calibration of the Kerr rotation angle can be performed on the Fe-wedge sample in the thickness range of 1–50 ML of Fe.

6.4 Common metrology at LSFs

Concerning access to radiation sources and beamlines, common data format and procedures will be defined in collaboration with the LSF scientists for offering to the NFFA users access to beamlines that will be characterised as far as concerns:

- Photon flux (monitored by calibrated diodes, with routine cross-checks)
- Photon polarisation
- Spectral purity (second and higher order intensity)
- Photon Energy stability
- Energy resolution
- Lateral resolution

Furthermore, calibration of thermometers for sample temperature measurements, Hall-probe characterisation of the magnetic environment of samples and spectrometers, linearity calibration of electron and photon detectors will be regularly performed. More standard quantities like total pressure in the UHV regime and residual gas analysis will be also monitored and calibrated in all relevant cases.

Evaporation / deposition rates for materials onto substrates will be calibrated by atomic absorption optical methods and microbalance methods, periodically verified externally by Rutherford backscattering at ion accelerator centres.

7. TOTAL CHARACTERISATION LINE

Part of the in-house methodological activity of the Advanced Analysis and Metrology facilities will be initially dedicated to the development of a “Total Characterisation Line”, as thoroughly presented and discussed in Deliverable 3.1 “Design study of NFFA infrastructure”. An adequate human and financial effort will be devoted to the implementation of this fully automated system, based on the idea of the assembly line, with a batch of samples moving inside a UHV pipeline through consecutive measurement stations, each equipped for a different analytical technique, and providing complementary information for the sample definition standard that will be well connected to the data repository for automated data storing and formatting.

When operative, the new system will be offered as a fast means for characterisation of basic and well-defined properties (being optical, chemical, electronic, as well as morphological), ensuring a full traceability of results. It will be therefore complementary to the standard, human-controlled activity that, being freed up from such simple, service-like, activity, will be entirely dedicated to address more specific and/or more challenging scientific questions.

8. COST ESTIMATE

NOTE: Total numbers based on 4 NFFA centres.

In the following: *Resolution* = spatial resolution; *Flexibility* = possibility of using the same instrument for different samples/conditions;

Speed = time response in terms of useful results; *Robustness* = sturdiness; *Access mode* = open hands on/limited hands on/hands off (see text §3.4);

Environment = required environment for operation

TECHNIQUES	Resolution	Flexibility	Speed	Robustness	Access mode	Environment	Units	Cost per unit (M€)	Total investment (M€)	Running cost per unit/year (M€)	Dedicated staff per unit	Total staff
BASIC TOOL SET												
Microscopy												
SEM+EDS	medium/ high	high	high	high	open	Lab	4	0,5	2	0,05	0,5	2
TEM/STEM	high	high	medium/ low	medium	limited	Lab with specific requirements	3	2	6	0,2	2	6
FIB	high	high	medium/ low	medium	open	Lab or cleanroom	4	1	4	0,1	0,5	2
AFM in air – large sample	medium	high	high	high/ medium	open	Lab or cleanroom/ air	4	0,25	1	0,02	0,1	0,4
AFM in air – High res	high	high	high/ medium	medium	limited	Lab or cleanroom/ air	4	0,2	0,8	0,02	0,1	0,4
RT STM/AFM in UHV	high	medium	medium/ low	medium/ low	limited	Lab/UHV	4	0,3	1,2	0,03	1,25	5
RT STM in air	medium/ high	medium	medium	medium	open	Lab or cleanroom/ air	4	0,1	0,4	0,01	0,5	2
VT STM	high	medium/ low	medium/ low	medium/ low	limited	Lab/UHV	4	0,4	1,6	0,04	1,25	5

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TECHNIQUES	Resolution	Flexibility	Speed	Robustness	Access mode	Environment	Units	Cost per unit (M€)	Total investment (M€)	Running cost per unit/year (M€)	Dedicated staff per unit	Total staff
Struct., Opt. &Comp. Analysis												
Four circles XRD	high	high	medium	high	Open	Lab	4	0,2	0,8	0,02	0,25	1
powder XRD	high	high	high	high	Open	Lab	4	0,1	0,4	0,01	0,25	1
basic SAXS/GISAXS	medium/high	high	variable	medium	limited	Lab with specific requirements	4	0,2	0,8	0,02	0,5	2
Profilometry	medium	high	high	medium	open	cleanroom	4	0,12	0,48	0,05	0,05	1
Electroluminescence	medium	high	high	high	open	Lab	4	0,05	0,2	0,005	0,1	0,4
Photoluminescence	medium	high	high	high	open	Lab	4	0,06	0,24	0,006	0,1	0,4
Ellipsometry/SE	variable	high	high	medium	open	Lab/air	4	0,1	0,4	0,01	0,5	2
DLS	high	high	high	high	limited	Lab	4	0,12	0,48	0,05	0,25	1
FTIR	high	high	high	high	open	Lab/Vacuum&air	4	0,08	0,32	0,01	0,2	0,8
μ-Raman	medium	high	medium	medium	open	Lab/air	4	0,15	0,6	0,06	0,3	1,2
Fluorescence	medium	medium	high	high	open	Lab	4	0,15	0,6	0,06	0,1	0,4
UV-Vis	medium	medium	high	high	open	Lab	4	0,2	0,8	0,08	0,1	0,4
TCSPC	medium	medium	medium	high	limited	cleanroom	4	0,25	1	0,1	0,5	2
LEEM/PEEM	high	high	medium/high	high	limited/hands off	Lab/UHV	3	0,5	1,5	0,05	1,5	4,5
XPS + AES	medium	medium/high	medium/high	medium	open/limited	Lab/UHV	4	0,35-0,40	1,4-1,6	0,04	1	4
Magnetic Charact.												
SQUID	high	medium	medium	high	hands off	Lab	4	0,25	1	0,02	1	4
VSM	medium	medium	high	high	limited	Lab	4	0,15	0,6	0,01	0,5	2
basic MOKE	medium	medium	high	high	open	Lab	3	0,03	0,09	0,003	0,2	0,6

NFFA - Nanoscience Foundries and Fine Analysis

TECHNIQUES	Resolution	Flexibility	Speed	Robustness	Access mode	Environment	Units	Cost per unit (M€)	Total investment (M€)	Running cost per unit/year (M€)	Dedicated staff per unit	Total staff
Thermal&Mechanical Charact.												
TG	high	high	High	high	limited	Lab	4	0,06	0,24	0,01	0,5	2
DSC	high	high	high	High	limited	Lab	4	0,06	0,24	0,01	0,5	2
Transport Properties												
AC/DC probe station	high	high	medium	high	Open	Lab	4	0,035	0,14	0,005	0,25	1
Cryostats (closed circuit cryo-cooler w external low field magnet, PPMS with high field magnet)	High	High	medium	high	open	Lab	4	0,1	0,4	0,01	0,25	1
ADVANCED TOOL SET												
Advanced Microscopy												
SEM+WDS+EBSD	high	high	medium	high	limited/hands off	Lab	1	0,8	0,8	0,08	0,5	0,5
TEM Cs corrected	high	high	low	high	limited/hands off	Lab with specific requirements	1	5	5	0,5	2	2
3D Atom Probe	high	high	low	high	limited/hands off	Lab	1	2	2	0,2	2	2
LT-STM/AFM	high	low/medium	low/medium	low	limited hands off	Lab/UHV	1	0,45	0,45	0,06	1,25	1,25
LT-STM High Magn. Field	high	low/medium	low	low	limited/hands off	Lab/UHV	1	0,6	0,6	0,075	1,25	1,25
HP-STM	high	low	low	low	limited/hands off	Lab/UHV	1	0,5	0,5	0,05	1,25	1,25

