Professor Michael Whelan

Interviewed by Bernadette Bensaude-Vincent, Oxford Materials Department, December 12, 2002

Bernadette Bensaude-Vincent (BBV) - Could you tell a few details about your own work on the experimental discovery of dislocations in 1956?

Michael Whelan (MW) - In the 1930s there was no experimental evidence for dislocations but theoretical predictions were built up about the way dislocations behave. What was really missing was any direct observations of dislocations, although slip lines on the surface of metals had been observed by optical microscopy. The question was how could that occurat the sort of stresses that were observed experimentally? If you work out the stress required to push one layer over another as a whole, it is much higher than it would be if you introduce the concept of a dislocation. A dislocation is like a wrinkle in a carpet; Peter Hirsch used this image. When the concept of a dislocation came to the forefront with many papers about the behaviour of dislocations, there were unbelievers, among the Russians in particular. At the Cavendish Laboratory in Cambridge in the group of W.L. Bragg they had suggested simple X-ray experiments to detect the small subgrains that were hypothetically formed in the plastic deformation of metals.

BBV - When did you start?

MW - I started in 1954 with Peter Hirsch. He was a post-doc in Cambridge. He had got his PhD about 1950 using X-rays to study the textures of metals. They had developed a microbeam X-ray technique under the supervision of Professor W.L. Bragg and Dr W.H. Taylor. It was essentially based on X-ray equipment - a high intensity X-ray generator with a rotating anode produced a beam of about 10 microns diameter for taking back-reflections photographs from a small area of a deformed metal.

BBV - So in 1954 X-ray diffraction was still the dominant technique for studying dislocations

MW - When I started the X-ray machine was still in the lab and there was another student still using it, Chris Ball, who eventually emigrated to Australia, and I started in the same room as the microbeam X-ray machine. I remember him telling me - you don□t have to worry about the scattered beams□. I tolerated it for about six months and then moved to another room. The work with X-rays had its limitations - you could not get a smallenough beam to examine certain metals, notably nickel and copper. The scattering is very weak with X-rays and typical exposures were about 24 hours. Anthony Kelly had the idea of going to one of the new electron microscopes that had been developed to do transmission electron diffraction. An electron diffraction pattern exposure time was only a few seconds. I was asked to investigate this electron microscope technique further. Some people had already worked on it. Heidenreich in particular at the Bell Telephone Labs in the USA, but not with the idea of actually seeing dislocations.

BBV - Did Heidenreich produce the first images of dislocations?

MW - If you read my article you will see that Japanese had also produced dislocation images. The Japanese were interested primarily inmoire fringes, but if you look at some of the pictures thatProfessor Hibi took of mica and graphite, you can see that they contained dislocation images. But they didn□t really understand the concept of a dislocation. I heard that from an eminent Japanese scientist, Professor R. Uyeda. We came from the X-ray side. We were interested in crystallography whereas many electron microscopists did not know much about diffraction. It was important to have a team which was expert in diffraction to move into this field.

BBV - You mean for interpreting images?

MW - Yes, the theory that you need to understand how the electrons interact in a crystal which contains dislocations is essentially diffraction theory. It is more or less the same theory that you have for X-rays except in the difference of scale. The theory was well known to crystallographers. It had been developed in this country by Darwin and Bragg andby Ewald and von Laue in Germany.

BBV - Where did electron microscopy come from?

MW - Electron microcopy was developed in the 1930s by the team of E. Ruska, M. Knoll and B. von Borries working in Berlin, and was initially used by metallurgists mainly to look at surfaces. Their interest initially was to extend what they did with the optical microscope, namely to examine surfaces. They made thin replicas of surface structure and examined them in the electron microscope. They had to shadow the replicas with heavy metals to obtain good contrast. This was the main area that metallurgists were engaged in during the late 1940s when electron microscopy

started to be used again after World War II, with the exception of Heidenreich in the USA and Raimond Castaing in France, who developed microprobe analysis techniques.

BBV - Did you know Castaing and interact with him?

