Electron Microscopy Facility for Research and Services in the Malaysian Nuclear Agency towards TSO

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Abstract

Scanning Electron Microscope FEI-Quanta 400 (SEM) made in the USA was commissioned in late 2003. This equipment is used in many areas of materials science, metallurgy, engineering, electronics, medicine, agriculture, biology and so on. This facility has helped the researchers in conducting research in their respective fields as well have been providing services to agencies, institutions, industries and local industry. Since 2004, there were 81 projects and 5000 samples analyzed using this facility in Malaysian Nuclear Agency, while 23 companies and 900 samples were from various agencies .In addition, revenue derived from these services has able to provide for the maintenance of this equipment. SEM is an important step in the nuclear material testing process. Nuclear material can be inspected for its performance by getting information from its morphology micrograph by using SEM. It opens up a whole new world that is unseen by the naked eye.

Abstrak

Mikroskop Imbasan Elektron jenis FEI–Quanta 400 (SEM) buatan USA telah ditauliahkan pada akhir tahun 2003. Alat ini banyak digunakan dalam bidang sains bahan, kajilogam, kejuruteraan, elektronik, perubatan, pertanian, biologi dan sebagainya. Kemudahan ini telah banyak membantu pegawai-pegawai penyelidik dalam menjalankan penyelidikan dalam bidang masing-masing disamping telah memberi perkhidmatan kepada agensi-agensi, insitusi-institusi luar serta industri-industri tempatan. Semenjak tahun 2004, terdapat lebih kurang 81 projek dan 5000 sampel telah dianalisa dengan menggunakan kemudahan ini di Agensi Nuklear Malaysia, manakala lebih kurang 23 syarikat dan 900 sampel dari berbagai-bagai agensi luar. Di samping itu, hasil pendapatan perkhidmatan yang diperolehi dari perkhidmatan luar juga telah dapat menampung keperluan untuk penyelenggaraan peralatan ini. SEM juga merupakan satu langkah penting dalam pencirian bahan nuklear. Bahan nuklear dapat dikaji prestasi pencapaiannya melalui mikrograf morfologi bahan tersebut yang dapat diperolehi menggunakan SEM. Ia membuka seluruh dunia baru yang tidak terlihat oleh mata kasar.

Keywords/Kata kunci: Scanning Electron Microscope, FEI –Quanta 400, Nuclear Material Morphology Study.

INTRODUCTION

A scanning electron microscope (SEM) is a type of electron microscope that images a sample by scanning it with a high-energy beam of electrons. It emitted electron from sources on a surface of the specimen to allow the surface morphology can be magnify up to 500,000 times greater than the original size.

A lot of studies have been done on the materials that are used in the construction of nuclear plant. A wide range of materials is used including different types of rubber, like Viton and EPDM, and metals, like stainless steel and Zirconium alloy.Most of the metals are used in the piping and infrastructure of the reactor. Zircaloy II and Zircaloy IV, for example, are used in the fuel rods in the core. Stainless steel and its different alloys are used primarily in piping, pressure vessels, and the super structure of the reactor building. The piping carries the water and steam around to the different areas of the reactor. The pressure vessels interconnect the pipes and help to control pressures. The structure houses the core and prevents radiation exposure in the event that there is a core melt down.EPDM rubber is used primarily outside of the reactor as a seal between and around pipes. The rubber also dampens the vibrations caused by the superheated water flowing through steam generators. EPDM rubber is also used in gaskets, which seal some doors airtight.

The core is where the nuclear reactions occur and where the materials are under the most strenuous conditions in the plant. To study the materials degradation, they are submitted to conditions similar to those they will experience while the plant is in operation. After these materials have been submitted to these conditions a number of different tests can be done on to determine changes in the materials properties. The results of these tests are used to determine where a material is placed in the plant or reactor. SEM is the most common equipment use to determine microscopic cracks in the metal or other object formed by this degradation.

SEM facility at Nuclear Malaysia was first introduced at 1986, where several studies in the field of minerals, metallurgy, medicine, pharmacy, agriculture, biology, semiconductor and environmental analyzed by this equipment. Over time, this system has experienced problems starting from malfunction of the vacuum system and followed by unclear imaging at high magnification. Camera system for capturing the images needed a higher provision for repair. Maintenance and repair of the equipment was satisfactory at the early stages, but had many problems arise regarding purchasing of spare parts and service given by maintenance engineer. To overcome this problem, a new system has been applied.

The first scanning electron microscope installed at Nuklear Malaysia is Philips 515 SEM with Energy Dispersive X-Ray Microanalysis of EDAX-DX4. 515 SEM is equipped with PV9100 EDAX microanalysis was purchased in 1986 with the price of RM 387,576.00. PV9100 EDAX microanalysis software malfunction in late 1987 and several improvements have been made by the Nuklear Malaysia's engineers at that time but to no avail. The microanalysis was later changed to EDAX-DX4 on 10/30/1997 with the value of RM142, 710.00. The vacuum pump was the last problem that can not be repaired and had to be disposed. History of repairs to the old system is shown in Table 1. It has been replaced by a new type of FEI-Quanta 400 made the USA was commissioned in late 2003. The users of this new equipment are composed of researchers from various divisions in Nuklear Malaysia, universities, other Research Institutes and local industries.