NFFA - Nanoscience Foundries and Fine Analysis

TECHNIQUES	Resolution	Flexibility	Speed	Robustness	Access mode	Environment	Units	Cost per unit (M€)	Total investment (M€)	Running cost per unit/year (M€)	Dedicated staff per unit	Total staff
ECSTM	high	low	medium	medium/low	limited	Lab/liquids	1	0,2	0,2	0,02	1	1
Fast STM unit	high	high	medium/high	high	open/limited	Lab	1	0,05	0,05	0,005	0,1	0,1
SNOM	high	medium	low	low	limited	Lab/air	2	0,15-0,2	0,3-0,6	0,02	1,5	3
Cryo-SNOM	high	low	low	low	limited	Lab/HV	1	0,25	0,25	0,035	1,5	1,5
HeIM	high	high	medium	high	limited/hands off	Lab	1	1	1	0,1	1	1
TERS	high	medium	low	low	hands off	Lab	1	0,3	0,3	0,03	1,5	1,5
Brillouin Microscope	high	medium	low	low	hands off	Lab	1	0,17	0,17	0,02	1,5	1,5
Struct., Opt. &Comp. Analysis												
SAXS/WAXS/GISAXS	medium/high	high	variable	medium	limited	Lab with specific requirements	1	0,3-0,6	0,3-0,6	0,03-0,06	0,5	0,5
UFTA	medium	medium	high	high	limited	cleanroom	2	0,25	0,5	0,1	0,5	1
TRPES	medium	medium	high	high	limited	cleanroom	2	0,25	0,5	0,1	0,5	1
CA LEEM/PEEM	high	medium/high	medium	high	limited/hands off	Lab/UHV	1	1	1	0,1	1,25	1,25
HP-XPS	medium	low/medium	medium	medium/low	limited	Lab/UHV with HP cell	1	0,5	0,5	0,05	1,25	1,25
XPS in liquids	medium	low/medium	medium	medium/low	limited	Lab/UHV	1	0,5	0,5	0,05	1,5	1,5
SIMS	high	high	low	low	limited/hands off	Lab with specific requirements	1	1,5	1,5	0,15	2	2
NMR	high	medium	low	high	hands off	Lab	1	1	1	0,1	1	1
GC and GC/MS	high	medium/high	high	high	open	Lab	2	0,075	0,15	0,03	0,05	0,1

NFFA - Nanoscience Foundries and Fine Analysis

TECHNIQUES	Resolution	Flexibility	Speed	Robustness	Access mode	Environment	Units	Cost per unit (M€)	Total investment (M€)	Running cost per unit/year (M€)	Dedicated staff per unit	Total staff
Microreactors	not appropriate	medium/high	high	high	open	Lab	2	0,03	0,06	0,003	0,05	0,1
Magnetic Charact.												
MOKE	high	medium	high	medium	hands off	Lab	1	0,17	0,17	0,02	1	1
AC-SUSCEPTIBILITY	high	low/medium	medium	high	limited	Lab	1	0,25	0,25	0,02	0,5	0,5
Thermal&Mechanical Charact.												
Nanoindentation	high	medium	high	high	hands off	Lab	1	0,1	0,1	0,01	1	1
Acoustic measurements	high	low	high	medium	open	lab	1	0,02	0,02	0,002	0,5	0,5
Transport Properties												
Electronic and Probe station	high	high	Medium	low	Limited	Lab	1	0,25	0,25	0,03	1,5	1,5
10mK Cryostat	high	high	low	low	Limited	Lab with specific requirements	1	0,25	0,25	0,05	1	1
300mK Cryostat	high	high	medium	medium/low	Limited	Lab	1	0,05	0,05	0,02	0,5	0,5
Additional resources required												
LSF Located Section							4				1,5	6
Total Characterization Line	medium/high	high	high	high	open	Lab	1 [*] /4	3	3 [*] /12	0,3	3 [*] /0,2 ^{**}	3 [*] /0,8

* development phase

** running phase

APPENDIX A – Brief Analysis of the Metrology Facility Tools

Microscopy Section:

a) Electron Microscopy

Scanning electron microscopy (SEM) Scanning Electron Microscopes are among the most versatile and widely used tools of modern science as they allow the study of both morphology and composition of materials, from inorganic to biological ones. In brief, in a SEM a beam is scanned over the sample surface in a raster pattern while a signal from secondary electrons (SE) or Back-scattered electrons (BSE) is recorded by electron detectors. The electron beam, which typically has an energy ranging from a few hundred eV up to 40 keV, is focused to a spot of about 0.4 nm to 5 nm in diameter. Latest generation SEM's indeed can achieve a resolution of 0.4 nm at 30 kV and 0.9 nm at 1 kV.

FESEMs models cover all fields of applications in nanotechnology, cryo, nanostructural materials analysis, semiconductor development and failure analysis as well as characterisation of biological samples (cfr. Environmental SEM). They should therefore be introduced as a basic facility in all the NFFA centres.

Compositional and Crystallographic Analysis with SEM Beyond the ability to image a comparatively large area of the specimen, SEM can be equipped with a variety of analytical techniques for measuring the composition, crystallographic phase distribution and local texture of the specimen. X-ray photons are generated when the beam impacts the sample. This radiation provides chemical information when analysed using energy dispersive X-ray spectrometry (EDS) and wavelength X-ray spectrometry (WDS). Backscattered electrons can also be used to form an electron backscatter diffraction (EBSD) image that can be used to determine the crystallographic structure of the specimen at a resolution of 20 nm. EDS is therefore a basic technique to be introduced in all NFFA centres. SEM's equipped with WDS and EBSD techniques can be instead proposed as advanced equipments in specific NFFA centres.

Transmission Electron Microscopy (TEM) and Scanning Transmission Electron Microscopy (STEM) are related techniques which can be considered as the most powerful tools to characterise nanomaterials and indispensable for nanotechnology. In both the cases, high energy electrons, incident on ultra-thin samples, allow for image resolutions that are on the order of 1-2 Angstroms. The electron beam travels through the specimen and, depending on the density of the material present, some of the electrons are scattered, while unscattered electrons hit a fluorescent screen at the bottom of the microscope, giving rise to a "shadow image" of the specimen with its different parts displayed in varied darkness according to their density (Fig. 3). In the STEM mode, electrons pass through the specimen, but, as in scanning electron microscopy, the electron optics focus the beam into a narrow spot which is scanned over the sample in a raster. The rastering

