

Scanning Probe Microscopy: Characterization,  
Nanofabrication and Device Application of  
Functional Materials

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# Scanning Probe Microscopy: Characterization, Nanofabrication and Device Application of Functional Materials

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## PREFACE

Today, a wide range of analytical techniques can be used for materials research. The most commonly used high-resolution surface analysis techniques are Scanning Electron Microscopy (SEM) and Scanning Probe Microscopy (SPM). Although both techniques resolve surface structure down to the nanometer scale, the different image formation mechanisms result in different types of information about the structure of the surface, making these techniques complementary. In SEM, an electron beam, guided by a complex array of lenses, interacts with the sample and electrons are emitted from the sample, either as back-scattered or secondary electrons. Secondary electron emission is commonly used for surface morphology analysis. The first SEM equipment was constructed between 1938 and 1942. Improvements in resolution and the development of several types of detectors for local compositional analysis (X-rays, Auger electrons, backscattered electrons, cathodoluminescence) took place since then. Compared to electron microscopy, SPM techniques are quite new. Scanning Tunneling Microscopy (STM), the earliest of the SPM techniques, was invented in 1981 at IBM Zurich Research Laboratory by G. Binnig and H. Rohrer. STM was the first instrument to generate three dimensional real-space images of surfaces with atomic resolution. This invention earned them the Nobel Prize in Physics in 1986. In STM a sharp conducting tip, with a bias voltage applied between the tip and the sample, scans the surface of the sample. The scanning motion is at the angstrom level and the tip does not contact the sample. The resulting tunnelling current, exponentially dependent on the tip – sample spacing, is the signal used to create the STM image. Since 1981, a large family of SPM related techniques, based on various types of interactions between the tip and the sample, has been developed. It has been demonstrated that the SPM approach allows manipulation of single atoms and molecules. Various SPM techniques such as atomic force microscopy (AFM), magnetic force microscopy (MFM), electrostatic force microscopy (EFM), scanning capacitance microscopy (SCM), near-field scanning optical microscopy (NSOM) and others were proved to be capable of measuring the local physical properties of materials with nanoscale resolution. As a consequence, presently there is an explosion in the application of SPM techniques in a wide spectrum of fields of science, ranging from condensed matter physics, chemistry and materials science, to medicine and biology. Currently, SPM is widely used for nanoscale characterisation of materials by using mechanical, electrical, magnetic, optical and chemical interactions between the probing tip and the surface.

Compared to electron microscopy techniques (SEM, TEM, HRTEM, etc), SPM is a low cost analytical method in terms of equipment, maintenance, accessories and sample preparation. In addition, it can operate in ambient environment forbidden to electron microscopy. Consequently, it is expected that a growing number of university research groups and R&D industry divisions will acquire such equipment for research, quality control and fabrication. At the same time, the role of SPM in the field of nanotechnology is also growing in importance. The continuous need for the miniaturisation of electronic devices, with improved speed and functionality, has been a



constant driving force pushing towards the development and manipulation of nanoscale devices. However further scaling of digital electronic devices necessitates fabrication and application of materials with nanoscale features. In this sense, SPM is becoming an indispensable tool, playing a key role in nanoscience and nanotechnology.

One of most rapidly evolving, yet relatively unknown, fields of material science and of functional materials is the field of ferroelectric thin films. These materials possess a unique set of physical properties, such as switchable polarization, piezo- and pyroelectricity and high nonlinear optical activity, which make them extremely attractive for a number of applications. Over the last 10 years there have been serious efforts to develop ferroelectric memories, which combine nonvolatility with high-speed access, almost unlimited endurance and extreme radiation hardness. Due to these significant advantages of ferroelectric memories, it is expected that they will continue to replace other types of nonvolatile memory systems in many applications. In addition, ferroelectric materials can be used in a variety of other devices that exploit their unique properties, such as piezoelectric transducers and actuators, infrared sensors, optical switches and computer displays. Recent advances in the processing of high quality ferroelectric films resulted in development of 4 Mb nonvolatile ferroelectric random access memories (NVFRAMs) at Samsung and Matsushita. However, the tremendous potential of ferroelectric films is far from being realized, as further developments in this area are hindered not only by the integration issues related to the present state of the NVFRAM technology, but also by a lack of fundamental knowledge related to reliability, performance and scaling of ferroelectric devices. Integration of ferroelectric thin films into Gigabit memory devices requires substantial improvement in the understanding of the properties and device physics of these materials, and this in turn requires the implementation of new tools suitable for *in situ* testing of ferroelectric nanostructures. One of the most promising approaches is based on using scanning probe techniques. Recently, SFM has been successfully applied for nanoscale characterization of ferroelectric thin films. Several qualitative experiments demonstrating the capabilities of SFM in controlling domains as small as 20-50 nm in diameter have already been performed. SFM was also used for nanoscale studies of degradation effects, such as ferroelectric fatigue and retention loss. Another very important branch of applications is related to SPM-based electrical characterization. As the characteristic dimensions of electronic devices continue to shrink, the ability to characterize their electronic properties at the nanometer scale has come to be of outstanding importance. Scanning probe microscopy has opened new opportunities to measure semiconductor electronic properties with unprecedented spatial resolution. For example, scanning spreading resistance microscopy (SSRM), and scanning capacitance microscopy (SCM) have already demonstrated device measurements with  $\sim 2$  and 10-20 nm spatial resolution respectively. Kelvin probe force microscopy (KPFM) has been used for measuring electrostatic forces and electric potential distribution and has found many diverse applications in recent years. In the area of functional molecular materials, SPM is also proving to be an invaluable tool. It is being used as a probe to contact molecular structures in order to characterize their electrical properties, as a manipulator to assemble nanoparticles and nanotubes into simple devices, and as a tool to pattern molecular nanostructures.

