

# Introduction to Focused Ion Beam Nanometrology

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# Introduction to Focused Ion Beam Nanometrology

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*To Lúcia, and my parents.*



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# Preface

When I was invited by Institute of Physics Publishing to write this book a little over a year ago, I went through a process I am sure is common to many writers. After a week of deliberation, I said yes. Some 24 hours later I wondered what was going through my mind when I had said yes. Why on the Earth did I agree to do it? I thought my days of writing large bodies of work started and stopped almost twenty years ago with completion of my PhD thesis. Compiling list of references, sourcing and preparing figures, and sitting for hours on end rewriting the same paragraph over and over and not being happy with it; I must have been mad. However, after one or two false starts I finally got going, and must say, as time progressed have slowly enjoyed the process more and more. It has been made considerably easier by discussions with my colleagues and the manufacturers, who have helped enormously and provided valuable input to this text. Special thanks go to Helen Jones, at NPL, Raphaela Scharfschwerdt at FEI and Diane Stewart at Zeiss.

I would like to thank all my colleagues at the the National Physical Laboratory who over the last ten years have presented me with many scientific challenges, often in areas where I had little or no knowledge. If ever there was an illustration of the power and diversity of the focused ion beam (FIB) it is that in those ten years I have worked with people in almost every division of the laboratory, from quantum metrology, through material science, to dimensional metrology and analytical science.

Indeed, conveying this diversity and the incredible usefulness of these instruments has been my main purpose in writing this book. Firstly, I would like to introduce this wonderful instrument, the FIB, to new and potential users, and to show existing users the additional areas where their instruments may find use. Very few instruments offer such a huge range of diverse applications. Secondly, such is the speed of development of the instruments and associated techniques that although previous (and extremely thorough) texts exist in the subject, they do not cover the latest developments involving new ion sources and methods. Finally, I would like to make the reader aware that some of these methods are not foolproof, that errors exist and are often overlooked. It is only by understanding the limitations of these processes that we truly learn how useful they are. This is not to say that the FIB is a poor technique by any means. Many of the topics covered in this book simply could not be carried out at all without the FIB instrument and many will get even better in terms of both throughput and accuracy as our understanding of them gets better, and creative people develop the instruments and methods further.

David C Cox

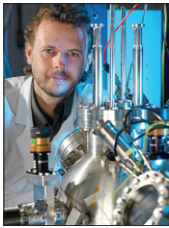
# Acknowledgements

I would like to thank my many colleagues from the National Physical Laboratory, in particular those who provided material for this book, proof read drafts, or engaged in useful discussions about its content. I would also like to thank those at the University of Surrey who have done similar. Thanks also to my friends at NTT basic research laboratory for providing such a stimulating place to carry out research and allowing me to help you in establishing your new FIB facility. I would also like to thank FEI and Zeiss for materials, some of which I know you worked hard to source, and of course the publishers both at IOP Publishing and Morgan & Claypool who have made this whole process as easy as such a thing can be. Finally thanks to Richard Leach for suggesting I do this project in the first place.

# Author biography

## David C Cox

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David C Cox received his PhD from the department of Metallurgy and Materials Science, University of Cambridge, UK in 2001. He is currently a senior research fellow at the Advanced Technology Institute, University of Surrey, UK, but has been seconded to the National Physical Laboratory (NPL), UK as a senior research scientist since 2005. Having a broad background in industry and academia, covering many aspects of materials science, physics and electronic engineering, he has published close to 100 articles at the time of writing. Largely associated with both the Quantum Metrology and Materials groups at NPL, his most recent research work has concentrated on the area of using the focused ion beam to fabricate devices for quantum metrology. Additionally, he has developed a strong interest in the wider aspects of focused ion beam fabrication and the fundamental understanding of how it can be used to study materials, together with the errors associated with some areas of the technique.

# Introduction to Focused Ion Beam Nanometrology

David C Cox

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## Chapter 1

### Metrology

#### 1.1 What is metrology?

Metrology is the science of measurement and can have several meanings, depending on whom or what it is being applied to. Broadly, these can be classified in three areas. Firstly, it is used to create legal definitions of quantities such as weights and measures, applied to our every day purchases of items and the environment around us. Secondly, all industrial activity is underpinned by metrology. Components manufactured in different parts of our increasingly globalised world must be compatible with components made and assembled elsewhere. These compatibilities might comprise more than simple dimensional agreement, but may also be dependent on other measures such as voltage or chemical composition. Thirdly, fundamental metrology is concerned with developing new methods of measurement, establishing agreed standards, definitions and units of measurement and providing traceable measurements from which standards can be created and applied. It is fundamental metrology that underpins all of the other metrological activities. The field of fundamental metrology is extremely broad, often complex, and sometimes quite abstract. A 2004 definition stressing the huge range to which metrology can be applied was offered by the International Bureau of Weights and Measures (BIPM) ‘The science of measurement, embracing both experimental and theoretical determinations at any level of uncertainty in any field of science and technology’ [1].