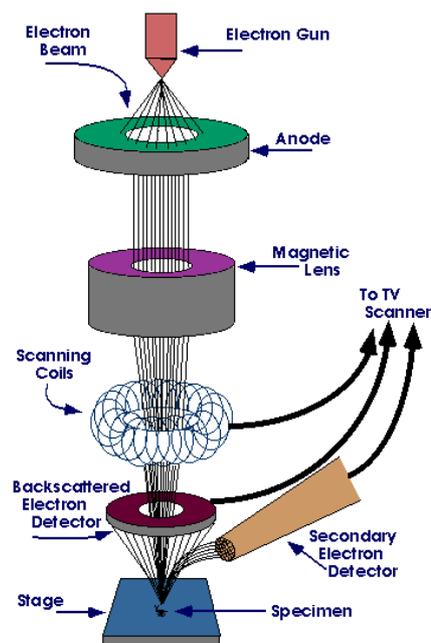


Fig. 8: Schematic drawing of a SEM

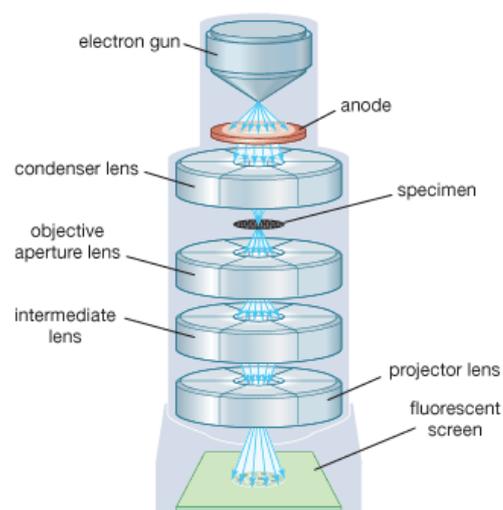


Fig 9: Schematic drawing of a TEM

of the beam across the sample makes these microscopes suitable for analysis techniques such as mapping by energy dispersive X-ray (EDX) spectroscopy, electron energy loss spectroscopy (EELS) and annular dark field imaging (ADF). These signals can be obtained simultaneously, allowing direct correlation of image and quantitative data. By using a STEM and a high-angle detector, it is possible to form atomic resolution images where the contrast is directly related to the atomic number. Traditionally, TEM/STEM have been mainly applied for imaging, diffraction, and chemical analysis of solid materials. For biological samples, cell structure and morphology is commonly determined whilst the localisation of antigens or other specific components within cells is readily undertaken using specialised preparative techniques and, when required specific TEM cooling, holder. A TEM can also be used to do *Electron Tomography*, which allows obtaining detailed three dimensional (3D) structural characterisation of 3D objects. This is accomplished by multiple views of the same specimen obtained by rotating the angle of the sample along an axis perpendicular to the beam. By taking multiple images of a single TEM sample at differing angles a set of images can be collected. For the wealth of fields of application, FETEM/STEM equipped with EDX, ADF, EELS have to be considered as basic tools to

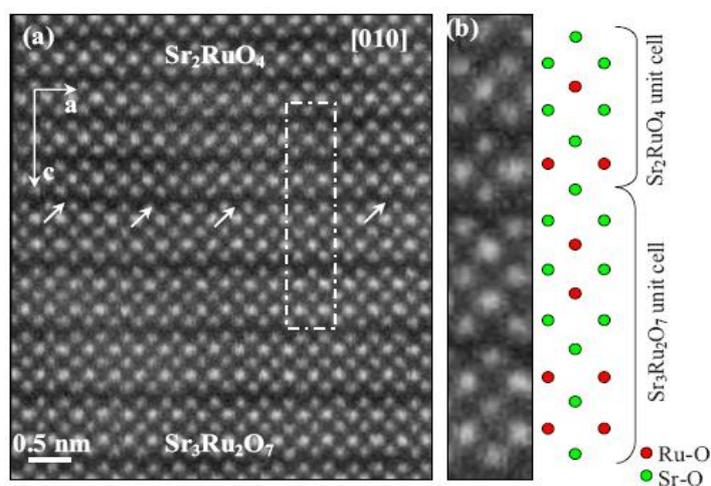


Fig. 10: HAADF-STEM image of $Sr_2RuO_4/Sr_3Ru_2O_7$ interfaces in $Sr_2RuO_4/Sr_3Ru_2O_7$ eutectic crystals taken by a C_s corrected Titan TEM/STEM [27].

introduce in all the NFFA centres. In the last few years, there has been a considerable revolution in electron microscopy with the arrival of aberration correctors for the objective lens [24,25] with the consequent improvement in the attainable resolution limits. The obtainable resolution limit now lies at around 0,1 nm or better in both TEM and STEM, and the improved images from these aberration-corrected microscopes are opening up new avenues in the characterisation of materials [26,27] (see Fig. 10). These new generation electron microscopes are of crucial importance for the development of nanotechnology and have to be considered as advanced tools in the NFFA technical layout.

Sample preparation is the most crucial part in TEM experiments. High quality TEM specimens have a thickness that is comparable to the mean free path of the electrons that travel through the samples, which may be only a few tens of nanometres. Preparation of TEM specimens is specific to the material under analysis and the desired information to obtain from the specimen. As such, many techniques have to be used for the preparation of the required thin sections and to be considered as crucial supporting facilities to the Microscopy facilities in all the NFFA centres. Sample preparation laboratories must be equipped with the basic tools (diamond saw, polisher, dimpler, electropolisher, ultrasonic cutter, precision ion polishing system, gentle mill, plasma cleaner) commonly used in conventional chemical and mechanical thinning procedures. Moreover, the following equipments must be present:

Focused Ion Beam (FIB) Scanning Electron Microscope with a Dual Beam source This facility has been already described in section 5.2 of D3.2. Beyond nanolithography purposes, due to its ability to perform precision *in-situ* milling, FIB technology has been proved to be a fundamental tool for studying 3D structural (tomography) and chemical relationships. With the help of computer algorithms for processing data and graphics packages for display, 3D systems can be reconstructed and the structure analysed to obtain both qualitative and quantitative information. This allows the tomographic reconstruction of many systems at spatial resolutions at the tens-of-nanometres level and volumes with dimensions of up one to tens of microns. one of the greatest applications of FIB technology is for TEM sample preparation [28]. Clear advantages of the FIB thinning over conventional Ar ion milling are the high spatial precision, the uniform thinning of materials, the large dimensions of the obtained electron

transparent area, and the high yield. Moreover, the in-situ “lift-out” technique enables the in-situ extraction of electron transparent regions suitable for TEM at specific regions of the samples by means of a mechanical micromanipulator located within the FIB chamber. It is remarkable that, the possibility of using a micromanipulator inside a FIB dual beam system along with the possibility of simultaneously milling, depositing and imaging is very recently triggering a number of novel and intriguing experiments aimed at performing the in-situ extraction, manipulation and microcontacting of reduced-size samples from a bulk matrix to characterise their functional properties [29,30]. This technique has opened a pathway to directly correlate nanostructures and functionalities of individual micro-constituents within complex structures.

Microtome is a sectioning device that allows for the cutting of extremely thin slices of material, known as sections. Microtomes are important devices in microscopy preparation, allowing for the preparation of samples for observation under transmitted light or electron radiation. Microtomes use steel, glass, or diamond blades depending upon the specimen being sliced and the desired thickness of the sections being cut. Steel blades are used to prepare sections of animal or plant tissues for light microscopy histology. Glass knives are used to slice sections for light microscopy and to slice very thin sections for electron microscopy. Industrial grade diamond knives are used to slice hard materials such as bone, teeth and plant matter for both light microscopy and for electron microscopy. Gem quality diamond knives are used for slicing thin sections for electron microscopy.

Three-dimensional Atom Probe Three-dimensional Atom Probe (3DAP or TAP, Tomographic Atom Probe) is the only material analysis technique offering extensive capabilities for both 3D imaging and chemical composition measurements at the atomic scale (around 0.1-0.3nm resolution in depth and 0.3-0.5nm laterally). Since its early developments, 3D Atom Probe has contributed to major advances in materials science. The sample is prepared in the form of a very sharp tip by means of FIB technology.

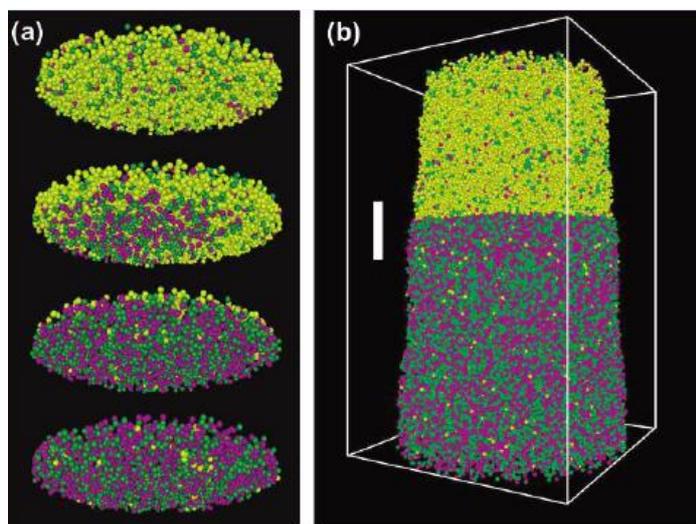


Fig. 11:- 3D-reconstruction of catalyst nanowire interface in three dimensions ([31] with minor modifications).

