The scale of the microstructure of many important engineering materials has decreased rapidly. There may now be millions or billions of electrical circuits on a single silicon wafer. To investigate the chemical and physical characteristics of such material systems, engineers need to make measurements in the subnanometer (< 10^{-9} m) range. This is possible with a high performance scanningtransmission electron microscope fitted with a field emission gun (FEG STEM). A high-brightness (> 200 kV) source is used for illumination instead of the visible light used in optical microscopes. The wavelength of illumination is less than 0.3 nm, so small that the electrons can either interact with columns of atoms in a specimen or pass between them. By analyzing what occurs as electrons exit

a specimen, important information can be obtained on the type, location, and bonding of its atoms.

Low-angle and high-angle electron scattering

The basis for image formation is the capturing of the electrons after they pass through a thin, electron transparent specimen. Electrons are focused on a specimen by adjusting mechanical apertures and electromagnetic lenses. As the electrons enter the specimen, they interact with columns of atoms and their electrons in various ways. Depending on the strength of the interaction, electrons are scattered weakly or strongly by the specimen. If the trajectory changes by less than a degree or so, an electron is said to be *transmitted*.



Figure 1. Model 3000 Annular Dark Field Detector.

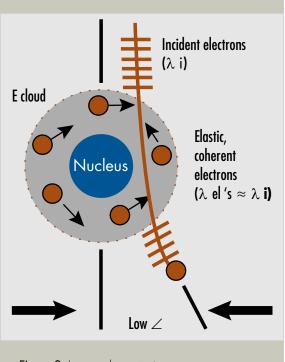
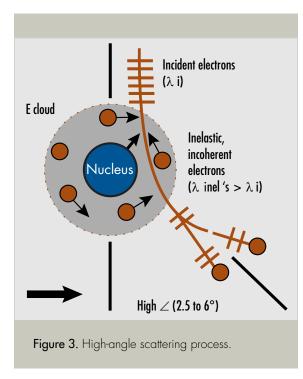


Figure 2. Low-angle scattering process.



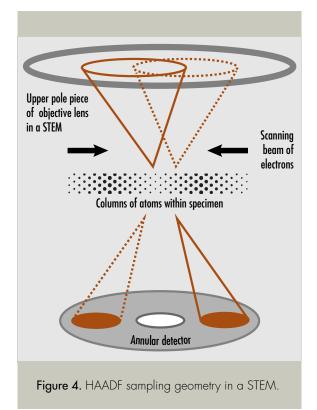
With the rise of quantum physics, materials scientists now speak about different types of radiation as being both particles and waves. Two descriptions of the scattering process have come to be used almost interchangeably. Electrons scattered through low angles (LA) are said to be *elastic* in the particle description and *coherent* in the wave description. They are elastic because the near collisions with the atoms in the columns involve conservation of total energy. They are coherent since their wavelength is not affected. The high angle electrons are inelastic because energy is lost in collisions with atoms in the columns. They are also incoherent since a loss of energy equals an increase in wavelength.

Bright field and dark field imaging

An image may be formed in the STEM using either the transmitted or scattered electrons. In bright field, regions of the specimen which transmit electrons appear bright while those that scatter appear dark. In dark field, this contrast scheme is reversed. If a dark field image is formed using selected LA electrons, it reveals how groups of atoms are stacked and oriented in the specimen. This contrast mechanism depends on constructive interference from equally spaced columns which scatter at the same angle. Analogous to visible light passing through a set of slits, we call this special scatter diffraction. In dark field, whatever is diffracting will appear bright and give the greatest diffraction contrast.

High-angle dark field vs. low-angle dark field contrast

An image formed using high angle electrons in dark field does not show diffraction contrast, since the inelastic, incoherent electrons do not diffract. The intensity of the high angle scatter basically depends only on the type of column atom. The higher the atomic number (Z) of the column atom, the greater the loss of energy as an electron bounces off it. Columns of high Z atoms will appear bright since they scatter more strongly. Low Z atoms scatter weakly and so appear dark.



Changes in chemistry on the sub-nanometer scale can be detected using this Z-contrast method. The resolution exceeds that of competing techniques such as X-ray microanalysis using EDS (energy dispersive spectrometry).

HAADF sampling geometry

There is one camera length setting that provides optimum imaging conditions for sampling high angle electrons. The incident beam is scanned across the specimen while the high angle scatter is measured by a detector. The detector is donut shaped (annular) so that the low-angle and transmitted electrons are not counted.

Because other detectors are near the specimen, the HAADF (high angle annular dark field) detector can be moved to another beam cross over point. A position near a projection lens can be used, and the same quality of information will be obtained.

HAADF detector

A HAADF detector's function is to count the high angle scatter. An annular YAP (yttrium

aluminum perovskite) crystal is used to capture the high angle electrons. When an electron enters the YAP, it scintillates by converting a portion of the energy into visible light (photons). The photons travel through a quartz cylinder, which is attached to a photo-multiplier tube (PMT). The surfaces of the YAP crystal and light pipe are coated with aluminum to shield them from interference.

The PMT (-1 kV) converts photons back to electrical current. Its three main sections are the photocathode, the electron multiplier, and the anode. In the photocathode, a photon is reabsorbed and a new electron is released. This electron accelerates through secondary emission electrodes (dynodes) in the electron microscope. In each step, multiple electrons are obtained for each original electron. After the multiplication is complete, a large current is collected at the anode. The gain may be on the order of 106-108, depending on the particular PMT.

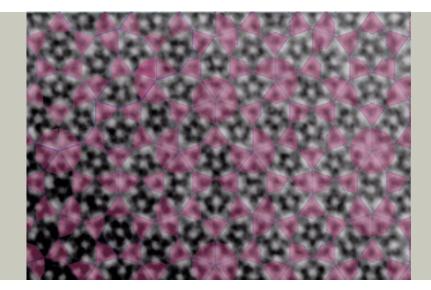
Interface with the STEM

The annular YAP and cylindrical light pipe are slid through a seal into the high vacuum STEM when needed. The mechanical movement is through a pressurized bellows that is actuated by air. The electrical supply enters through a high voltage divider and drives the PMT. [2] A scan generator is used to drive the STEM's scanning coils and a CRT in a synchronous manner. The output from the HAADF is amplified and then used to modulate the brightness levels of the CRT. A range of gray scales is the result. If the analog signal can be converted to a digital signal, the image can be filtered and refined.

HAADF applications

The advantages have been demonstrated by many investigators, including Saitoh et al.

It was suspected that $Al_{72}Ni_{20}Co_8$ crystals contained special ring patterns. After HAADF imaging, the low-Z Al atoms appeared dark while the high-Z Ni and Co appeared bright. Ring patterns that had been calculated could be superimposed, as shown in Figure 5.



References

[1] Westman S. A New Method for Particle Detection Using Scintillation Crystals in CRYRING [thesis]. Stockholm: Royal Institute of Technology; 1998. [2] Thomas G and Goringe MJ. Transmission Electron Microscopy of Metals. Wiley, New York, 1979.

[3] Saitoh K, et al. Structural study of an $Al_{72}Ni_{20}Co_8$ decagonal quasicrystal using the high angle annular dark field method. Jpn J Appl Phys, 36:L1400-L1402, 1997.

Figure 5. HAADF image of Al, Ni, and Co atoms.



E.A. Fischione Instruments, Inc. 9003 Corporate Circle Export, PA 15632 USA Tel: +1 724.325.5444 Fax: +1 724.325.5443 info@fischione.com www.fischione.com

©2013 E.A. Fischione Instruments, Inc. All rights reserved. Document Number AN007 Revision 02 11/2013