MW - Yes, I wrote to him as a research student and sent him some of my electron micrographs. I met him first at a conference in Madrid in 1956 at the end of my second year of research. I remember him taking me for a ride in his car through Madrid. He was a scary driver. There was a session at an EMSA conference in Portland Oregon in 1999 that was devoted to the memory of Castaing.

BBV - Did you take your Ph D in physics or in crystallography?

MW - In the physics department in Cambridge, known as the Cavendish Laboratory.

BBV - Did you consider yourself as a physicist or as a *crystallographer?*

MW - A bit of both actually. I was interested in crystallography but Cambridge had traditionally a broad natural sciences undergraduate course. You studied for three years. In the first and second years you had to study a number of subjects, for example in my case physics, mathematics, chemistry and crystallography. In the third year I specialized in physics, but in the crystallography course during the first two years I got interested in X-ray diffraction. Therefore, when I started in the Cavendish Laboratory I decided that I would join the group working on X-ray diffraction.

BBV - When did you shift from X-ray to electron microscopy?

MW - When I started. As I have said, Kelly had initiated the work, but he left the Cavendish to work in the USA. He had taken some transmission electron micrographs with Peter Hirsch and Jim Menter, who worked in the Laboratory of Physics and Chemistry of Rubbing Solids, a subdepartment of the Physical Chemistry Department at Cambridge. He had an electron microscope with which you could do selected area electron diffraction . Selected area diffraction was a relatively new technique at that time. It enabled you not only to take an image of the specimen, but also to insert an aperture into the microscope column, selecting an area down to about a micron or half a micron in size to obtain a transmission electron diffraction pattern. You had to have a thin specimen, about 1000 Angströms thick. The simplest thin specimen you could easily obtain at that time was beaten gold foil. It could be beaten down to about 1000 Angströms thickness. It would transmit electrons at that thickness but it was heavily deformed. They saw things in the images that they could not interpret. Peter Hirsch had the idea that they could be stacking faults on inclined planes. If you can see a stacking fault it ought be possible, he thought, to see a dislocation because the dislocation in a face centric cubic metal can dissociate in two partial dislocations, like a little ribbon with the space in between them being a stacking fault. So if you could see the fault you might be able to see the dislocation by virtue of the ribbon . That was the initial suggestion that Peter Hirsch made. It was simply a hunch. Experiments had to be carried out and interpreted. That was my job.

BBV - What kind of microscope did you use in 1954?Was it easy to get electron microscopes in research laboratories?

MW -At that time the Cavendish Laboratory had two electron microscopes in the Electron Microscope group headed by Dr V.E. Cosslett. One was a pre-war instrument manufactured by the Siemens Company of Berlin in 1938. You could not do electron diffraction with it. It had been picked up in Germany by the British Army as a war reparation. Ernst Ruska developed the first electron microscopes in the early 1930s based on work done in the 1920s. The first commercial firm to produce a microscope was Siemens, who engineered a microscope that Ruska had basically designed. It was a two-stage magnification instrument with a single condenser lens. You could not do selected area electron diffraction with it. You need three stages of magnification for that.

I took some electron micrographs using this instrument. The resolution was not good, but some of the micrographs of beaten aluminium showed images of dislocations. This was not realised at the time. It was only much later, with hindsight, when the micrographs were re-examined, that dislocation images were recognised.

In early 1956 I was able to use the new Siemens Elmiskop in Dr Cosslett□s group. This had much better resolution, and dislocation images were recognised in the summer of 1956.

BBV - What do we see on these pictures?Picture 1 plate 404 and Picture 2 plate 1833

MW -Plate 404 This relatively low magnification micrograph shows the subgrain structure of beaten aluminium foil. Subgrain boundary walls are visible. These are made up of dislocation arrays. The subgrains are about 1μm in size. Inside the subgrains there are many extinction contours. These arise from dynamical electron diffraction effects due to buckling and thickness variations in the foil.

Plate 1833 Here at higher magnification we see subgrains of size 1 or 2 μm in diameter. In the centre we see a cross-grid network of screw dislocations constituting a low angle twist boundary, on or close to the (100) crystallographic plane. In the next subgrain we see a slip trace (the contrast feature with a 90^{o} bend). A dislocation has moved across the grain, first on one {111} plane and has then cross-slipped to another {111} plane, leaving behind the trace of its path. In imaging dislocations by so-called «diffraction contrast», we do not attempt to resolve the atomic arrangement. There is a strain caused by the displacement of atoms near a dislocation, and it is the region of strain that is made visible as a dark line by diffraction contrast.