The cooled tip is biased at high DC voltage (5-20 kV). The very small radius of the tip and the high voltage induce a very high electrostatic field (tens V/nm) at the tip surface, just below the point of atom evaporation. Under laser or HV pulsing, a few atoms are evaporated from the surface, by field effect (near 100% ionization), and projected onto a Position Sensitive Detector (PSD) which simultaneously measures the time of flight of the ions and the (X,Y) position of the ion impact on the detector. By repeating this sequence, the atoms are progressively removed from the tip, and a 3D image of the material can be reconstructed at the atomic scale (see Fig. 11 [31]). This equipment should be included as advanced facility in the NFFA centres.

b) Scanning Probe Microscopy

Scanning Probe Microscopy (SPM) is a class of techniques based on a nano-scale probe that is scanned over the surface of the sample (Fig. 12), recording the probe-surface interaction as a function of position. Due to the small size of the probe end, the lateral resolution of scanning probe microscopes reaches the nanometre and sub-nanometre scale. Initially developed for purely morphological characterisation down to the atomic level, SPM progressively expanded its capabilities, resulting in a large number of different applications, including bottom-up atomic scale assembly. Scanning Probe

Microscopes are presently wide spread in nano-science laboratories for the investigation of material properties at the nano-scale. The main SPM techniques are:

Scanning Tunneling Microscopy (STM) An STM is based on the measurement of the tunneling current between a sharp metallic tip (probe) and a conductive sample. Atomic resolution can be obtained (Fig. 13 [32]), due to the exponential dependence of the tunneling current on the tip-sample distance, but topographic and electronic information are always convoluted. In addition to scanning across the sample, information on the electronic structure at a given position on the sample can be obtained by sweeping voltage and measuring current at a specific location, in so-called Scanning Tunneling Spectroscopy (STS). Tip conditions at the

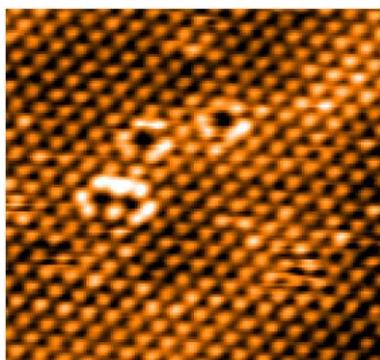


Fig. 13: Atomic resolution STM image of a $\text{CeO}_2(111)$ surface (see also [32]).

single atom level are almost unpredictable and unstable but greatly influence the experimental results therefore presently a standardisation of the performance is very difficult. STM can be operated both in air and in UHV, and, when specifically built, at high pressure (HPSTM) or in liquids (Electrochemical STM) and in a wide temperature range (from a few K to \sim thousand K). Although always limited to conductive samples, STM has therefore become crucial in a variety of different research fields, spanning from basic science to energy, catalysis, biophysics.

At liquid helium single molecule spectroscopy (both elastic – STS - and inelastic – IETS -) paves the way to chemical control at the single atom level, while spin-polarised STM (SP-STM), involving the use of magnetic coated tips and operation in high magnetic fields, allows obtaining spin-dependent surface information at length scales down to the single atom, like spin effects on the electronic and transport

properties in nanostructures. STM is used also for the investigation of dynamical processes but a too low frame rate is presently a major limit. Special, dedicated, home-built microscopes as well as fast units to be used as a retrofit to commercial instruments have been recently developed to increase the frame rate up to video rate but currently are not commercially available.

RT and VT STMs operating both in air and in UHV are necessary in all NFFA centres, while a distribution of LT-STM, SP-STM, EC-STM and HP-STM in specific NFFA centres within the advanced offer is advisable. A single dedicated Fast-STM or a few Fast modules will also be offered as soon as available as part of the advanced tool-set.

Atomic Force Microscopy (AFM) An Atomic Force Microscope uses as probing signal the deflection of a cantilever due to attractive/repulsive forces between a small tip attached at one end of the cantilever and the sample surface. Two operational modes are possible: “Contact AFM”, when the tip is in soft physical contact with the sample, and “Non-contact NC-AFM”, when the cantilever is vibrated near the surface. In the latter case the changes in tip-sample distance reflect in changes in the resonant frequency. Conversely to STM, AFM can be used also for the investigation of non-conductive sample. It is also more flexible and, in its basic version, has the advantage of providing purely morphological information. Atomic resolution can nowadays be reached even though less routinely than with STM.

AFM can be operate in air as well as in vacuum or in liquids and is becoming increasingly more and more relevant in nanoscience, including bionanotechnology as well as for semiconductor industry. The presence of an AFM working in air combined with an optical stage for large samples [see e.g. 33] as well as an AFM in air for high-resolution measurements, and one UHV AFM in each NFFA is highly advisable.

AFM related techniques AFM turned out soon to be very flexible and likely to a variety of applications at the nano-scale. Modified tips/instrumentation can measure chemical, magnetic,

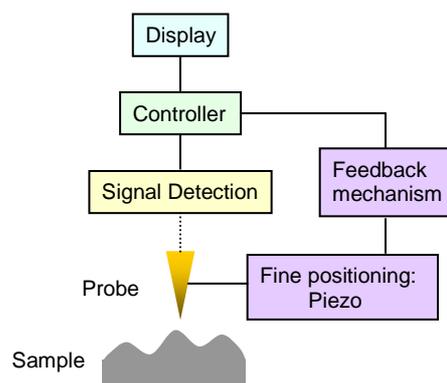


Fig. 12: General schematics of a Scanning Probe Microscope

electrical, thermal and optical surface properties. The most important AFM-related scanning probe techniques that have to be taken into consideration among the offer of NFFA centres are: Lateral AFM (LFM) or Friction Force Microscopy (FFM), that maps relative differences in the frictional forces between the probe tip and the sample surface, thus, among other applications, identifying transitions between different components in polymer blends and composites, identifying contaminants on surfaces, delineating coverage by coatings and other surface layers; Chemical Force Microscopy (CFM) that uses tips functionalised for specific chemical or biological species; Electrostatic Force Microscopy (EFM) for measuring electric field gradient distribution above the sample surface, due for instance to locally charged domains; Scanning Thermal Microscopy (SThM) that maps the local temperature and thermal conductivity; Scanning Kelvin Probe Force Microscopy (SKPFM) that, by detecting contact potential differences between the probe and the sample, allows mapping work function changes at the nano-scale. Due to its constantly increasing diffusion, particular attention has to be paid for Magnetic Force Microscopy (MFM) and Magnetic Resonance Force Microscopy (MRFM) for characterisation of magnetic properties (see below). All these more specialised applications of AFM are usually offered as options with normal AFMs by the dealers and can thus be easily added to the NFFA offer. However, for specific centres and/or specific applications (for ex. MFM or SKPFM or bio-oriented) dedicated microscopes can be considered.

Magnetic Force Microscopy (MFM) This type of microscopy is designed to observe magnetic domains both at zero field or under the action of a magnetic field, applied either along the film plane or perpendicular to it. MFM can be also performed on patterned magnetic structures and, in conjunction with MOKE measurements and micromagnetic simulations, it provides insightful knowledge about the magnetisation reversal processes occurring at the micron or submicrometre scale (see Fig. 14).

