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THE UK MEIS FACILITY : A NEW FUTURE

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Abstract

The Medium Energy Ion Scattering facility at the Daresbury Laboratory, one of only a few such facilities in the world, has served the UK community since 1996. It provided a 50-400 keV ion beam and a very comprehensive experimental station where samples could be studied and the energies and angles of the scattered ions measured. It has now closed, but will be relocated some 50 miles to the University of Huddersfield: it will be recommissioned and should be available to users by the end of 2012. We report on progress, and on the facilities which will be available for users at the new site and under the new management.

MEIS ANALYSIS

The MEIS technique uses an energetic H or He beam to probe the surface structure of materials. It is a low energy enhancement of Rutherford backscattering/channelling capable of giving compositional and structural information as a function of depth with a substantially improved depth resolution[1, 2, 3]. The energy of the scattered ion depends on the mass of the nucleus with which it collides, modified by the inelastic energy loss on its incoming and outgoing trajectory. Thus MEIS depth profiling is based on the energy analysis of He or H ions, typically between 50-200 keV, backscattered from the sample under investigation.

For a crystalline target, the number of scattered ions at a particular angle depends not only on the Rutherford scattering formula but also on channeling and blocking effects. If the ion beam is aligned with a major crystallographic axis the surface atoms shadow deeper atoms. This alignment makes the technique surface specific. Ions scattered from the lower layers will have their outward paths blocked at certain angles by upper layer atoms. The variation in scattered ion intensity with angle thus relates to the geometrical arrangement of surface atoms. The accuracy of determining atom positions is of the order of sub pm, comparable to techniques such as LEED and SXRD.

Data is collected in the form of a 2-D spectrum of the backscattered ion yield as a function of both energy and scattering angle. When using double alignment conditions (in which the ion beam enters in a channelling direction and is detected along the blocking direction) energy spectra are obtained by making a cut through the 2-D spectrum at a fixed exit angle (the blocking direction). The spectrum thus obtained can then be converted into quantitative depth profiles of most species present in, say, the first 30 nm of the surface layer, for nuclei with a mass as low as carbon.

The energy of a scattering peak in the spectrum is related to the depth at which a collision took place and is the result of both elastic and inelastic energy loss processes. The conversion of the energy scale into a depth scale relies on an accurate knowledge of the inelastic energy loss rates (dE/dx), which is well documented [4, 5]. When considering a single species such as e.g. a semi-dilute implant of Sb in Si, this is comparatively straightforward [6, 7] but becomes substantially more complex in the case of the analysis of multilayers, each layer having strongly different energy loss rates. This requires spectrum simulation [8]. For the Sb in Si system mentioned, the conversion of scattering yield to concentration is normally achieved through an internal calibration by comparing the yield with that obtained from an amorphised Si sample, taking into account the squared ratio of the scattering cross sections for Si and Sb ($\propto (Z_1/Z_2)^2$, where Z is the atomic number). This procedure assumes that the cross sections for scattering for scattering at these medium energies are Rutherford-like, which is not wholly correct since electron screening of the nucleus still occurs. This deviation is accounted for by the Andersen correction [9].

This cross section correction is combined with a second correction that accounts for the degree of neutralization of the He particle when emerging from the sample surface, which is based on a collation of experimental data made at the FOM Institute in Amsterdam [10]. The neutralisation correction is important since only charged particles are analysed in MEIS. It is considered that the application of this correction has now resulted an accuracy of the concentration determination of better than 5%. At the same time the precision of the depth determination of a scattering event is better than 0.2 nm. This ability makes MEIS a highly valuable characterization tool of e.g. ultra shallow implants and dielectric films of nanometer thickness, etc. In addition the technique has been extensively used for the characterization of the registry, relaxation and expansion of submonolayer (and monolayer) overlayers of species on single crystal surfaces, the growth thereof as well as inter-layer diffusion effects.