Only one beam is used to form the image - here the directly transmitted beam. Other diffracted beams are removed by an aperture in the objective lens of the microscope. The dislocation images reflect the fact that electrons are scattered outside this aperture. This is diffraction contrast.

BBV - What does it tell you about the material?

MW - You don□t aim at resolving the atomic structure. Metal physicists think in terms of a sort of quasi-particle. They are not usually interested in details about the atomic positions. They are interested in the concept of a dislocation as a line defect with properties such as energy, line tension, inter-line forces. What you want to see is a line.

BBV - How can you sort out the line defects that are dislocations from other spots or lines?

MW - With some experience you can easily identify dislocation lines. Other contrast features arise from artefacts. The instrumental resolution required to observe dislocation lines is not high.

It is of the order of 100 Angströms. To see atoms you need a resolution of about 1 or 2 Angströms. Modern instruments have this resolution. To image the crystal lattice, you need to include more than the directly transmitted beam. You include a number of diffracted beams and resynthesize the atomic image by using several beams. You can use modern electron microscopes to actually see the atomic arrangement with a very thin specimen. You can gain a sort of projection of the atomic lattice structure. But then again, you have an interpretation problem. The question remains - what does the image mean?

BBV - On which metal did you first observe dislocations?

MW - We first observed dislocations in aluminium foil.

BBV - Following your first observation and publications I guess that there was a great excitement among physicists that increased the demand for commercial microscopes

MW - In post-war years, the Siemens company improved on the design of their pre-war instrument and manufactured the Elmiskop 1. It was presented at the London International Electron Microscope Conference in the summer of 1954. The first to be delivered from the factory was acquired by the Electron Microscope Group at the Cavendish with a grant from Nuffield foundation. It was designed mainly for biologists. Although you could do selected area diffraction with it, there was an incovenient system for energizing the lenses which made it difficult to obtain electron diffraction patterns initially. But after a few years Siemens modified the electronics to enable fine focus illumination to be used with electron diffraction. For many years the Siemens Elmiskop had most of the world market.

BBV - Was it difficult to operate the early electron microscopes

MW - Most of the difficulties in those days was preparing thin enough specimens. Also adjustment of the electron beam was done mechanically, requiring some agility of the operator. Nowadays adjustments are made by magnetic deflection. Practically the only thing which is still mechanical in modern instruments is the actual movement of the specimen stage.

BBV - How long did you take to familiarize yourself with the electron microscope technique?

MW - There was an assistant in the Electron Microscope group Bob Horne who was in charge of the Elmiskop instrument. I seem to remember that he had never studied for an undergraduate degree but he had been an electronic assistant during World War 11. He told me that he had been involved in recording ground-to-air voice communications during bombing raids on Germany using wire recordes borrowed from the BBC. The recorded voices were broadcast again during later raids to confuse the enemy□s defence fighter aircraft! He acquired skills in electronics during the war. We collaborated and the first papers were co-authored by Hirsch, Horne, and myself. Horne took the pictures. He operated the instrument. The difficulty for me was to get my own hands on the instrument. But there were three viewing windows on Siemens Elmiskop. The operator□s window and two side windows, so it was possible for me to observe through a side window. Later when we first saw dislocations, Peter Hirsch would be observing at the other side window. To record the movement of dislocations we attached an external ciné camera, recording through the front window. We needed not only an operator but also somebody trying to tilt the specimen to give it best contrast and somebody to operate the ciné camera. So three of us, Hirsch, Horne and myself worked altogether. Another difficulty was the availability of the machine. It was the only microscope in the university. I had an afternoon session per fortnight. During the rest of the time the instrument was mainly used by biologists. (Lord Victor Rothschild was one of them. He was an eminent biologist and was Chairman of the Agricultural Research Council. He was one of the few members of the Rothschild family who did not go into the family banking business. He stayed in Cambridge doing research, and later directed research at Shell laboratories and became an advisor to Margaret Thatchers government). But when we started obtaining exciting results we got more time on the instrument. Ultimately around 1957 we got our own instrument. And we had 2 or 3 of those instruments before we left Cambridge to come here.