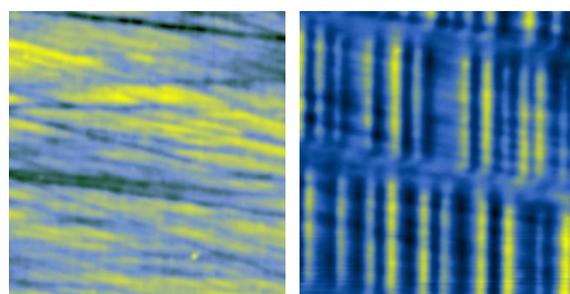


Fig. 14: Magnetic Force Microscopy: topographic (left) and magnetic (right) contrast on a $10\mu\text{m}\times 10\mu\text{m}$ area of a 40Gb Hard Disk. Courtesy of A.P.E. Research (images obtained with the A100-SGS AFM)

Scanning Near Field Microscopy (SNOM) makes it possible to overcome the diffraction limit of standard (far-field) optical microscopy by using evanescent photon field. SNOM can thus combine optical measurements with the typical spatial resolution of AFM. In *aperture SNOM*, a small aperture at the tip of a tapered optical fiber that is scanned over the sample acts as the light source or as the detector to collect the intensity of the near field generated at the aperture. In *apertureless SNOM*, conversely, the metalized tip of a standard AFM probe illuminated from the far field simply provides the near field. Cryogenic SNOM are particularly suited for optical spectroscopy of semiconductors. At low temperatures (liquid He) optical processes are less influenced by non-radiative recombination effects: phonons are frozen and therefore inactive, so the only possible channel for the energy is from photons to excitons and vice versa. The resulting increasing of the radiative emission yield is the more stringent condition motivating the experimental efforts towards L-He temperatures.

c) Emerging Microscopes

The Scanning Helium Ion Microscope (HeIM) is a new imaging technology based on a scanning helium ion beam. The two striking features of this tool are the ion source and the nature of the beam/sample interaction. The long-lasting helium-ion source consists of a sharp needle held under high vacuum in cryogenic temperatures providing intense ion currents from a volume no larger than a single atom, with a spot size of about 0,25 nm. Compared to a SEM, the secondary electron yield is quite high, allowing for imaging with currents as low as 1 femtoamp with increased benefits in resolution, material contrast, charge control, and surface sensitivity with respect to conventional SEM. This microscope has an enormous potential in development of nanoscience and should be included as advanced facility in the NFFA centres.

Tip Enhanced Raman Spectroscopy (TERS) combines a raster scanning probe device with a Raman spectrograph. The core of this unit is a silver or gold tip of suitable size that, when illuminated, greatly enhances Raman signals of molecules situated in its vicinity, in an area smaller than the normal far-field diffraction limit. The spatial resolution is approximately the size of the tip apex (20-30 nm). TERS has been shown to have sensitivity down to the single molecule level. TERS has a wide range of applications for example, in surface science, heterogeneous catalysis, single molecule spectroscopy, and biology.

Confocal Brillouin Microscopy is a recently developed technique providing cross-sectional Brillouin imaging based on elastic properties as contrast mechanism, capable of monitoring fast dynamic changes in elastic modulus [34]. Presently this microscope is not commercially available but NFFA will benefit from developing a home-made Brillouin microscope as part of the advanced tool set for non-invasive measurement of mechanical properties at the micro-scale.

Structural, Optical and Compositional Analysis Section:

a) X-Ray Laboratory

X-Ray Diffraction (XRD) is an ideal and basic tool for nanoscience, because the spatial resolution is in the same order of the X-ray wavelength, typically 0.1 to 0.15 nm. Bragg-peak positions and their intensities provide, the crystal lattice structure and the electron density map of the unit cell of the studied material, respectively.

In the case of materials characterised by high intensities of the diffractive peaks (single crystals, epitaxial films, etc.), diffractometers generally work with a standard hot filament X-ray tube and by mounting additional monochromators for both incident and diffracted beam, which allow a very accurate measurement of the spot positions (four circles diffractometer). However, in the case of materials with lower diffractive intensities (powders, partially crystalline polymers, biological materials, etc), the intensity of the incident X-ray beam can be increased by using a rotating anode source. The time of the recording process is drastically reduced by using position-sensitive detectors (PSD) and/or 2D-area detectors, which detect in one image a complete diffraction pattern (powder diffractometer), thus allowing whole processes such as polymer melting to be followed. Both kinds of equipments should be inserted as basic tools in all the NFFA centres.

Small-Angle X-ray Scattering (SAXS) provides a method for structural characterisation at the size range of one to several nm, based on the diffuse elastic scattering around the primary beam at small angles. The angular intensity decay around the scattered beam is characteristic for the size and shape of the scattering structures, which are inhomogeneities in the electron density in the sample, for example particles in a matrix or a solvent or a combination of two material phases. Simple set-ups allow a fast characterisation of mesostructures (size, shape, interface properties, internal surface, orientation, etc.), at a size range that is complementary to other methods like X-ray diffraction, light scattering or electron microscopy, but without the need for invasive preparation (even in solution or wet condition). Appropriate sample environments and additional set-ups allow in situ experiments for the observation of e.g. structural changes during synthesis [35], sorption/desorption behaviour [36] or mechanical response at the nano-level to loads [37]. For research on mesostructured materials it offers an interesting complementary method to sorption experiments by providing information on the total porosity (vs. accessible porosity by sorption experiments). Specialised set-ups allow the structural examination of thin films by *Grazing Incidence Small-Angle X-ray Scattering (GISAXS)* [38].

b) Optics Laboratory

Ellipsometry is a contact-free, nondestructive method for characterisation of the dielectric and optical properties (refractive index, absorption and thickness) of layered nanostructures in the size range of < 1 nm to several μm . The samples are usually measured in reflection and the information is given on basis of the change of polarisation of the reflected light. Typical applications are the characterisation of manufactured devices (semiconductors, nanoelectronics, thin films) and surface characterisation including film growth, roughness and adsorption processes [39]. Interesting experimental options are

given by in-situ applications during e.g. surface modification treatments [40], combinations with imaging techniques or other (non-destructive) analytical methods, e.g. (micro-)Spectroscopy in *Spectroscopic Ellipsometry (SE)* [41].

Dynamic Light Scattering (DLS) DLS is a technique used for the study of particle size distribution. In DLS laser light passes through the sample (in solution) and the scattered light is detected, e.g. with a photomultiplier tube. The time-dependent intensity fluctuations in the scattered light, arising from the Brownian motion of particles, can be related (via the autocorrelation function) to the diffusion coefficient of the molecules/particles in the solution. The diffusion constant can in turn be used to estimate the hydrodynamic radius of the particle. DLS can also be used to probe the behaviour of complex fluids such as concentrated polymer solutions and to study polydisperse solutions.

Profilometry is a non-destructive and easy-to-use technique used to measure the profile a surface in order to quantify its roughness. Vertical resolution is usually at the nanometre level, though lateral resolution is usually poorer. In the field of nanotechnology it can be used for qualitative and quantitative control of lithography, etching and deposition procedures for the calibration and the optimisation of microfabrication processes and thick film deposition. In *contact profilometers* a diamond stylus is moved vertically in contact with a sample and then moved laterally across the sample for a specified distance and specified contact force. A profilometer can measure small surface variations in vertical stylus displacement as a function of position. A typical profilometer can measure small vertical features ranging in height from 10 to 65.000 nm. The radius of diamond stylus ranges from 5 μm to about 25 μm , and the horizontal resolution is controlled by the scan speed and scan length. An *optical profilometer* is a non-contact method for providing much of the same information as a stylus based profilometer. There are many different techniques which are currently being employed, such as laser triangulation (triangulation sensor), confocal microscopy and digital holography. Optical profilometers do not touch the surface and therefore cannot be damaged by surface wear or careless operators. Many non-contact profilometers are solid-state which reduces the required maintenance significantly. The spot size, or lateral resolution, of optical methods ranges from a few micrometres down to sub micrometre, which is roughly an order of magnitude smaller than typical stylus tips.

Fourier Transform Infrared Spectroscopy (FTIR) is a technique for collecting infrared spectra. An IR beam that has been sent through an interferometer is passed through the sample. Frequencies which match the natural vibration frequencies of the molecules present are absorbed by the sample. An interferogram is collected and converted into a spectrum by using the Fourier transform. The spectra can be used to study the three dimensional structure of proteins, the chemical composition of a sample, energies of vibrational modes, molecular conformation, inter- and intramolecular interactions. The technique is not a nano-technique in itself, but can be used for example for quality control of proteins for incorporation into products such as arrays or nanoparticles.

