Quantitative characterisation at the nanoscale by electron microscopy

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1-Introduction

The transmission electron microscope (TEM) is a choice instrument for the characterisation of materials and devices at the nanoscale. Apart from high spatial resolution, TEM offers a number of signals which can be exploited to obtain chemical and structural information concerning the specimen. Recent years have seen considerable advances in the use of electron microscopy and new techniques have been developed thanks to improved methodology, instrumentation and data acquisition systems.

Here we present a review of three such techniques particularly well suited for the study of nanoscale devices: strain mapping by high-resolution electron microscopy (HREM), elemental mapping by energy filtered microscopy (EFTEM), and measurements of magnetic and electric fields by electron holography.

2-Strain mapping by HREM

High-resolution electron microscopes form images of the atomic lattice itself. By measuring the position of the lattice fringes in the image, it is therefore possible to determine distortions of the lattice with respect to a given reference. In practice, this can be achieved using real-space of Fourier-space methods[1,2].

Figure 1 shows an example of the analysis of a strained semiconductor island grown on a substrate of GaAs[3]. The image was taken on a Philips CM20 UT operating at 200kV with a point resolution of 0.19 nm. Displacement of the lattice fringes with respect to the substrate was measured using the geometric phase technique[2]. The deformation of the lattice can then be calculated by taking the gradient of the displacement field.

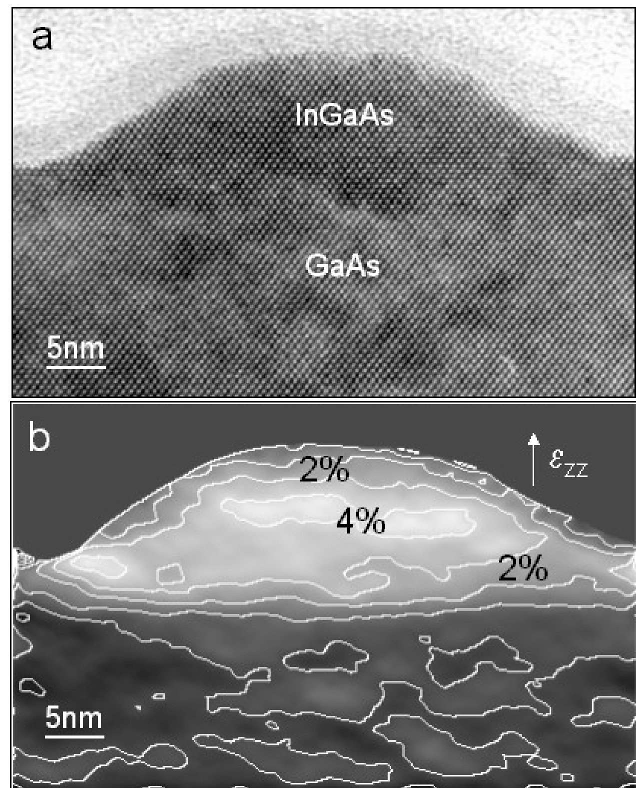

Figure 1. Strain mapping of an island of $In_{0.35}Ga_{0.65}As$ grown on a [110] GaAs substrate by molecular beam epitaxy (MBE): (a) high-resolution image; (b) deformation map calculated from the displacement field in the growth direction (contours every 1% of z-deformation). Deformation is due to both compositional changes and strain.

The distortions introduced by the imaging process itself should always be evaluated and taken into account when using such methods[4]. There are, for example, certain experimental conditions for which measurements are optimal[5]. Great care also needs to be taken with specimen preparation so as not to introduce additional strain. Due to thin film relaxation, strains measured in experiments are not necessarily representative of the bulk state. Ideally, finite element calculation should be carried out as part of a study to investigate such phenomena[3].

3-Elemental mapping by EFTEM

Energy filters have been perfected in recent years for TEM and are becoming standard equipment on modern microscopes. They are composed of imaging and aberration correcting lenses, which allow images to be created of electrons having undergone specific energy losses in travelling through the specimen. These losses can be characteristic of particular chemical species. A sequence of images at different energy losses is taken of the specimen. Typically, two images are acquired before a particular characteristic loss, or “edge”, and one after. The first two enable a modelling of the background level which is subtracted from the third image : the so-called three window technique. By acquiring images at different energy losses, it is possible to build up a projected elemental map of the specimen, element by element.