As an illustration of the information that can be gained, we take an example from the field of nanoelectronics [11]. Fig. 1 shows energy spectra taken using 100 keV Helium ions incident on the [-1 -1 1] direction and scattered along the [111] direction of a silicon substrate onto which had been deposited a 3 nm layer of conducting TiN. This layer forms one plate of a Metal-Insulator-Metal capacitor (MIMcap)[12]. Onto this is deposited a 3 nm layer of

high permittivity stoichiometric strontium titanate and a final 2 nm layer of TiN. As the measured scattering energy depends on the mass of the target nucleus and also on its depth in the specimen, the figure shows two Ti peaks, the lower energy one corresponding to the inner layer, with the ions travelling further through the material and losing more energy. The sample was heated to around 650 °C for 15 seconds to assess the effect of thermal processing in device manufacture, and the spectra before and after this treatment are labelled D11 and D12. The lines are model simulations to establish the layer structures; these models do not include the signal from silicon, which is not of interest.

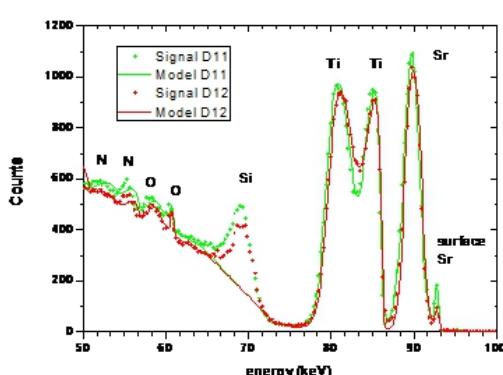


Figure 1: MEIS analysis of a MIMcap structure.

The details of these spectra show that the nominal layer thicknesses are indeed quite accurate (within 0.1 nm), that Ti has diffused into the STiO layer during annealing, and that there are some strontium atoms segregated onto the surface as well as some surface reoxidation of the specimen. (The narrowness of the peaks, as well as their position, shows they result from the surface and have not been affected by energy loss in the material.)

MEIS AT DARESBURY

The Accelerator and Analysis Stations

MEIS was built using apparatus from components of the former Nuclear Structure Facility at Daresbury. The ion source typically provided H⁺ or He⁺ ions at energies between 50 and 400 keV, with currents of 0.1 to 1.0 μA in a 1 × 0.5 mm spot on target. The beam from the source is focussed by an electrostatic quadrupole lens, a large mass selection magnet which, once set, also defines the beam energy, a second electrostatic focussing quadrupole and a set of collimators. The layout is shown in Figure 2

The beam is then scattered off a sample in the scattering chamber, and the scattered ions are analysed in a toroidal electrostatic device, and measured by a 2 dimensional channel plate detector, which records the angle and energy of the scattered ions. The target can be aligned with three translational and three rotational degrees of freedom, and heated to any temperature up to 1300 K. This exper-

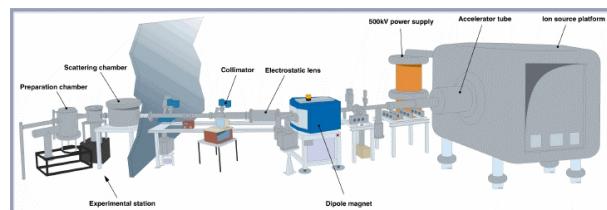


Figure 2: The MEIS accelerator beam line at Daresbury.

imental station includes a preparation and storage area in which up to six samples can be stored, characterised and treated using LEED, Auger, sputter cleaning, evaporation sources and gas dosing, with sample heating facilities. It typically takes only of order 10 minutes to load a sample from air. A fuller description is given in ref[13].

Some Results from MEIS

We list some highlights of the results achieved over the years of operation of MEIS at Daresbury. The selection is chosen with a view to showing the breadth of studies that can be uniquely performed by MEIS; the compilation of a list of the most important results would be another matter.

MEIS has been used for the investigation of the nature of gold/palladium nanoparticles on a substrate [14]. Bimetallic catalysis is important in many industrial chemical reactions, such as the manufacture of vinyl acetate, and the reactivity depends strongly on the surface properties so it is important to understand their nature. Gold and palladium were vacuum deposited on silicon, and detailed studies of the MEIS data show that bimetallic nanoparticles are formed on the surface with an Au enriched shell and Pd enriched core.