In *μ -Raman Spectroscopy* the sample is illuminated with a laser beam and the light scattered by the material is analysed by a conventional optical microscope coupled to a Raman spectrometer. The Raman effect gives information on the material chemical composition, state, aggregation, and even factors like stress, orientation or temperature to cite some. μ -Raman is suitable for the examination of thin films, coatings, microelectronic integrated circuits, minerals, polymers and ceramics, biological and medical specimens, pigments in art works and others. As in any optical microscopy its spatial resolution is close to the micron. By using novel techniques, information can be gained from structures on nanometre scale (e.g. Raman may be used to classify the diameter of carbon nanotubes, given that the frequency of the radial breathing mode is related to the tube diameter). Superior spatial resolution is possible by simultaneously collecting AFM image and Raman data reaching a resolution of approximately 250 nm; or taking advantage of the enhanced spatial resolution offered by Tip Enhanced Raman Spectroscopy (TERS) with a resolution up to 20 nm. While μ -Raman will be available in all NFFA centres, TERS will be offered as part of the Advanced Tool Set of the Microscopy Section in one or more specific centre(s).

Fluorescence spectroscopy involves using a beam of light, usually UV light, that excites the electrons of molecules of certain compounds and causes them to emit light of a lower energy, typically, but not necessarily, visible light. The technique is complementary to absorption spectroscopy.

Ultraviolet and visible absorption spectroscopy (UV-Vis) is the measurement of the attenuation of a beam of light after it passes through a sample or after reflection from a sample surface. Absorption measurements can be at a single wavelength or over an extended spectral range. UV-Vis spectroscopy is usually applied to molecules or inorganic complexes in solution. The UV-Vis spectra have broad features that are of limited use for sample identification but are very useful for quantitative measurements. Since the UV-Vis range spans the range of human visual acuity of approximately 400 - 750 nm, UV-Vis spectroscopy is useful to characterise the absorption, transmission, and reflectivity of a variety of technologically important materials, such as pigments, coatings, windows, and filters. A UV-Vis system, with a high-end commercial UV-Vis spectrophotometer extending the measurable spectral range into the NIR region as far as 3.300 nm is desirable in each NFFA centre.

Photoluminescence (PL) is a non-contact, non-destructive method of probing the electronic structure of materials. Incident photons are directed onto a sample where they are absorbed (photo-excitation) and the sample will then release photons. Quantum mechanically PL can be described as an excitation to a higher energy state and then a return to a lower energy state accompanied by the emission of a photon. Available energy states and allowed transitions between states are determined by the rules of quantum mechanics. The period between absorption and emission is typically extremely short, in the order of 10 nanoseconds. PL is often used in the context of semiconductor devices where the energy of the incident photons is above the bandgap energy. PL spectra are useful to determine the bandgap energy, the composition of heterostructures, the impurity levels, the crystal quality, and to investigate recombination mechanisms. Several variations of photoluminescence exist: in the photoluminescence excitation (PLE) the spontaneous emission from the sample is detected at fixed photon energy and recorded as a function of the pump frequency; time-resolved photoluminescence (TRPL) is where you excite luminescence in a sample with a light pulse and then look at the decay in photoluminescence with respect to time. Due to the specific typical use of the tool, it might be located in the material growth instead of the metrology NFFA facility.

Electroluminescence (EL) is similar to photoluminescence, except that in EL the electron-hole recombination and the photon emission is obtained by current injection. EL is usually performed on the finished devices (such as LEDs and solar cells) since it needs a device structure to inject current. EL is determined by a number of factors such as the optical properties and physical structures of the optically active layers, the electrical properties of two conductive regions which are used for cathode and anode contacts, and the properties of the electrical contacts through which the electrical current is injected.

Femtosecond laser pulses, with durations of the order 10^{-15} s, provide a very powerful tool for examining atomic and molecular processes. In particular, their short temporal duration allows using pump-probe techniques to watch atomic and molecular processes in a time-resolved fashion. Furthermore, the high laser intensities that can be achieved with short laser pulses allows to study the interaction of atoms and molecules with strong laser fields, opening the door to a new class of physics where measurements on the attosecond (10^{-18} s) timescale become possible. *Ultra-fast laser spectroscopy* is used to examine the energy states and electron dynamics of molecule – light interaction. The main methods, to be considered in NFFA centres, are described below.

Ultra-Fast Transient Absorption (UFTA) is typical of pulse-probe experiments, where a pulsed laser is used to excite the electrons of molecules from their ground states to higher-energy excited states. A probing white light source is used to obtain an absorption spectrum of the compound at various times following its excitation. As the excited molecules absorb the second pulse, they are further excited to even higher states. After passing through the sample, the probing light beam reaches an avalanche photodiode array, and the data is processed to generate an absorption spectrum of the excited state. Since all the molecules in the sample will not undergo the same dynamics simultaneously, this experiment must be carried out many times, and the data must be averaged in order to generate spectra with accurate intensities and peaks. Unlike TCSPC, this technique can be carried out on non-fluorescent samples.

Time-Correlated Single Photon Counting (TCSPC) is used to analyze the relaxation of molecules from an excited state to a lower energy state. Since various molecules in a sample will emit photons at

different times following their simultaneous excitation, the decay must be thought of as having a certain rate rather than occurring at a specific time after excitation. By observing how long individual molecules take to emit their photons, and then combining all these data points, an intensity vs. time graph can be generated that displays the exponential decay curve typical to these processes. Since it is difficult to simultaneously monitor multiple molecule, individual excitation-relaxation events are recorded and then averaged to generate the curve. This is done by splitting a pulsed laser beam into two paths. A pulse along one path travels to a photomultiplier tube (PMT), while another path travels through the sample. The first pulse is detected by PMT and activates a time-to-amplitude converter (TAC) circuit. This circuit begins to build a charge on a capacitor which will only be discharged once the PMT sends another electrical pulse to the circuit. This electrical pulse comes after the second laser pulse excites the molecule to a higher energy state, and a photon is eventually emitted from a single molecule upon returning to its original state. Thus, the longer a molecule takes to emit a photon, the higher the voltage of the resulting pulse. The central concept of this method is that only a single photon is needed to discharge the capacitor.

Time-Resolved Photo-Electron Spectroscopy (TRPES) is similar to UFTA, the difference being that the second laser pulse ionizes the molecule. The kinetic energy of the electrons from this process are then detected, through various methods, e.g. energy mapping, time of flight measurements. The process is repeated many times, with different time delays between the probe pulse and the pump pulse. This builds up a picture of how the molecule relaxes over time. A variation of this method looks at the positive ions created in this process, and is called Time-Resolved Photo-Ion Spectroscopy (TRPIS).

c) Surface Analysis

Photoemission Electron Microscopy (PEEM) records electrons emitted from a sample in response to the absorption of ionising radiation. The electrons are accelerated by a strong electric field between the sample and the outer electrode of the objective lens, and the image is magnified hundred- or thousand-fold by a series of magnetic or electrostatic electron lenses. An electron-sensitive detector records the electron emission. Today a spatial resolution below 10 nm can be routinely achieved using UV light and of several 10 nm using X-rays. Aberration correction schemes are about to improve the resolution down to a few nanometres close to the physical limit of emission microscopes: the mean free path of low energy electrons [42].

