THE SCANNING ELECTRON MICROSCOPE – A TOOL FOR THE FAILURE INVESTIGATOR

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INTRODUCTION

The advent of recent TV series focusing on forensic investigations like CSI has brought many advanced analytical techniques to the public attention. Materials professionals have of course been using these techniques for many years. One of the primary tools which play an important role in both TV forensic fiction and the daily work of a professional materials failure investigator is the Scanning Electron Microscope (SEM). Today the SEM is one of the most valuable tools in both academic and commercial materials science laboratories providing unparalleled insights into the causes of materials failures. SEM's can be used to analyze a wide range of materials, components and devices and provide detailed information on their physical, chemical and electrical properties.

THE BASIC OPERATING PRINCIPLES OF THE SEM

The advancements in computer control and software interfaces means it is no longer necessary to have a detailed understanding of the theoretical aspects of how an SEM functions in order to use it in a competent manner. However, in order to maximize the quality of the information you get from the instrument and to fully understand it, it is essential that both the operator and those interpreting the results have at least a basic appreciation of how the instrument functions and the signals are generated. The first clue to these questions is in the name, Scanning Electron Microscope, i.e. an *"electron"* beam is *"scanned"* over a sample to produce a *"magnified"* image.

The basic architecture of a generic SEM is shown in figure 1. At the top of the instrument the electron gun is responsible for the generation of a tight beam of electrons. It is the use of electrons which holds the secret to the SEMs tremendous resolution. The wavelength of visible light limits the ultimate resolution of optical microscopes however; the wavelength of electrons is much smaller allowing dramatically better resolution. Until the nineties the majority of SEM's generated electrons through thermionic emission i.e. the heating of a Tungsten filament by passing a current through it. Tungsten filaments are an effective way of generating electrons and are still widely used today due to their low cost. The second generation of electron guns uses a Lanthanum hexaboride filament which has the advantage of producing significantly more electrons from a smaller virtual source, this in turn enhances both imaging quality and theoretical resolution. The third generation of electron guns utilizes the field emission effect and produces dramatically more electrons from an even smaller virtual source. State-of-the art field-emission guns (FEG) use a Schottky emitter, which is constructed from a tungsten filament with a tip as small as 10 nm, coated with a thin layer of zirconium oxide to raise electrical conductivity. As a result, this FEG-SEM provides higher spatial resolution, better reliability, improved signal-to-noise ratio, and longer life than previous technologies (N. Erdman, September 2009).

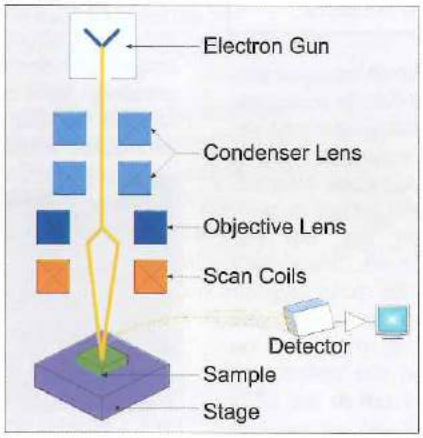

Fig 1: General layout of an SEM

The electrons are attracted out of the gun using a high electrical potential of the order of 0.1 -30keV. The electrons are focused by the various lenses as they travel down the column. The focused beam is rastered (scanned in lines across the sample then moved down to the next line in a similar way to typing on a typewriter) over the sample surface. At every point where the electron beam impinges on the sample, an interaction takes place and secondary electrons are emitted. These electrons have a low energy and are attracted to the detector using a small positively biased mesh. The detector counts the number of electrons and codes the first pixel on the monitor with a proportional grey value (i.e. zero electrons and the pixel is black and maximum number of electrons and the pixel is white). The beam then moves on in synchronization with the monitor and in this way an image is built up.

