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Petr Klapetek

QUANTITATIVE DATA PROCESSING IN SCANNING PROBE MICROSCOPY

SPM Applications for Nanometrology

Micro & Nano Technologies Series

Quantitative Data Processing in Scanning Probe Microscopy

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Preface

The first idea of writing this book came when giving a lecture series on quantitative scanning probe microscopy (SPM) for chemists, which reminded me of the three big gaps: first, between what a normal user needs and expects from SPM and what his/her instrument can offer; second, between what the instrument manufacturer or dealer promises and what the reality is, and finally, gap between the outputs the top scientists on the field can reach (doing nothing else for their whole life) and what can be a standard result of someone that wants to spend his/her life doing something different (even if it is only a different field of science). Even if we could find a similar situation also in other fields of science, I feel that in SPM it is extremely pronounced and the differences are often very hard to be understood. The book wants to bridge some of the gaps, having in mind that this is probably an everlasting process and only a little bit will be done even with a big effort.

Being not very satisfied with books having every chapter written by different authors as a self-containing scientific article, I tried to concentrate on the creation of a more compact piece of text that would be as consistent as possible, which, in my feeling, means having a single author participating on all the chapters. However, this could not be done without large support of all the co-authors that are much better experts in particular SPM fields than me, and I need to thank them all for supporting this book.

I would like to thank Andrew Yacoot for many important suggestions after reading the first version of manuscript and Anna Charvátová Campbell and Miroslav Valtr for carefully reading the final manuscript and for providing some of the up to now unpublished data. For discussion on specific chapters and problems, providing interesting samples and supportive measurements I would like to acknowledge also Jindřich Bílek, Jiří Man, Jakub Zálešák, Jana Kotková and Jakub Holáň.

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As most of the book was written during evenings and nights, it would be not possible without the large support of my family—my wife Eva and sons Jan, Vojta and Myšour.

Any web content referenced in this book can be accessed on the companion website at this link: http://booksite.elsevier.com/9781455730582.

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CHAPTER

Motivation

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1.1 WHY "QUANTITATIVE" SCANNING PROBE MICROSCOPY?

Despite its tremendous advancement in the last 20 years, scanning probe microscopy (SPM) is still not understood as a really quantitative experimental technique. Besides dimensional SPM measurements that are regularly accepted as providing accurate results, there is an increasing number of other physical quantities measuring and mapping modes that are producing more or less qualitative results, with no firm relation to any absolute value. The reason is very simple. The nanoscale tip-sample interaction is very complex, containing many different components, and some of them are still not properly understood. Moreover, the geometry and composition of both tip and sample are not known (we measure only result of their mutual interaction), which makes all the models even more complicated. The more advanced SPM techniques we use, the farther they are from being a metrology tool.

As most of the SPM users have no access to a specialized metrology SPM system, their ability of converting results to quantitative ones (i.e. accurate and traceable, with known uncertainty) is limited to a proper instrument calibration, a good measurement strategy and a detailed analysis of obtained results. Proper understanding of basic physical phenomena taking place between tip and sample is crucial for this task. Similarly, knowledge of all typical artifacts, accuracy bottlenecks and use of artifact detection, and removal techniques should be a daily bread for an SPM researcher.

The aim of this book is to provide the reader with a reference for a quantitative SPM analysis in practical situations, namely when using commercial instruments. We provide a description of the basic ideas necessary for the quantitative understanding of different SPM techniques operation, including dimensional, mechanical, electrical, magnetic, thermal, and optical modes. We discuss the physical model of the tip-sample interaction for each particular case and list basic calibration and traceability strategies. The influence of different tip and sample geometry or their material properties on quantitative measurements is analyzed. Techniques for modeling realistic data and processing the measurement results are reviewed and their practical application to user data is described. Whenever possible, publicly available software tools are reviewed from the point of their applicability and accuracy.

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This book would like to address the typical questions of a newcomer or a casual user. Scientific literature is full of excellent results obtained using SPM, like individual atom chemical identification [1] or building fascinating nanostructures. When this is compared to the possibilities of a standard commercial instrument, even a new and really expensive one, the reader can get significantly disappointed. The data that are produced by the instrument are at first sight only colorful images. Most of the measured quantities have unphysical units like nanoamperes for force or volts for temperature. If there is some calibration of quantitative data treatment built-in in the instrument, it is limited to the very basic approaches only, even those proved in the literature as very poor ones. In this book we would like to show what needs to be done to get maximum of data that a "standard" commercial instrument produces and which directions to take to improve accuracy and reliability of such results.

For illustration, we can sketch several sample questions that this book would like to address:

- How can I calibrate my SPM to get accurate results?
- How precisely can I measure dimensions of nano-objects?
- How can I interpret force-distance curves?
- What spatial resolution can I expect in thermal/magnetic/electric/optical measurements and how to evaluate it?
- Can I resolve different chemical species in ambient SPM measurements?
- Can I measure local material refractive index using SPM?