Similar optics in combination with an electron gun instead of a photon source (see Fig. 15) are used in Low Energy Electron Microscopy (LEEM), where diffracted electron beams give a real-space image of the surface. The microscope is capable to monitor surface structure at nanometre scale in real time. As PEEM, LEEM is an analytical surface science technique that enables real-time observations of dynamic processes at surfaces. Such phenomena include: phase transitions, adsorption, reaction, segregation, thin film growth, strain relief, sublimation [43]. Lateral resolution spans from 8 to 20 nm, according to the model. Recently Aberration Corrected LEEM/PEEM systems have been proved to reach a resolution below 2 nm (see [44]). The presence of LEEM/PEEM systems in all NFFA centres is highly recommended. A single AC LEEM/PEEM system in one of the centres is advisable.

X-Ray Photoelectron Spectroscopy (XPS) is a widely used surface spectroscopy technique that can be used for quantitative measurements of the surface composition. X-rays impinge on the surface of a sample resulting in the ejection of electrons with varying energies. The electrons leaving the sample are detected by an electron spectrometer according to their respective kinetic energies. The technique gives information about elemental composition, the chemical state of elements, the lateral distribution and to some extent also depth distribution of elements. A simple XPS chamber is highly desirable in all NFFA

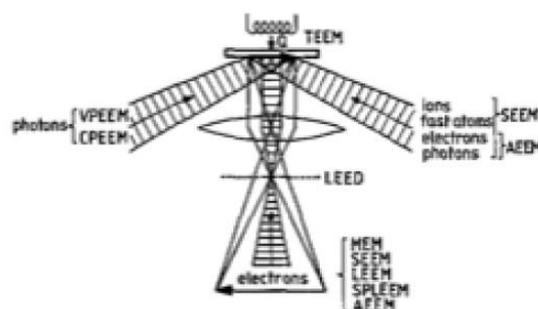


Fig. 15: Schematic Representation of a Low Energy Electron Microscope [43]

centres. Specialised set-ups capable of working at higher pressures (up to ~0,1 mbar [45]) or in liquids (provided the vapour pressure is low enough, as e.g. for ionic liquids [46,47]) have been tested in the last decade and could be included among the offer of NFFA centres specialised in high-pressure or liquid environment studies, for the optimal preparation of experiments at LSF's where beamlines operating in similar environments are accessible.

Auger Electron Spectroscopy (AES) is a popular technique for determining the elemental composition of the top few layers of a surface. It cannot detect hydrogen or helium, but is sensitive to all other elements, being most sensitive to the low atomic number elements. Underlying the spectroscopic technique is the Auger Effect, which is based on the analysis of energetic electrons emitted from an excited atom after a series of internal relaxation events. When coupled to a sputter gun AES can provide depth profile analysis.

d) Composition Analysis

Solid State Nuclear Magnetic Resonance (NMR) is a spectroscopic technique based on the reaction of certain atoms when they are immersed in a static magnetic field and exposed to a second oscillating magnetic field. Different materials have resonances at well established frequencies. Importantly, the position of the resonance frequency depends strongly on the crystallographic structure or chemical environment of the atoms. Thus, by analysing the different resonances present in a NMR spectrum, the structure of a given material can be inferred. Moreover, the intensity of the resonance lines depends on the magnetic properties of the materials, thus in certain cases some magnetic properties of materials can also be deduced from NMR. Solid State NMR is an extremely sensitive tool to determine the structure and chemical environments of materials.

Secondary Ion Mass Spectrometry (SIMS) In SIMS the sample is bombarded with an ion beam which results in the sputtering of ions from the sample (secondary ions). The secondary ions from the sample are analysed in a mass spectrometer according to their energies and mass-charge ratios. SIMS can be used for mass spectrometry of surfaces, e.g. nanostructured surfaces, fibres, biological tissues, interfaces and volumes for all elements and molecules up to 10.000 u. It can also be used for depth profiling, thus allowing 3D biological imaging. Measurements must be performed in vacuum.

Gas Chromatography (GC) is used to separate volatile components of a mixture. In a single step process, gas chromatography can separate a mixture into its individual components and simultaneously provide a quantitative estimate of each constituent. The instrumentation ranges from very costly and complex instruments to simple, inexpensive thin layer plates. [48] Both a GC with standard FID and TCD detectors for analysis of low-mass products and a Gas Mass GC (GC/MS - combination of GC and Mass Spectrometry) for high-mass elements are suggested as support tools in most NFFA centres.

Magnetic Characterisation Section:

Magneto-optic Kerr effect magnetometry (MOKE) This technique allows sensing the instantaneous magnetic state of a material by focusing polarised light onto its surface. The basic principle behind MOKE is that the strength of the magnetisation of a given material affects the change in the angle of polarisation of the light that is reflected off its surface. Since the laser spot can be focused down to a few microns, the magnetic properties (e.g. coercivity, squareness ratio) of single micrometer or submicrometre structures can be evaluated. Note that although this technique can be very sensitive, no quantitative measure of the magnetisation is provided. Nevertheless, from the shape of the hysteresis loop, both static and dynamic magnetisation reversal processes can be investigated in detail. Longitudinal and polar configurations are used routinely to detect the in-plane or the out-of-plane magnetisation components, respectively. This technique is well suited to characterise either flat or micron- and submicron-patterned films.

Superconducting quantum interference design (SQUID) This technique is extremely powerful to measure the moment versus applied magnetic field (e.g. hysteresis loops) or moment versus temperature curves of magnetic nanomaterials in a broad temperature range. Its sensitivity is very high, therefore a small amount of material is enough to ensure reliable signals. In addition to hysteresis loops,

SQUID measurements also allow precise determination of the Curie, Néel or blocking temperatures of ferromagnetic, antiferromagnetic or superparamagnetic nanoparticles.

Vibrating sample magnetometer (VSM) Similar to SQUID, VMS is very suitable to measure hysteresis loops or critical temperatures (Curie, Néel, blocking) of different types of magnetic materials. Some of the advantages of VMS over SQUID magnetometry are: the possibility of carrying out magnetic measurements up to high temperatures (SQUID is typically limited to 400 K or so) and the speed of measurement (the field sweep rate in VSM is faster than in SQUID). Conversely, VMS is less precise in magnetisation determination than SQUID.

Ac-susceptibility measures the response of a material in which an ac magnetic field is applied. This technique is particularly sensitive to diverse magnetic states and magnetic transitions. Moreover, in contrast to dc magnetic measurements, some information about the magnetisation dynamics can be obtained. The results are usually given in in-phase and out-of-phase signals, which, given their different physical origin, allow for a more in depth characterisation of the material than similar dc-approaches. This technique allows inferring the nature of the magnetic materials present in a sample.

Thermal and Mechanical Characterisation Section:

Thermogravimetry (TG) is a technique by which the weight of a substance, in an environment heated or a cooled controlled rate is recorded as a function of time or temperature. The equipment consists of a precision balance, a furnace with a programming facility, a reaction chamber and a suitable recording system. The assembly has to be capable of continuously registering any weight changes in the test sample whilst the latter is being heat-treated. TG gives absolute changes in sample weight so that the calculated extent of reaction is not affected by the heating rate used, although the start and finish temperatures are a function of heating rate because of kinetic barriers. TGA is commonly employed in research and testing to determine characteristics of materials, to determine degradation temperatures, absorbed moisture content of materials, the level of inorganic and organic components in materials, decomposition points of explosives, and solvent residues. It is also often used to estimate the corrosion kinetics in high temperature oxidation. Evolved gases from the sample can be analysed for example in a coupled mass spectrometer.

Differential scanning calorimetry: Nano (DSC) is a technique for measuring the energy necessary to establish a nearly zero temperature difference between a substance and an inert reference material, as the two specimens are subjected to identical temperature regimes in an environment heated or cooled at a controlled rate.

There are two types of DSC systems in common use: power-compensation and heat-flux. In power compensation DSC the temperatures of the sample and reference are controlled independently using separate, identical furnaces. The temperatures of the sample and reference are made identical by varying the power input to the two furnaces; the energy required to do this is a measure of the enthalpy or heat capacity changes in the sample relative to the reference. In heat flux DSC, the sample and reference are connected by a low-resistance heat flow path (a metal disc). The assembly is enclosed in a single furnace. Enthalpy or heat capacity changes in the sample cause a difference in its temperature relative to the reference; the resulting heat flow is small compared with that in differential thermal analysis (DTA) because the sample and reference are in good thermal contact.