Titanium dioxide is an important support material for catalysis, and SXRD and LEED studies did not agree on its surface structure. A MEIS analysis [15] confirmed the structure to be that suggested by the LEED data, with the surface bridging oxygen atom relaxed outwards.

When antimony is deposited on copper, on a Cu(111) plane, MEIS studies [13] showed that the elements mix and a layer of Cu₃Sb alloy is formed, about 100 Å deep. At temperatures above 210 °C the structure changes and the Sb content of this phase is lower. Above 305 °C the structure changes to a Cu₂Sb stacking faulted surface layer.

Studies of thin layers of hafnium dioxide, HfO₂, on silicon dioxide were done using MEIS, to investigate the behaviour of this material as a very high dielectric-constant gate material replacing SiO₂ which suffers from problems of tunnelling in subnanometer layers in CMOS circuits. This work was performed [8] as part of the analytical network for micro- and nanoanalysis (ANNA) project. MEIS was used to study the structure of a 7 nm wide stack of multilayer nanofilms, 2 nm deep, with 5 different components: TiN, Al₂O₃, HfO₂, SiO₂ and Si. The layer structure could be studied with subnanometer resolution, and the interdiffusion between layers measured.

Thin layers of rare earth (Gd, Tm, Er, Y, Hb and Dy)

silicide layers on silicon can form nanowires for spintronics [16]. MEIS can be used to measure their structural parameters (bond lengths and layer spacings) with an accuracy of order 0.01 Å, and distinguish between hexagonal and tetragonal structures. It was discovered that the temperature at which the surface is grown affects the structure.

CURRENT STATUS

MEIS ceased operation on 21st November 2011. It was then disassembled, and parts checked to verify whether they needed repair or replacement, and for radioactivity (a possible leftover from its use as part of the NSF). It is currently in transit, as many crates in several lorry loads, between Daresbury and a new location in the recently established Institute for Accelerator Applications at Huddersfield. Here it will be reassembled and commissioned. We anticipate this will be completed by the end of 2012.

The new system will use most of the existing components, particularly in the analysis station, but some changes are being made, such as a redesigned beamline which operate only up to 200 kV, rather than 400. Experience showed that the higher energies were never used, and the extra voltage added considerably to the complexity of the apparatus.

FUTURE OPERATIONS

MEIS can be used in many of the areas of research currently considered of the highest importance : semiconductor device fabrication, catalysis, light metall alloy structures, biomedical joint replacements, photovoltaic materials, nanometrology, and tools for the hydrogen economy.

Research

MEIS is a powerful tool for a broad range of studies, and the University of Huddersfield is hoping that it will continue to be used by a broad community. The flexibility enjoyed by a university means that we can welcome proposals which are of scientific interest and importance without the need for explicit funding at every stage of the process. The existing user community, with their own programmes of research, will be very welcome to continue to use MEIS as soon as it is back in operation.

We hope that researchers, within the host university and outside it, who had not previously used MEIS as a technique will discover its possibilities and exploit them. Workshops and seminars are being arranged for this purpose.

Commercial Use

MEIS is a valuable tool in the semiconductor industry, being able to characterise the thickness and composition of layers laid down on a silicon wafer during the fabrication of an integrated circuit. We anticipate interest from process development establishments and industry, and will encourage them to use MEIS for the investigation and understanding of their device manufacturing processes. The university has a strong tradition of working with industry and businesses, including the recent opening of an on-campus

Enterprise and Innovation Centre, and such activities will fit very naturally into the operation of the facility.

SUMMARY

The MEIS facility provided many useful and interesting results during its time at Daresbury: its high current and high speed DAQ system enabled specimens to be examined rapidly, and the facilities offered by the target station gave users a powerful and flexible system for manipulation and treatment. These will be retained in its new location, and we hope that the existing user community will soon be able to resume their use of MEIS, and that new users, from universities and from industry, will discover its potential and exploit the possibilities it opens to them. Anyone who might be interested is invited to contact the authors.

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