To contribute to the development in these technological and scientific fields, there is a need to “collect” and disseminate this new and growing knowledge at the cross disciplinary level. The NATO Advanced Study Institute (ASI) on “Scanning Probe Microscopy: Characterization, nanofabrication and device application of functional materials,” held in Albufeira, Algarve, Portugal from the 1<sup>st</sup> to the 13<sup>th</sup> October 2002, by bringing together highly expertise researchers from the nanoscale techniques and functional materials disciplines, by presenting the fundamentals, technological advances and the needs for further developments in their respective fields and by stimulating active discussions, contributed to foster new scientific contacts and to develop new ideas in the field.

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Aveiro, Portugal, June 2004

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## ON THE NATO ASI

The main objective of this ASI was to disseminate knowledge concerning the new and emerging applications of SPM to the field of material science, especially in the areas of characterisation, device application and nanofabrication of functional materials. Timing of the proposed meeting was extremely appropriate, as the subject reflects the growing importance of SPM as a key tool for further development of nanoscale science and technology. Rapid progress in the field of SPM and functional materials demands energetic efforts on organising meetings to contribute to the scientific education of a new generation of engineers and scientists with new expertise and deeper understanding of nanoscale device physics.

The ASI was attended by 83 researchers, post doctoral and students from 24 countries, including: Austria, Belgium, Canada, Czech Republic, France, Germany, Greece, Israel, Italy, Japan, Korea, Latvia, Portugal, Poland, Romania, Russia, Spain, Slovenia, Sweden, Switzerland, Turkey, Ukraine, United Kingdom and United States of America.

To achieve the proposed objectives the scientific programme of the ASI comprised three main parts: Part I - SPM Techniques and Functional Materials, Part II – SPM in Functional Materials: Characterization and Part III – SPM in Functional Materials: Nanofabrication and Device Application. Introduction, development and the expanding capabilities of SPM, as a powerful nanoscience technique, were addressed in Part I. To complete Part I, the scientific and technological importance of the fundamental knowledge of structure / properties / applications relationships of functional materials were presented and discussed. In Part II recent progress in nanoscale SPM characterization of advanced functional materials, namely semiconductors, magnetics, dielectrics, ferroelectrics, were covered. The newest advances on fabrication of nanostructures and the links between nanofabrication and nanoscale characterization were discussed in Part III.

During 10 working days these topics were systematically presented and treated in depth by an interdisciplinary team of leading scientists in lecture format. This tutorial activity was complemented by rump sessions on related subjects, namely: (i) Comparison and direction of SPM methods and (ii) Fabrication: future of the bottom up approach. In addition, to stimulate scientific contacts and discussion, poster sessions and short presentations by the participants were held on their own scientific activities in the field. At these discussions the participants were motivated to expand themselves, to be innovative in the field of characterisation and fabrication with SPM, and to present their own innovative ideas on the topic.

The theme for this ASI has its own scientific value. Its uniqueness is in the combination of the fundamental nanoscale research with the progress in fabrication of realistic nanodevices. In addition, it developed new educational advances. By bringing together leading researchers from the material science and SPM communities, relevant information and experience was conveyed that allowed scientists to learn more about the actual developments and future trends of each field. Contacts among the scientists

were fostered and in this way contributed to the development of this new and technological important interdisciplinary field of science. For PhD students and post-doc scientists, participation in this meeting led to significant improvement in their knowledge of the basic properties of functional materials, as well as in application of SPM techniques. With SPM becoming a ‘must-know’ technique in many scientific disciplines, this meeting helped to improve the qualification level of university graduates from different countries and provided manpower with new expertise in the field of nanotechnology and SPM.

## **ON THE BOOK**

This book is the output of the ASI NATO meeting on Scanning Probe Microscopy: Characterization, nanofabrication and device application of functional materials. The book content reflects the scientific content of the school by itself presenting the main lectures that were given, and some of the participants works that were also presented and discussed during the school. The book is organised in four parts. Part I, Fundamentals of Functional Materials, is an introductory chapter that addresses the general properties of functional materials, highlights some of the unsolved problems of functional materials and reports the progress in silicon technology from the perspective of scaling to submicron devices and the expected performance at the end of the silicon scaling era. Part II, Fundamentals of Scanning Probe Techniques, presents the principles and basics of various SPM techniques, such as near-field optical microscopy, kelvin probe force microscopy and non-contact – AFM, showing the capacity of the techniques to measure the local physical properties of materials with nanoscale resolution. The application of SPM techniques to the characterization of specified functional materials such as piezoelectric ceramics and ferroelectric materials is discussed in Part III, Application of Scanning Techniques to Functional Materials, which also presents the utilization of such techniques to the fabrication of some nano electronic devices. Part IV, Contributed papers, includes some of the R&D work related to the utilization of SPM techniques to functional materials, as presented by the participants.