All scientific disciplines have their own well-developed, *and mostly agreed*, use of language and terminology and in this respect metrology is no different. An example of this in the metrological context is the use of the word ‘uncertainty’ in the preceding paragraph. One thing common to all metrologists is that when we discuss our measurements we tend not to emphasise the absolute values we measure, but we stress the uncertainty (or error) of our measurement as we strive to improve the

science of the measurement itself. We can never be certain of our measurements except in very special cases, such as being certain that no electrical current will flow in an open circuit. As soon as we close the circuit and begin to take measurements we must take into account the precision, accuracy, reliability and (un)certainty associated with taking the measurement [2]. Beginning with precision, we could think of this in simple terms, such as how many decimal places do we have on our meter? However, this approach is incorrect and in fact our precision is also closely linked to how reproducible our measurements are. If we find we take many measurements of the same thing and they agree to three decimal places very well on a system capable in principle of measuring more, we can be confident of only the three decimal place measurement and this determines our precision, with the variation below this contributing to our uncertainty. Turning to accuracy, even if our measurement system agrees very well on identical measurements it does not necessarily translate that it is accurate. To determine accuracy we need to compare our instrument readings to a standard, traceable to one of the national measurement institutes. Reliability is closely linked to precision, but it is also a measure of the accuracy over time. Finally, the uncertainty of our measurement is a product of all of these things and as we make measurements we see variation from sample to sample, day to day and instrument to instrument. This dispersion in the measurements is our measurement uncertainty, with the relative uncertainty being given by the measurement uncertainty divided by the measured value. For readers for whom these concepts are new, a downloadable collection of measurement guides including an introduction to measurement uncertainty can be found at [3].

Throughout this book many examples will be given, or referenced, that quote values and units of measurement. However, the main aim of this text is not to emphasise or assign great importance to these values, but to make the reader aware of the measurement possibilities of using the focused ion beam (FIB) and the likely sources of error and uncertainty and the limits of this very versatile range of instruments.

## 1.2 Metrology in the FIB

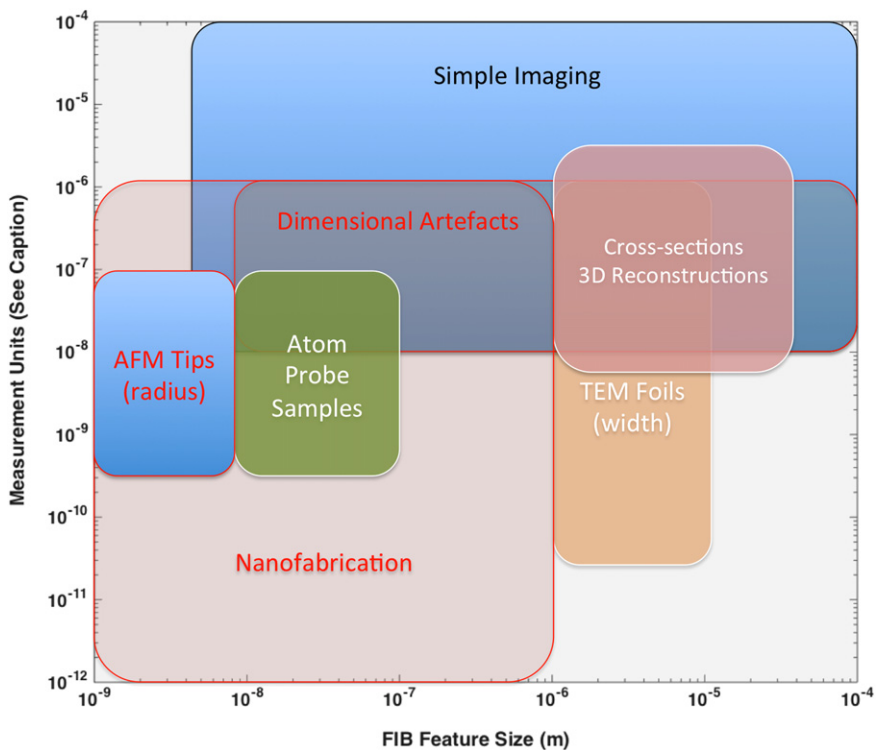
Based on these metrological definitions and key terms, what exactly do we mean by nanometrology? If one thinks of dimensional metrology, and applies the standard SI units and prefixes, a simple definition is the measurement of features from  $1 \times 10^{-9}$  m up to  $1 \times 10^{-7}$  m, this range being in agreement with commonly held definitions of what constitutes a nanoparticle, for example. However, we can use FIB to aid us in the measurement of many other properties, and as we shall see in chapter 4, when using FIB for the measurement of residual stress it is possible to determine stress levels of  $1 \times 10^9$  Pascal, 18 orders of magnitude from nano! Limiting ourselves only to dimensional metrology is also complicated, for example, when we produce a transmission electron microscope (TEM) sample with FIB and analyse it in a state-of-the-art TEM we can easily resolve columns of atoms and their spacing, some two orders of magnitude smaller than a nm. This could be argued to be metrology