BBV So it was a rapid increase. How expensive were these instruments?

MW - In those days you could buy one for about £8000. I remember Professor Nevil Mott coming - he was the head of our department - and looking at the first Elmiskop we obtained and he said - «ah, two houses!». In the 1950s you could buy a professoriel style house in Cambridge for about £4000. Money was obtained from various sources. Part of the money came from the Royal Society and the rest came from the Department of Scientific and Industrial Research. The instrument was for our groups use, but it was situated as an attachment to Dr Cossletts group; so that his technicians could advise us.

Initially we had a beam of 100 microns diameter which is quite large. The images were of poor quality, but they were not much better than Heidenreichs images because he too had not used a double condenser lens. The initial emphasis was to look at electron diffraction patterns. So we had to use the single condenser illumination. One day Bob Horne switched the lens arrangement to the system that biologists, used but with which you could not do electron diffraction. We noticed that the images obtained very clear and picturesque. After a while we saw changes taking place in the image, and the dislocations started moving around. I called Peter Hirsch to come and have a look. He said we must get a ciné camera to record the dislocation motion. One of our assistants who had worked at the National Physical Laboratory and knew something about movie cameras, advised us on who to contact at the NPL to borrow a ciné camera.. We mounted the camera on the microscope, put some high speed film in it and we made a ciné film of the motion of the dislocations. We produced a silent movie. Peter Hirsch and I presented the film at various conferences and we received requests from people who wanted to obtain a copy of it for teaching students. So we started a business in the Cavendish to sell it at cost price. We still have the negatives. Recently we put it on video. I showed this movie in Japan recently on a trip to raise funds for my college at Oxford. A generation of students has grown up, who have never seen this old movie. So it is entering a second life!

BBV - How many instruments did you get when you moved in Oxford in 1966?

MW - We bought a new Elmiskop. With the help of research students we had converted an instrument in Cambridge to study the energy losses of the electrons when they are transmitted through the specimen. We moved it to Oxford. Then with the help of another competent research student we built another instrument based on a design of Castaing which enabled energy filtered images to be taken. There was an old EM6 electron microscope already here in John Martins lab. But we were in a Department annexe on the ground level of another building.

BBV - Did you need a special environment for using those electron microscopes?

MW - It was not a bad building. Because it was on the ground floor vibrations were minimized. Later we bought instruments with higher resolution that required special foundations, we had concrete blocks put on the floor to minimize any vibrations coming from traffic outside the building.

BBV - How did you teach electron microscopy?

MW - I started giving a graduate level course on the dynamical theory of scattering to research students. At the undergraduate level initially there was not much teaching of electron microscopy. There was only brief mention of it, with no experimental work. That has now changed. Undergraduates can have practical classes in electron microscopy.

BBV - Do you remember when these practical classes started?

MW - I dont remember exactly. Probably in the late 1970s. I used to give a course on optical and electron microscopy to undergraduates. But there was no practical work; in the practical classes they could only take X-ray pictures. Now in the practical classes they use various electron microscopes, both scanning and transmission. Compare this with the days when we started with an instrument available one afternoon a fortnight.

BBV - What is the importance of electron microscopy now in this department?

MW - It is used as a routine technique now in the department. Anyone working in any area of materials science can get advice from the electron microscope group.

BBV - Is it also the case for the Scanning Tunnelling Microscope?

MW - That is a different technique, a surface technique. What is interesting is that they awarded the Nobel prize to Binnig and Rohrer for inventing the STM, and at the same time they awarded it to Ernst Ruska, the German scientist who developed the electron microsocope in the early 1930s, almost 60 years before. There were in fact three people involved in this work in Germany, but the other two were already dead by the time the Nobel Prize was so belatedly awarded for the invention of the electron microscope.

BBV - What is your view of Materials Science?

MW - It is a miscellany of problems all related to the way atoms behave collectively in the solid state and even to some extent in the liquid state.

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