The main application of DSC is in studying phase transitions, such as melting, glass transitions, or exothermic decompositions. These transitions involve energy changes or heat capacity changes that can be detected by DSC with great sensitivity

Nanoindentation This technique is used to measure the mechanical properties (hardness, Young's modulus) of thin films and bulky materials. The measurement itself consists of indenting a diamond tip (usually with pyramidal geometry) into the material surface. During this process, both the load and the penetration depth are continuously recorded. Therefore, this method is also suitable to measure dynamic mechanical processes (such as anelasticity or viscoelastic effects).

Acoustic measurements of elastic constants (Pulse-echo technique) This is a method used to determine the elastic constants of materials (shear modulus, bulk modulus, Young's modulus and

Poisson's ratio). The method consists of measuring the speed of propagation of ultrasound longitudinal and transversal waves as they are emitted through the cross section of a piece of material. Although this is a bulk characterisation technique, it can be employed to measure the elastic constants of nanostructured (i.e. with crystallite size down to nanoscale) of bulk specimens.

Transport Characterisation Section:

Transport properties vs. temperature. Transport properties are generally investigated as a function of temperature. The major part of the experimental setup is in the electronic which should be able to cover an as broad as possible range of samples. For low-impedance samples (e.g. metal, superconductors, etc), a nano-voltmeter and a constant current source are generally used for DC characterisation, while AC-regime of transport properties (e.g. noise properties) are generally investigated by using a lock-in generator and/or a network analyser. However, high-impedance samples (e.g. insulators, semiconductors, etc) demand for a constant-voltage source combined with a low-current meter (i.e. pico-ammeter) and/or capacitance meters. Depending on the specific temperature range of investigation, different equipments can be used. Dipsticks inserted in dewar containing cryogenic liquids (e.g. helium, nitrogen) can provide a quick investigation of the transport properties of the samples. However, higher control on the temperature (stabilisation at fixed temperature, cooling/warming ramps, etc.) can be obtained by using cryocooler (e.g. close-circuit cryogen-free cryostat, helium gas flux cryostat, etc). Both dipsticks and cryocoolers should be available in all the NFFA centres.

Transport properties in magnetic field. Transport properties can be also investigated as a function of additional external phenomena (e.g. magnetic field, electric field, laser irradiation, etc.). In particular the presence of a external magnetic field is usually supplied by using conventional (up to about 1 Tesla) and/or superconducting magnets (higher than 1 Tesla), surrounding and/or in proximity of the sample under test.

Commercially available Physical Properties Measurement Systems (PPMSs), which allow all the described investigations, should be considered as a basic equipment for transport characterisation of the samples to be inserted in all the NFFA centres. Dedicated equipments for quantistic measurements (low noise and low current) with criostats working down to 10 mK and 300 mK will be introduced as advanced tools in few selected NFFA centres.

APPENDIX B – Standard Organisations

Following is a list of organisations worldwide involved in standards for nanotechnology. The list includes organisations developing formal standards (International Standard organisation, National Standard Bodies and regional standard organisation) organisations developing informal standards, as SDOs (standard developing organisations) and organisations expert in the field of nanometrology.

International Standard Organisations

- International Organisation for Standardization (ISO) – TC 229, Nanotechnologies.
Website: http://www.iso.org/iso/iso_technical_committee?commid=381983
- International Electrotechnical Commission (IEC) - TC 113, Nanotechnology standardization for electrical and electronic products and systems.
Website: http://www.iec.ch/dyn/www/f?p=102:7:0:::FSP_ORG_ID:1315

National Standard Bodies

Following a (partial) list of NSBs having specific Technical Committees or Commissions on nanotechnology. The complete list is available on the ISO TC 229 website.

- American National Standards Institute's Nanotechnology Standards Panel (ANSI-NSP).
Website: http://www.ansi.org/standards_activities/standards_boards_panels/nsp/overview.aspx?men
- Japan Industrial Standards Committee - Council on Nanotechnology Standards in Japan (JISC/CNSJ)
Website: <http://www.jisc.go.jp/eng/pi/index.html>
- Standardization Administration of China - Committee on Nanotechnology (SAC/TC279).
Website: <http://www.sac.gov.cn/>
- Standards Council of Canada – Canadian Advisory Committee for ISO TC229.
Website: <http://www.scc.ca/>
- Korean Agency for Technology and Standards (KATS) - Materials and Nanotechnology. Standards Division.
Website: <http://www.kats.go.kr/>

At European level:

- British Standards Institute – Committee for Nanotechnologies (BSI –NTI/1).
Website: <http://www.bsigroup.com/en/Standards-and-Publications/Industry-Sectors/Nanotechnologies/BSI-Committee-for-Nanotechnologies/>
- DIN/DKE Deutsches institut fur Normung - Steering Committee on Nanotechnology.
Website: <http://www.dke.de/dke/>
- Association Française de Normalisation – Nanotechnologies (AFNOR- X457).
Website : <http://www.afnor.fr/portail.asp>
- UNI – U22 - Italian Organisation for Standardisation CT U22-Nanotechnologies

Regional Standard Organisations

- CEN TC 352 . Nanotechnologies.
Website: <http://www.cen.eu/cenorm/sectors/sectors/nanotechnologies/index.asp>
- CENELEC – The European Committee for Electrotechnical Standardisation.
- ETSI - European Telecommunications Standards Institute

Standards developing organisations (SDOs) 60

Following a (partial) list of SDOs most involved in nanotechnology:

- ASTM Committee on Nanotechnology (ASTM - E56).
Website: <http://www.astm.org/COMMIT/COMMITTEE/E56.htm>
- IEEE Nanotechnology council.

NFFA - Nanoscience Foundries and Fine Analysis

Website: <http://grouper.ieee.org/groups/nano/>

- SEMI - Semiconductor Equipment and Materials International.
Website: <http://www.semi.org/>
- VAMAS (Versailles project on Advanced Materials and Standards).
Website: <http://www.vamas.org/>

Expert organisations in the field of nanometrology, nanotechnology, technology transfer and specialised training

- EUSPEN (European society for precision engineering and nanotechnology).
Website: <http://www.euspen.eu/page338/About-Us/Euspen>
- NPL (National Physical Laboratory, UK).
Website: www.npl.co.uk
- ASCAMM Fundacio Privada, Spain.
Website: <http://www.ascamm.com/ca/index.php>
- SP Technical Research of Sweden.
Website: <http://www.sp.se/sv/Sidor/default.aspx>
- JRC (European Commission, Joint Research Center, Institute for Materials and Measurements).
Website: <http://irmm.jrc.ec.europa.eu/html/homepage.htm>
- PTB (Physikalisch Technische Bundesanstalt).
Website: www.ptb.de

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- ¹⁷ <http://www.ntmdt-tips.com/catalog/gratings/tdg/products/TDG01.html>
- ¹⁸ http://www.ntmdt-tips.com/catalog/gratings/afm_cal/products/TGT1.html
- ¹⁹ <http://www.bruckerfmprobes.com/c-232-test-samples.aspx?pagenum=1>
- ²⁰ http://www.ntmdt-tips.com/catalog/gratings/snom_gr/products/SNG01.html
- ²¹ http://nanoprobes.aist-nt.com/index.php?main_page=product_info&cPath=142&products_id=240
- ²² http://www.rm-certificates.bam.de/de/rm-certificates_media/rm_cert_layer_and_surface/bam_l200repe.pdf
- ²³ http://www.bam.de/pdf/service/referenzmaterialien/zertifikate/layered_materials/bam_l002.pdf
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