carried out in the TEM, of course, but the sample was still prepared by FIB and it is often the best sample preparation method to enable the measurement to be undertaken. For these reasons, when we refer to nanometrology in the FIB we do mean dimensional metrology on the scale of nm, but we can extend our definition to include using the ion beam to either image, expose, or create features or structures that are on the scale of nm. At the upper limits, some of these things will extend beyond  $1 \times 10^{-7}$  m, and may in one or more dimensions be tens of  $\mu\text{m}$ , but the FIB instrument is more than capable of working at the nm length scale and is commonly found working in this regime.

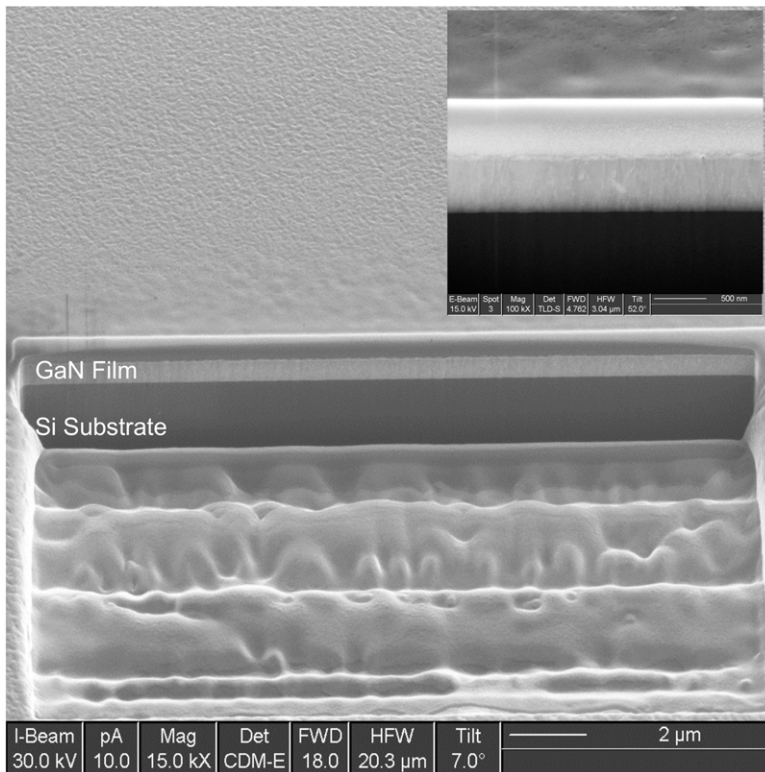
As we have alluded to, practising metrology with focused ion beam instruments is complicated by their high degree of versatility. We can use the instrument to make a direct measurement, we can use it to prepare a sample for measurement in another instrument, or we could even use it to make or modify a completely new instrument or component that can in turn be used to measure something completely unrelated to FIB (figure 1.1). Even in the first case the range of measurements we can make is large due to the varieties of this type of instrument. For example, few FIB systems these days are supplied without a scanning electron microscope (SEM) column, further increasing our measurement capability. However, a detailed discussion on scanning electron microscopy is beyond the scope of this book, where the reader need only be aware that very large numbers of FIB systems also incorporate SEM columns sharing the same vacuum, detectors and sample handling system. We will only discuss electron beam microscopy where necessary to assist in the discussion of the measurement taking place. For an excellent introduction to the SEM and SEM-based techniques, the reader is directed to [4]. Additional third-party-vendor equipment can also take these instruments beyond simple imaging by the addition of x-ray detectors and electron backscatter diffraction (EBSD) cameras. However, while both x-ray emission and backscatter diffraction signals are normally generated by electron beam, the FIB is used to produce the polished faces and slices for both techniques and the preparation of these will be covered in later sections. Finally, the long established and historic use of Ga as the source of ions is being added to with noble gas sources using light elements, He and Ne, largely for imaging, and additionally heavier elements from plasma-based sources such as Xe. These plasma sources offer significantly larger volume ion milling, owing to their high emission currents and the high sputter yield of the heavy ions, bringing FIB into new areas previously deemed beyond the scope of Ga source instruments. Similarly, although currently less common, metallic alloy source based instruments are available, often combining both light and heavy element options in the same instrument.