An example of such an experiment is shown in Figure 2 using a JEOL 3010 microscope operating at

300kV equipped with a post-column energy filter (Gatan). The sample is a ultrafine powder of $Fe_{0.2}Ni_{0.8}$ made by the novel technique of cryogenic evaporation-condensation[6]. Each elemental map was determined from three images at the characteristic losses for Fe, Ni and O. The typical spatial resolution of the technique is 1-2 nm (note the oxide layer on the particles which was measured to be 3 nm from high-resolution microscopy images) depending on the specimen quality and thickness. To obtain fully quantitative maps of absolute concentrations, particular care must be taken with modelling diffraction and multiple scattering effects.

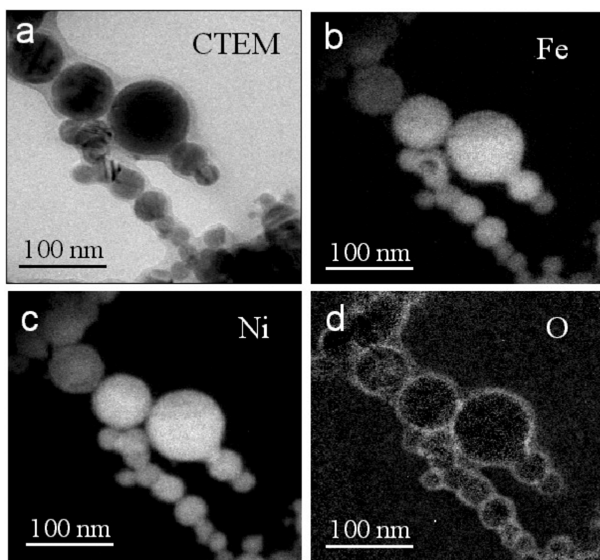

Figure 3. Elemental mapping of FeNi nanoparticles using the three window technique: (a) conventional bright field image; (b) map of Fe; (c) map of Ni; (d) map of O.

4-Measurement of magnetic and electric fields by electron holography

Electron holography is nowadays possible due to the field-emission gun. This electron source provides a highly coherent electron beam which can be split in the electron microscope above the specimen using a charged wire, or “bi-prism”. An electron hologram is formed by interfering the beam which has travelled through the specimen with that having travelled only through the vacuum. The phase shift of the wave function due to the specimen can thus measured. Any magnetic and electric field present will automatically phase shift the electron, according to the laws of electro-magnetism.

Figure 3 shows the result of an experiment in electron holography performed on a Philips CM300-ST FEGTEM operating at 300kV equipped with a Lorentz lens. This extra lens allows the objective lens of the microscope to be switched off during experiments leaving the specimen in field free conditions. Holograms were taken after magnetising the specimen in two opposing directions. This allows the elimination of the electric field component to the phase[7].

Acknowledgements

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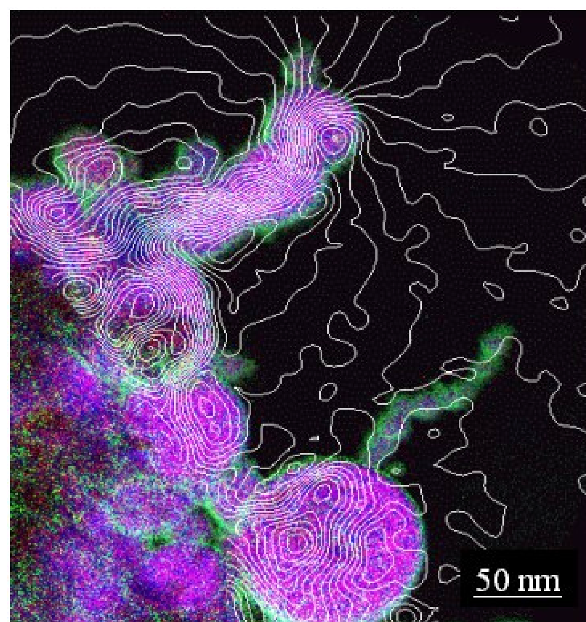

Figure 3. Remanent state magnetic field measured in and around FeNi particles by electron holography. The field lines have been superposed on an elemental map of the specimen.