And what does the book not address? We focus on data processing and analysis; all the information related to the measurement process itself is limited to its influence on the obtained data. Technical discussion on how microscopes are built is therefore only short and we do not describe most of the modes that need special equipment, which is not available in any commercial instruments, even if these could lead to challenging results. If the use of some additional equipment is suggested, it is usually outside of the microscope, used only for obtaining some additional information (e.g. calibration of the probes). The majority of this text is related to ambient measurements, even if most of the approaches are valid also in vacuum conditions. We do not discuss special issues related to ultrahigh vacuum (UHV) and low temperature SPMs as this is still a statistically minor field of SPM use. We also limit the description of numerical models and tools to basic understanding and use. For a more detailed description, reader should follow books on concrete numerical methods, that are also numerous.

1.1.1 Book Organization

When referring to a scanning probe microscope, some concrete instrument comes usually to mind, typically the one that we own. We treat it as a single instrument. But if we start discussing all the interactions, probe types, and quantities that can be measured we see that we have many different instruments mixed together, linked only by the basic concept—scanning with a small probe in proximity to the surface.

If we want to discuss quantitative aspects of all these distinct instruments, the easiest way is to go method by method, always showing the key phenomena, necessary instrumentation, metrology issues, and related data processing steps. This approach is followed in most of the chapters.

The book starts with a description of what is common to all the methods. In this and the next chapter we discuss the very basics of SPM that are probably known to the reader—more to reference them than to bring some novel information. We review briefly the key instrumentation principles and basic phenomena behind the measurement. We review basics of metrology in order to help following the discussions on quantitative data processing. We also describe how the SPM is related to other analytical methods.

The following three chapters describe more in detail the concepts that are common to all SPM techniques. The third chapter focuses on basic data acquisition and storage models in SPM, including a discussion of factors limiting SPM precision during the measurement, like drift or noise. The fourth chapter describes basics of data visualization, correction, and processing. The fifth chapter refers to dimensional measurements using inter-atomic force-based feedback that is common to most of the discussed techniques. It also covers major issues of uncertainty analysis in SPM based dimensional measurements. As dimensional measurements are the key part of every more complex SPM technique, this analysis is valid also in all the following chapters.

The next eight chapters are related to different analytical techniques available in SPM. They have almost the same basic structure as shown below.

First, key phenomena that can be measured and that are used as a source of the analytical information are discussed (normal and lateral forces, electric field, thermal transport, etc.). This should cover the basic questions of *why* to use these techniques and *what* we can measure with them. Theoretical analysis starts from very simple models and even very basic concepts to make reading easier for SPM users from non-physical fields. More complex approaches are discussed briefly as well, however their rigorous development is often left to referred monographs as their detailed description would be outside the scope and size of this book. Theoretical descriptions often include also basic information about numerical modeling techniques, that can be helpful in the interpretation of the data. Simply usable software is often recommended for the practical demonstration of the efficiency of the numerical methods even if the results and general remarks are valid for all the other software packages as well.

The instrumentation necessary for each particular analytical method is reviewed, to show *how* the discussed quantities can be measured. This section is namely focusing on possibilities how to get the method calibrated and traceable. As the book is namely concentrated on data processing and interpretation, we do not discuss special modes and techniques that need special equipment (usually custom built) and would be probably not available for the reader.

The most important section on data interpretation follows, showing how both the analytical models and numerical techniques can be used for better understanding of what was measured. Typical error sources and artifacts are reviewed and ways how to detect and suppress them are discussed. This covers the main question discussed through the whole book: *how precise* SPM can be for different quantities measurements. The chapter is then finished by examples of what the reader could practically try, often using attached software, and with some tips for making the measurement as quantitative as possible. Recommended further reading is listed as well as this book does certainly not contain all the information that the user would need when going deeper.

There are two special chapters at the end of the book. The first one (Chapter 13) lists all the available data—measurement samples and software available on the associated web content. The second one (Chapter 14) provides some more technical details about how the different numerical methods work and what they are based on.

1.1.2 Available Numerical Techniques

While reading the book, you might ask why there is such an emphasis on numerical modeling techniques? It is clear that the availability of any numerical method cannot substitute the understanding of physics behind the problem. Numerical modeling could be understood as no better than the "second best" approach. But for many SPM related phenomena the analytical approach needs so many assumptions that it cannot cover the tip-sample interaction in whole, including all effects seen in the real world (e.g. roughness or material inhomogeneities). That is why we need numerical modeling.

Numerical modeling tools are very popular nowadays in physics, as the large computational power and ubiquity of computers makes their use very simple. Numerical methods that required supercomputers some 10 years ago are now accessible within a few minutes or hours on a regular computer and with the extreme market demands for innovations we can expect them to run even on a toaster 10 years from now. The increasing market need for consumption is from this point certainly a large benefit for computational physicists. A large number of both commercial and non-commercial software tools has been created for different physics areas and applications. Even if they can help a lot in SPM data analysis, there is a significant barrier for a beginner to use them. Some of the numerical packages are intended for general purposes, but to set up a simulation of a concrete problem needs many hours of preparation. Others are optimized for the particular field of application of their authors and application for any other field is almost impossible. In this book several numerical methods and software packages are discussed in detail for illustration of data processing concepts. It should be said that these were selected on the basis of their simplicity (and author's knowledge) and the reader is encouraged to try any possible alternative that would fit his/her needs.

For practical purposes the numerical techniques description is scattered throughout the book, discussing each particular technique in the chapter when it has some application. Here we summarize the techniques discussed in the book:

- Classical molecular dynamics and quantum molecular dynamics based on Density Functional Theory (DFT) are discussed in Chapter 6 when forces