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An overview of Scanning Electron Microscopy analysis

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Abstract: Scanning electron microscopy (SEM) is a one of the pioneering technique for inspecting and analyzing of the microstructure morphology on the projected location of the processed or base material. SEM images have contributed to the research and analysis of morphological images of processed/bases of different materials. The brief history of the SEM, its development, principle, and components, are addressed in this article. An automated high-throughput online statistical analysis of the material morphology in varied, complicated SEM pictures is still difficult to do.

Keywords: Components of scanning electron microscopy, Energy dispersive spectrometer, Scanning electron microscopy.

1. Introduction

The technology for characterizing materials at the nanoscale is scanning electron microscopy (SEM). SEM may reach sub-nanometer spatial resolution1 by photographing samples with electrons rather than photons, exposing topological and compositional characteristics hidden by conventional light microscopy [1]. As a result, SEM is frequently used in various disciplines, including forensics, chemistry, physics, biology, material research, and nanofabrication. One of the most famous experimental techniques for investigating and analyzing micro- and nanoparticle imaging characterization of solid objects is the scanning electron microscope (SEM). SEMs, images provide more information regarding the topography, composition and morphology of samples, frequently used in broad range of scientifically research, engineering, industrial and commercial applications sector, produces two-dimensional images using differential charging to contrast features on a surface. Generally two types of microscopy are available; first one is optical microscopy which is the oldest and also known as light microscopy and second one is scanning electron microscopy. The main differences between both are i) Optical Microscopy works on beam of light. However, SEM using beams of electrons. ii) The maximum magnification range of Optical Microscopy is around 1000x and shows the images 400 to1000 times of the original size of the object, while using SEM magnifies the image 300,000x of the original size. iii) The visible capability of optical microscope to small and thin samples only. However, SEM endow with a more meticulous field with grey scale images. iv) SEM is more expensive and more maintenance required as compared to optical microscope. The best field emissions SEMs are approaching a resolution of 1.5 nm, and SEMs can typically have a resolution of tens of nanometers, is one of the reasons it is used for particle size investigation. The resolution of these instruments' more advanced models can reach roughly 2.5 nm [2]. SEM involves scanning the sample's surface with a low-energy electron beam emitted to the material. As the beam approaches and enters the material, several interactions occur that cause photons and electrons to be emitted from the sample surface or in a close place that causes photons and electrons to be emitted from the sample surface proximity to it [3]. Depending on the type of SEM being used, various detector types are employed to capture the receiving signals generated by interactions between the electron and the material to create an image. This instrument may also be used in conjunction with other related energy-dispersive X-ray microanalysis techniques (EDX, EDS, and EDAX) to determine the composition or orientation of certain crystals or features.

Fig.1. Optical microscopy[4]

Fig.2. SEM set up [5]

2. Brief History-Development of SEM

The barrier to better resolution imposed by the limitations of visible light was eventually removed by the development of the electron microscope by Max Knoll and Ernst Ruska at the Berlin Technische Hochschule in 1931[6]. Theoretically, 10 nm-resolution electron microscopes were being developed and manufactured by the late 1930s; by 1944, this further decreased to 2 nm. Better vacuum systems, brighter electron guns, and improvements in electron lens technology all helped to improve resolution by reducing aberrations and providing a crisper image. Therefore, improving the resolution of electron microscopes was a key factor in developing the instrument. Ladislaus L. Marton created the first micrograph of a biological specimen while working on a basic electron microscope in Brussels to research the photoelectric effect. In 1937, Berlin's Manfred Von Ardenne created the first scanning-transmission electron microscope. The first commercial electron microscope was developed by Ruska at Siemens in Germany in 1938. Each company tried to gain a piece of the European market: Philips, Siemens, and Carl Zeiss. Japanese experts began meeting in 1939 to discuss the best approach to constructing an electron microscope. Instrumentation and method were gradually improved during the 1940s and 1950s, with resolution increasing as lenses and power sources were made more reliable, and brighter electron guns produced higher-energy electrons to probe the samples [7]. Mahl's 1941 examined the sample surface of thin-paper impressions through TEM. In 1965, the first commercial scanning electron microscopes (SEMs) were released, introducing materials scientists to a new world of study. In the 1960s and 1970s, ultrahigh voltage TEM devices gave electrons higher energy to pierce thick samples more deeply. Beginning in the 1970s, the SEM transformed into a real analytical electron microscope (AEM) with the development and incorporation of additional detectors (electron microprobes, electron energy loss spectroscopy (EELS), etc.). Researchers now have access to brighter electron sources, which improve imaging and resolution. These sources are the field emission gun and the lanthanum hexaboride filament (LAB6), developed in the 1960s and 1970s.Determining the crystal structure was greatly facilitated by tilting specimen stages that allowed inspection of the specimen from various angles. Environmental electron microscopes, which enable researchers to analyze samples under better-acclimatized temperature and pressure, significantly increased the range of pieces investigated in the late 1980s and the 1990s [8]. Other trends included the development of specialized equipment for the biological or physical sciences and their simplicity for use by untrained operators. In response to this expanding industry, electron microscopes designed specifically for the integrated circuit (IC) industry were created in the 1970s and 1980s. The use of computers to automate the operation of electron microscopes and to analyze the micrographs that resulted from such operations expanded the technological possibilities, particularly when computers shrank in size in the 1980s [9].