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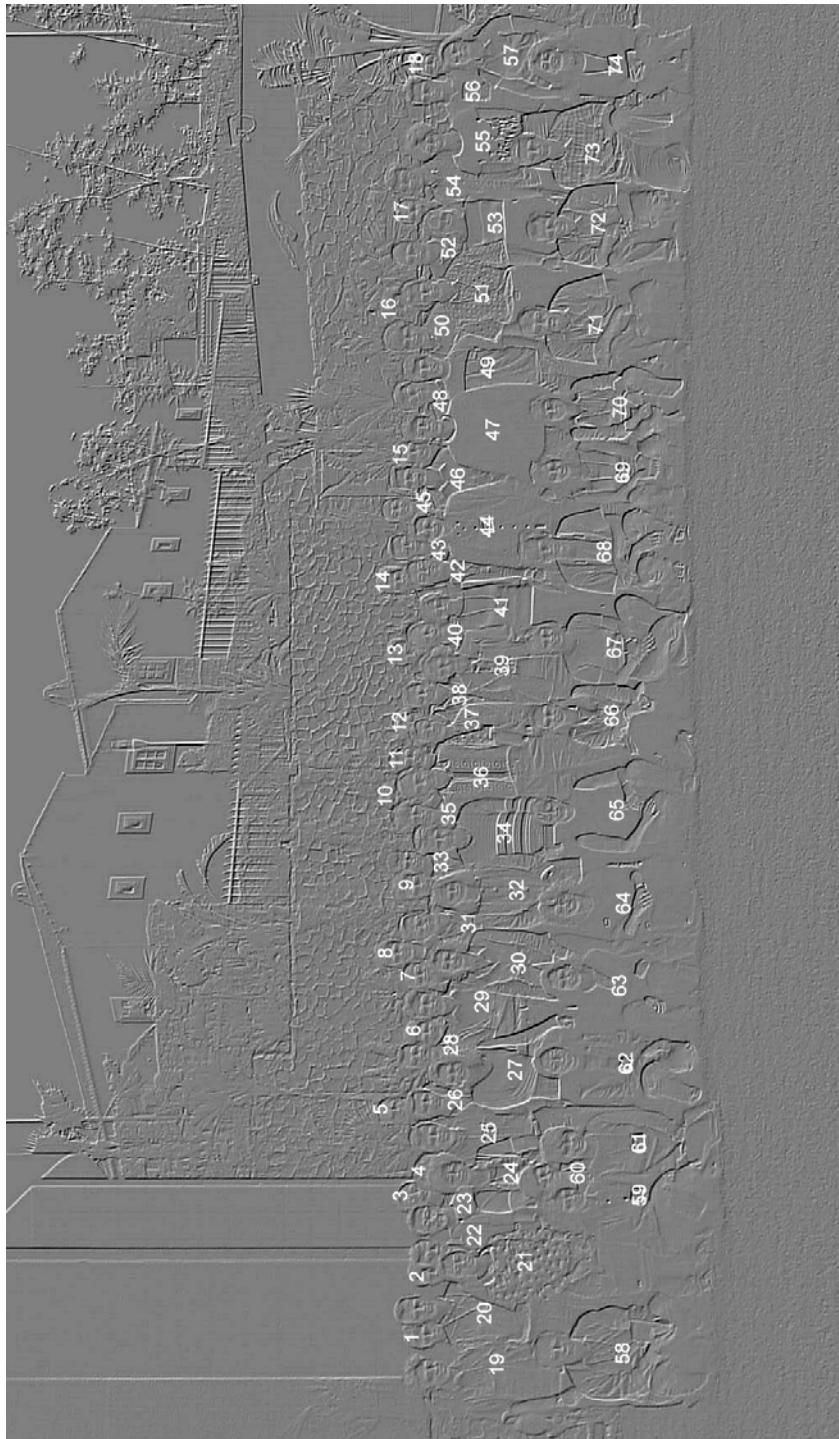
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One of the directors (P. M. Vilarinho) expresses her gratitude to Angus Kingon. The idea of organising such an event came from the discussions had with Angus Kingon during her stay at North Carolina State University (NCSU), USA, on a sabbatical leave. The recognition that Scanning Probe Microscopy was an emerging technique, namely in the field of Functional Materials and that was not addressed in a systematic way, combining expertises coming from different fields of materials science, such as physics, processing and device construction, was the embryo of this ASI. Alexei Gruverman is also thanked for his valuable contribution and help in finding the lecturers and for the critical analysis of the proposal for such ASI.









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# **Part I – Fundamentals of Functional Materials**

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