As electrons cannot travel in air, the gun, column and chamber are all held at a high vacuum, the higher the vacuum the less scattering occurs and the more sharply the beam can be focused onto the sample. Modern instruments are also equipped with powerful computers which allow automatic optimization of imaging parameters such as the brightness, contrast, focus and astigmatism.

IMAGING DEFECTS

With failure investigations we are typically examining the unknown. Therefore there is a real danger of misinterpreting imaging artifacts as real features and thus forming erroneous conclusions. In order to use the SEM to carry out a failure investigation it is essential that the operator understands the different types of imaging defects which can result from the instrument, the sample, operator's inexperience or external environmental effects. The most common types of imaging defects and some common sources are highlighted in table 1. The failure investigator needs to have both a theoretical understanding of these issues as well as the practical experience to recognize them when they arise.

SEM ANALYTICAL MODES

One of the reasons for the success of the SEM is the wide range of analytical modes which come both as de facto standards in modern instrument and the numerous optional additions which dramatically extend the instrument capabilities. Not all these techniques are of use to the failure investigators, however, the following sections outline the methods and information which can be used from some of the most useful analytical modes.

SEM ANALYTICAL MODES – SECONDARY ELECTRON IMAGING

Secondary electron imaging is the most commonly used mode in the SEM.
Secondary electrons are highly sensitive to surface topography and produce images with the highest resolution, see figure 2. In terms of failure analysis, secondary electron images can be used to investigate a wide range of characteristics from the morphology of a corrosion product, the nature and path of a crack, the failure mode of metals (e.g. ductile or brittle failure of a metal figure 3 & figure 4). This imaging mode is particularly suitable for examining

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Table 1: Common SEM imaging defects (JEOL, 2000)

Type of imaging Defect	Common Causes
Lack of sharpness	Improper accelerating voltage setting Instability of gun emission Poor alignment of apertures Insufficient astigmatism correction Improper focal depth Excessive magnification Specimen charging Magnetic or electrical fields (sample or external)
Low image quality	Inappropriate accelerating voltage selection Inappropriate probe current setting Inappropriate astigmatism correction Noise caused by excessive gain (detector) Improper contrast brightness Improper specimen preparation process Improper positioning of sample to detector
Noise	Instability of accelerating voltage Charge up of specimen surface Dusty monitor External magnetic field Mechanical vibrations
Image distortion and deformation	Specimen charge up External charge stray magnetic field Electron beam damage Deformation of specimen during preparation Image drift caused be column charging Specimen drift due to heating or charging

surface morphology due to the high resolution coupled with a large depth of field. When carrying out failure investigations it is important to realize that secondary electron imaging is a surface imaging tool and all the information in the image is coming from the top few atomic layers. Therefore if there is an oxide layer or corrosion product on the surface it may not be visible in the optical microscope but in the SEM this is all you will see and not the real sample below. As a consequence of this it is of fundamental importance that the failure investigator considers the way the image is created when making interpretations and understands exactly from where the signal is generated.

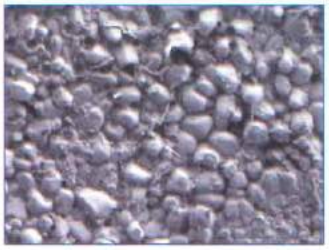

Fig 2: Secondary electron image showing the 3D grain structure of an advanced alloy (etched)

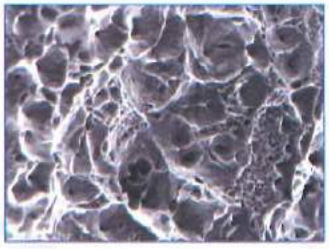

Fig 3: Secondary electron image of a dimpled ductile metallic failure

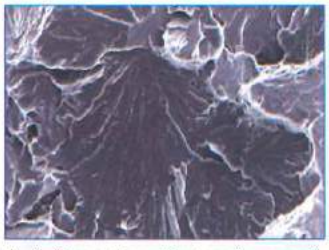

Fig 4: Secondary electron image of a brittle metallic failure

As a more specific example figure 5 shows a secondary electron image of a crack in a stainless steel heat exchanger plate. The surface of the heat exchanger plate had a number of such cracks and most of them were through thickness. Secondary electron imaging shows clearly the surface roughness (orange peel effect) surround the crack as well as the end of the crack which has blunted. The surface condition was brought about by deformation when grains rotate to orientate their slip systems relative to the stress axis. The crack tip blunting indicates considerable plastic strain on cessation of crack propagation.