We can consider three distinct length ranges in FIB microscopy, the size of the sample to be studied, the size of the field of view of our interest in the sample and the resolution at which we can obtain information from the region of interest. Samples studied in the FIB can range from only nm in the case of dispersed nanoparticles, up to 150 mm diameter at the larger end of most laboratory systems. In figure 1.1 we can see that the typical length scale over which a focused ion

beam might be used covers some five orders of magnitude, from nm through to hundreds of micrometers, depending on the type of instrument and application. Although the maximum field of view can approach one mm, the instrument is likely to be used to resolve or expose detail with a resolution of a few nm on areas up to around  $100\ \mu\text{m}$  at the upper end. As an example, the single most common application for FIB is as a tool to reveal the internal structure of samples. In cases such as this we may be removing tens of  $\mu\text{m}$  of material, but we are only interested in imaging a few nm of a thin film to check for uniformity. For example, figure 1.2 shows a simple cross-section cut into a gallium nitride (GaN) film deposited on silicon (Si) wafer.



**Figure 1.1.** Selected examples of the length scales applicable to focused ion beam based metrology. The horizontal axis indicates a typical scale of the feature created or imaged by FIB. The vertical axis indicates the likely range of measurement activity. In most cases this is a simple length scale in metres, but the nanofabrication activities in particular may create devices where other properties are measured, such as magnetic flux or temperature. Text and boxes in black indicate an activity carried out directly in the FIB instrument, white denotes an activity where samples are prepared in a FIB and then measured in another instrument (or with an SEM column on the instrument) and red denotes an activity where devices or artefacts are created in FIB but used for other metrology.



**Figure 1.2.** Ion-milled cross-section of polycrystalline GaN film deposited on Si wafer. The FIB has been used to remove the material in the foreground, revealing the film thickness and texture. There is also a protective strap of ion beam deposited platinum directly above the sectioned area. The section is 20  $\mu$ m wide, 3  $\mu$ m deep and the film is approximately 500 nm thick with individual GaN grains of less than 50 nm. The image is an ion beam induced secondary electron image. The inset shows a higher magnification secondary electron image (from the primary electron beam).

The simplest measurement we can perform is based on imaging, utilising secondary electron or secondary ions ejected from the sample by the energetic ion beam, where the highest spatial resolution can be below 1 nm. Focused ion beam can additionally be used to measure material properties beyond dimension, such as composition, texture and residual stress. Furthermore, we can construct or modify devices that can in turn also be used to measure dimension, magnetic or electrical properties. We can also construct artefacts with sufficient precision for them to be used to calibrate other equipment. Table 1.1 describes some of the areas we will discuss in later chapters. These topics will be divided into two broad categories; materials science applications targeted exclusively at understanding materials properties and composition, and FIB-fabricated devices and artefacts that may also have some materials science applications, but can be used in a far wider measurement context.



**Table 1.1.** Examples of areas where FIB can be used as a measurement tool, or used to fabricate a device to make a measurement, and a brief method summary of each.

Measurement	Method summary
Imaging	Either secondary electron or secondary ion. Ultimate resolution determined by type of source. Primarily for topographic imaging, but ion channelling can lead to contrast in polycrystalline materials.
EDX	FIB-polished sections or slices for electron beam excitation of x-ray signals, measuring percentage concentration of elements present.
EBSD	FIB-polished sections or slices for measurement of grain size and texture via electron beam.
3D reconstruction	Sequential FIB slices to produce 3D reconstructions of either dimension, grain size and texture or composition of samples.
TEM foil	FIB-prepared TEM foil for dimensional, compositional determination with better than atomic resolution.
Residual stress	FIB-milled holes in the $\mu\text{m}$ range combined with image correlation to determine residual stress levels near the sample surface.
SIMS	Inclusion of a mass spectrometer allows material composition determination with few 10 s nm spatial resolution of sample surfaces.
Atom Probe	Successive atomic layers are removed from FIB-prepared sharp tips.
AFM tip	FIB-sharpened tips can achieve atomic resolution.
Dimensional artefacts	FIB-fabricated step edges and lines for calibration artefacts.
SQUIDs and Hall bars	FIB-fabricated magnetometers.

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## Chapter 5

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