3. Sample Preparation

First, the sample selection based on the sample's size, shape, state, and conductive properties is considered. The sample should be clean to clarify the image; for this purpose, the sample is polished with different grit sizes (100 to 3000) of emery paper and then needs diamond cloth polishing up to micron level. If the sample is non-conductive properties, need to coat it first using a sputter-coater [10]. Conductive coats include gold, silver, platinum and chromium. For mounting samples coated with a 40–60 nm thick layer of carbon or metal, such as gold or palladium, and then examined under the microscope, use a metal stub [11]. The coating is necessary because the metal film is highly stable and secondary electron yield is higher. Before placing the sample in the vacuum environment of the microscope, it must be scorched. A low accelerating voltage about 1KV is used to scan the sample and put up the electron beam at an angle. When the vacuum is reduced, more gas molecules are present in the sample chamber, which causes them to get ionized by electrons. These positive ions then travel to the specimen and neutralize the charging. Use a fixative, or, where necessary, dehydrate it by application of alcohol to maintain its structural details during the process. Otherwise, water vaporization can obstruct the electron beam, affecting the clarity of the image. Biological samples are chemically fixed in the usual way and dehydrated by using acetone or ethanol and letting it evaporate. The sample is then dried at a crucial stage to reduce specimen distortion, which typically results from drying pressures. For dry samples, there is no requirement to perform this process.

4. SEM Components

The components of SEM' which is used discussed below:

i) Electron source
ii) Condenser lens
iii) Scanning coils
iv) Objective lens
v) Secondary electron detector

The source for the electron production is typically a field emission gun or a tungsten filament lamp or Cerium hexa-boride cathode at the zenith of the microscope's column. A significant electric field is produced by the field emission gun, which attracts electrons away from their atoms and produces high-resolution images. Therefore, for a field emission gun to maintain a clean tip free of impurities and oxides, extremely high vacuum are necessary [12]. By heating a filament (cathode) consisting of a thin tungsten wire (approximately 0.1mm), thermoelectrons are released from the filament (about 2800K). When the anode is given a positive voltage, the thermoelectrons are grouped into an electron beam and flow into the metal plate (the anode). The electron beam passes through a hole created at the anode's centre. The lifetime of tungsten filament is about 100 hours in intense conditions. Ceriun hexaboride cathode produced ten times brightness of the tungsten filament lamp, improved 15 times lifetime, and also provides the better signal to noise ratio[13].

The condenser lens is positioned underneath the electron gun. The beam's size and electron number are controlled via the condenser lens. The resolution of the image will depend on the beam size. The "aperture" has a tiny metal plate-built hole in it. The electron beam that passes through the condenser lens illuminates this aperture plate [14].

The beam is rastered onto the sample using scanning coils. Apertures and lenses are frequently used together to regulate the beam size.

The objective lens is the last lens in the sequence of lenses that create the electron beam. Objective lens plays a significant role in determining the SEM resolution. The beam is focused to a very small spot on the sample by this lens because it is the one closest to the sample. The diameter of the electron probe is ultimately determined by the objective lens, which is used for focusing. Despite all of the work done before the objective lens's action, an optimally-fine electron probe cannot be created if the objective lens's performance is poor. Making the best-performing objective lens is essential.

The specimen emits secondary electrons, detected by the secondary electron detector and strikes the scintillator, drawn to the high voltage and emits light. This light is transmitted toward a photo-multiplier tube (PMT) and transformed into electrons, and the resulting electric signal is amplified [15].