Fig 5: Stainless Steel heat exchanger plate showing blunting at the crack tip

SEM ANALYTICAL MODES – LOW VACUUM IMAGING

In conventional SEMs, beam charging of insulators can be problematic; traditionally this issue has been addressed by sputtering a few nanometers of conductive coating such as carbon or gold onto the surface to prevent charging and facilitate imaging. It should be noted however, that although the coating is indistinguishable from the real sample surface, in the resolution range of the SEM, it is still not the true surface. Indeed the electrons which form the secondary images will largely be generated in the coating rather than from the sample. In most cases this will not influence the image however, in certain circumstances it can lead to misinterpretation, further the integrity of the sample is effectively destroyed and the technique is no longer non-destructive. The environmental (or variable pressure) SEM were created largely to eliminate the need to coat the sample. These instruments can image non-conductors without the need to coat them. Further, for the failure investigator these instruments operate at low pressure and so we can now image samples which are dirty, contaminated or wet without the need to clean and prepare them thus preserving a lot more evidence for the investigation. The environmental SEM is an even more valuable tool to the failure investigator as it allows direct imaging of corroded samples (figure 6), those contaminated with oil, those which are hydrated and even biomedical implants which may have living cells on their surface.

Fig 6: Corroded surface of a low alloy steel pipe

SEM ANALYTICAL MODES – BACKSCATTERED ELECTRON IMAGING

An additional SEM analytical mode used by the failure investigator is backscattered imaging. Backscattered electrons can be thought of as primary beam electrons which are reflected from the sample. These electrons have a much higher energy than those of the secondary electrons and so they can escape from much deeper in the sample. Backscattered electrons are created by the interaction of the primary beam electrons with the atoms of the sample. As a result the number of backscattered electrons is proportional to the atomic number of the sample. The contrast of backscattered images is therefore related to the elements present in the sample and only marginally by the surface topography of the sample. The failure investigator can use backscattered imaging to identify foreign materials on a surface, distinguish boundaries between layers and

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Fig 7: Backscattered electron image of the interface of a failed electronic joint

Fig 8: Backscattered image of a corrosion fatigue crack in mild steel

identify specific particles within a given sample. Figure 7 shows an example of a backscattered image from an electronic joint which failed at the interface, the micrograph clearly shows elemental contrast both at the interface and within the bulk of the solder. Figure 8 shows a crack formed by a cyclic stress/corrosion mechanism known as "corrosion fatigue". The image is formed by backscattered electrons and defines the layers of oxide in accordance with the elements present. This shows that initiation of the event took place over a long period of time and the environment changed periodically. Cessation of the cyclic event during crack propagation is indicated by the large areas of corrosion at intervals along the crack.

SEM Analytical Modes – Energy Dispersive X-ray Spectroscopy

Energy-dispersive X-ray Spectroscopy (EDS/EDX) is considered to be an essential addition to the majority of scanning electron microscope purchased for the materials laboratory. EDX is an electron probe X-ray microanalysis technique that uses characteristic X-rays that are created by the interaction of the electron beam with the elements in the sample. An EDX spectrum consists of a number of peaks which correspond to the specific elements in the sample. For an unknown sample, quantitative data can be obtained by comparing the peaks with the spectrum of a standard material. Data acquisition and analysis is a simple and relatively rapid process as the complete spectrum of energies is acquired simultaneously. The typical resolution of an EDX detector is 70 to 130eV which means that most elements can be resolved. However, there are certain combinations of elements where the X-ray lines overlap which means that an inexperience failure investigator may mistakenly identify elements which are not really present. In addition, all EDX system have light element detection limits and the quality of the quantitative data is highly dependent on a number of factors which the analyst must be aware of. Common uses of EDX by a failure investigator is a non-destructive method used to identify elemental compositions of example corrosion, plating defects and contamination issue. Figure 9 shows an EDX map of the different elemental compositions on the surface of a Copper sample that was "pitted" with zinc and lead. The copper component is coloured red, zinc as yellow and lead as blue. Figure 10 shows that EDX can be used to resolve only the copper element and clearly highlight the failure.