5. Principle of Sem

A highly focused electron beam is used to scan an object's surface with an SEM, a type of electron microscope, to create images of the object. In the process, electrons interact with an object's atoms to produce signals that reveal the object's composition and topography. The configuration of the constituent atoms is investigated by using 2D beam scanning on the sample surface and picture collection from gathered secondary electrons. The electron beam produces the scan pattern, and the image is created by combining the beam's position with the detected signal. [16]

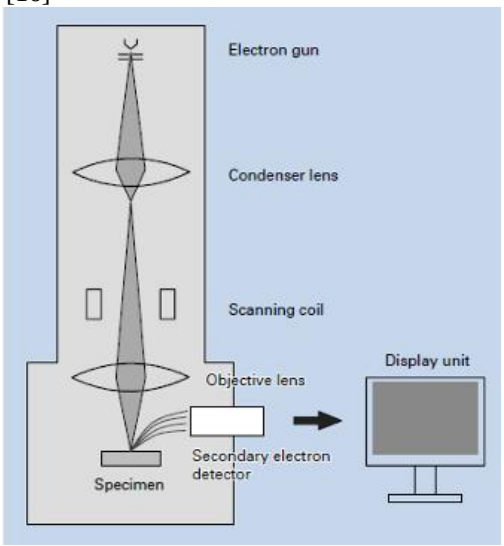

Fig.3. Schematic diagram of scanning electron microscopy

5.1 Scanning Process and Image Formation

A tungsten filament cathode-equipped electron gun emits an electron beam with an energy range between 0.2 and 40 keV is normally produced by an electron gun in a conventional SEM. One or two condenser lenses focus the electron beam, which normally has a spot with a diameter between 0.4 nm and 5 nm. In the electron column, often in the last lens, the beam travels via pairs of scanning coils or deflector plates that bend the beam in the x and y axes such that it scans in a raster pattern over a rectangular area of the sample surface [17]. The

interaction volume is a teardrop-shaped specimen area where the primary electron beam interacts with the sample, losing energy through repeated random scattering and absorption. The beam is deflected as it passes via scanning coils or deflector plates, allowing it to scan over the sample surface in a raster pattern. Due to the energy exchange between the electron beam and the sample, high-energy electrons are reflected by elastic scattering, secondary electrons are released by inelastic scattering, and electromagnetic radiation is released [18]. The sample and the electron beam exchange energy, which produces electromagnetic radiation, secondary electrons, high-energy electrons, and electromagnetic radiation, all of which can be detected by specialized detectors. Images showing the distribution of specimen current can also be produced by detecting the beam current absorbed by the specimen[19]. The signals are amplified by several electronic amplifiers and presented on a computer monitor as fluctuations in brightness. The resulting image is a distribution map of the intensity of the signal being emitted from the scanned area of the specimen since each pixel of computer video memory is synced with the beam's position on the specimen in the microscope [20]. Older microscopes take pictures on film, but most modern equipment takes pictures on a computer.

5.2 Advantages

This test gives data that can be used to characterize microstructures, including fracture, corrosion, grains, and grain boundaries, at a resolution of as low as 15 nanometers on a digital scale and imaging in all directions through x-y-z (3D). Applicable for almost all kinds of samples (Both conducting and non-conducting) and all imaging is calibrated to a traceable standard, making it simple to apply analysis to recorded images to determine coating thicknesses, grain sizes, and particle sizes.

5.3 Disadvantages

Even though it is a great chemical analysis and surface typography test, some samples are unsuitable for SEM with EDS. Here are a few justifications for thinking about various materials analysis types.

- A vacuum-like setting: SEM samples often need to be stable in a vacuum. Higher pressures can, however, be employed to image nonconductive, volatile samples that are sensitive to vacuum.
- It is possible to find artefacts: Before testing, good insulator samples must be coated, typically with gold or carbon. However, this procedure might produce artefacts. Nevertheless, preparation and analysis by an experienced SEM testing lab guarantee that these artefacts have little to no influence on test findings.

6. Energy Dispersive Spectroscopy (EDS)

X-rays are one of the emissions produced whenever the electron beam strikes a sample target [21]. The distinctive x-rays of various elements are separated into an energy spectrum using an energy-dispersive (EDS) detector. EDS system software again examines the energy spectrum to ascertain the abundance of particular elements [22]. EDS may be used to build element composition maps across a much larger raster region and to determine the chemical composition of the samples down to a spot size of a few microns. These abilities offer basic compositional details for a range of materials.