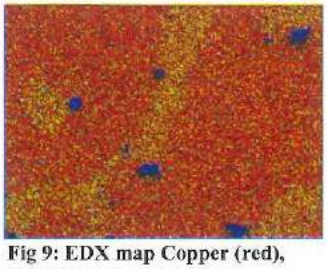

Fig 9: EDX map Copper (red), Zinc (yellow), Lead (blue)

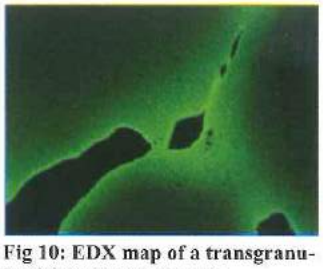

Fig 10: EDX map of a transgranular failed copper sample

SEM Analytical Modes – Electron Backscattered Diffraction

Today more and more emphasis is being put on enhancing the property sets of materials whilst simultaneously making them lighter, cheaper and more environmentally friendly. This has generated a whole host of new and high tech, materials which bring with them a whole host of new failure mechanisms. To understand these failure mechanisms and the new material characteristic, novel techniques are finding their place in the failure investigators everyday tool kit. One of the most exciting fields of science and one of the fastest developing additions to the techniques employed by SEM users is electron backscattered diffraction (EBSD). Now failure events such as transgranular and intergranular cracking can be better understood by looking deep into the crystallographic characteristics of the material itself. Using an electron beam interacting with a specimen surface, cones of diffracted electrons impinge on a phosphor screen to generate "Kikuchi Patterns". These are then analysed and provide a description of the crystal orientation in the area of scrutiny. In this way the nature of the microstructure can be described e.g. preferred direction (texture), grain boundary misorientation and intragranular misorientations (Adam J. Schwartz, 2000).

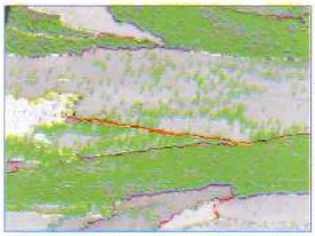

Fig 11: Deformation in individual grains of a stainless steel deposit on a mild steel substrate.

Figure 11 shows grains of stainless steel in a deposit on mild steel substrate. The green pixels show angular tilts in the crystal structure brought about by deformation. Note that not all grains show these tilts. This is because slip systems in individual grains are aligned differently due to the direction of solidification. Slip systems suitably aligned with the stress axis can exceed their critically resolved shear stress and plastically deform, whereas, grains with slip systems that do not align with the stress axis remain undeformed. This example was taken from a study that aimed to assess the effects of heat treatment on residual stress of transition a specific weld interface used in a high pressure component of a offshore oil rig. EBSD has opened many doors in the field of materials science and will continue to do so, soon it will be as unthinkable for a materials lab not to include EBSD as standard equipment when they purchase a new SEM just as it is currently the norm to equip new SEMs with EDX systems.

Closing Remarks

Modern SEM developments have allowed for examination of samples with an extremely wide range of magnifications. They also minimize sample damage, contamination and the need for tedious sample preparation. The wide range of analytical operational modes makes the SEM an essential tool for failure investigators to interrogate the root cause of materials and device failure. Although modern SEMs are easy to use a sound appreciation of instrumentation, the different analytical mode's and how the signals are generated is however essential to optimize the information obtained and accurately interpret it.