6.1 EDS Working

EDS systems are frequently incorporated into SEM or EPMA equipment. Energy-dispersive X-ray spectroscopy sometimes referred to as EDS, EDX, or EDXA, is an effective method that enables the user to examine the elemental content of a selected sample [23]. The main mechanism by which EDS works is the ability of high-intensity electromagnetic radiation to eject "core" electrons from an atom. A sensitive x-ray detector, a liquid nitrogen dewar for cooling, and software to capture and analyze energy spectra are all components of EDS systems [24]. At the end of a long arm that is also cooled by liquid nitrogen, the detector is installed in the sample chamber of the main instrument. The most popular detectors are Si(Li) crystals that run at low voltages to increase sensitivity, but recent developments in detector technology have made it possible to use so-called "silicon drift detectors" that run at greater count rates without liquid nitrogen cooling [25]. In an EDS detector, a crystal absorbs incoming x-ray radiation through ionisation, releasing free electrons that become conductive and provide an electrical charge bias [26]. Thus, the energy of each unique x-ray is converted by x-ray absorption into electrical voltages of the corresponding size, and the electrical pulses match the distinctive x-rays of the element. When performed in "spot" mode, a user can quickly acquire the entire elemental spectrum. EDS is a great survey method to quickly detect unknown phases before quantitative analysis since supporting software makes it possible to identify peaks easily [27-29]. EDS can be employed in a semi-quantitative mode to estimate chemical composition by the peak-height ratio of a standard related to a standard, and EDS can be employed in a semi-quantitative mode.

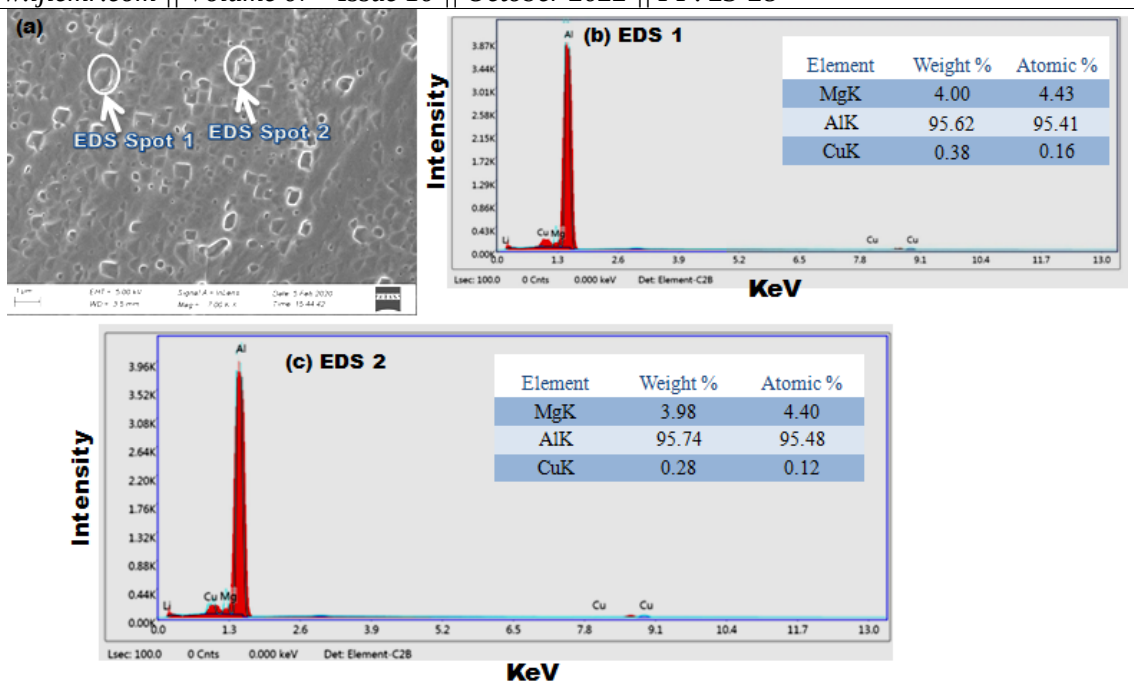

Fig.4. SEM and EDS analysis of friction stir welded AA2050-T84 alloys [30]

7. Conclusion

Different modes and procedures for obtaining high-quality imaging of various samples are available with scanning electron microscopy. This paper briefly introduces the basic knowledge of scanning electron microscopy. It can assist SEM users and researchers in quickly mastering the fundamental procedures needed to investigate the target point of samples. Users can determine the nature of processed or bare materials by learning the fundamentals and being familiar with the setups of the microscope.

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