Table 1: Maintainance table of Philips SEM 515

Date of Malfuction	Types of Malfunction	Repairer	Repair Cost (RM)
12.11.1987	- \`POWER ON' switchmalfunction - Monitor for EDAX - Stabilization malfunction	- Nuklear Malaysia's Technician - Supplier's Engineer	
7.3.1987	- EDAX	Supplier's Engineer	3,570.00
1.10.91	- Thermal switch	Supplier's Engineer	1,499.60
24.31.94	- SEM Monitor Display		
8.6.95	Tungsten Filament break		
17.8.95	Tungsten Filament break		
15.9.95	Tungsten Filament break		
5.10.95	Tungsten Filament break		
17.10.95	Tungsten Filament break		
26.10.95	Tungsten Filament break		

2.11.95	Tungsten Filament break		
24.11.95	Tungsten Filament break		
12.1.96	Tungsten Filament break		
12.2.96	Tungsten Filament break		
30.10.97	- EDAX hard disk - SEM, diffusion pump - Display module	Supplier's Engineer	-22,190.00 Change display module and serviced diffusion pump -142,710.00 Change new EDAX DX4
20.6.98	Heater for Diffusion pump	- Nuklear Malaysia's Technician	
18.9.98	hard disk EDAX DX4	Supplier's Engineer	
9.11.98	hard disk EDAX DX4	Supplier's Engineer	2400.00
19.2.99	hard disk EDAX DX4	Supplier's Engineer	
26.5.99	Loss of image because of power board	Supplier's Engineer	4,308.40
26.9.99	hard disk EDAX DX4		
2.10.99	Monitor Display		
4.11.99	Heating element for Diffusion pump (break)		2,500.00
12.6.2000	Heating element for Diffusion pump (break)		
28.7.2000	Heating element for Diffusion pump (break)		
1.8.2000	Heating element for Diffusion pump (break)		4,250.70
10.10.2000	Aperture at electron gun		1,439.00
22.11.2000	Water chiller		2,500.00
17.1.2001	Heating element for Diffusion pump (break)		
14.3.2001	Leaking in electron gun column		
6.2.2002	- No contrast image on monitor display - No grey scale - Leaking in electron gun column		
17.10.2002			- 7,400.00 moving cost SEM to Block 34,
2.1.2003	- no vacuum if filling Liquid nitrogen in dewar - Leakage Liquid nitrogen dewar - No contrast image on monitor display - Leaking in electron gun column		- 77,150.00 N_2dewar leak repair - 40,000.00 EDAX camera system

SEM SERVICES FROM 2004-2010

SEM in Nuklear Malaysia has been used in various field of research such as Biology, Medical, Materials Science, metallurgy, Semiconductor, Geology etc. Some of the capabilities of SEM are:

i) **Surface Topography** - 3-dimension surface of sample can be obtain. The surface can be rotated and tilted at any angle required to get a clearer image.

ii) **Micro-Chemical Analysis**- Content elements of metal and non-matrix and phase seen on the surface can be determined either qualitatively and quantitatively.

iii) **Structural Analysis** - With the additional attachment -Scanning Transmission Electron Microscopy (STEM), the crystal structurestudy can be done using electron diffraction methods.

iv) **Dynamic experimental**- Changes in shape and microstructure of a material that is stretching (tensile) can be viewed simultaneously during the experiment when the device is equipped with thetensile stage.

v) **Internal images**- Microstructure in materials usingdark field image and bright-fieldimages can be seen when it is equipped STEMaccessories. Images obtained almost the same as transmission electron microscopy.

vi) **Quantitative Phase and image Analysis**- When the device is equipped with 'Image Analysis software, the work of analysis such as measuring the size, number of items, the determination of the percentage content of phase and so can be done.

There are a total of 81 research projects that have benefited from Nuklear Malaysia’s SEM services with 43 Nuklear Malaysia’s researchers handing over 3270 samples for SEM analysis from 2004 to 2010. Details of these figures are shown in table 2 and 3. Table 4 shows details of the 23 companies, universities and other research institutes been charge for SEM analysis services for a total of 999 samples sent from 2004 to 2010.

BASIC SCANNING ELECTRON MICROSCOPY

Electron source - An electron beam is emitted from an electron gun fitted with a tungsten filament cathode. Tungsten is normally used in thermionic electron guns because it has the highest melting point and lowest vapor pressure of all metals, thereby allowing it to be heated for electron emission, and because of its low cost. At high temperature, electron is freed and focused by condenser lenses to a spot about 105nmto 200nm. This electron beam is then focused to objective lens with opening size of 100µmto 300 µm. Objective lens s used to produce final spot size interaction to the sample. Final spot size is most important in producing a good resolution of the sample. The best resolutions produced by electron microscope nowadays are from 3.5nmto 5 nm.

Electron Scanning - Electrons emitted to the lenses will be reflected to a point on the surface of the specimen. Reaction at the surface occurs through scanning electron beam mechanism of the spot. It was scanned by a scanning generator. There are two types of scanning namely linescan and frame scan.

Interaction between electron and sample- Surface morphology is obtained as a result of the reaction end point is scanned byelectrons the dots on the surface of the specimen.The energy exchange between the electron beam and the sample results in the reflection of high-energy electrons by elastic scattering (backscattered electrons), emission of secondary electrons by inelastic scattering and the emission of electromagnetic radiation.

Electron detector- The electrons produced from the reaction with the surface of the specimen as backscattered electrons, secondary electron, plasma, ionization, x-ray, bremstrahlung and phonons lead to the detection of electrons. Electron detector consists of an electron collector that is 'Faraday cage', scintillator,'light-pipe' and the photo-multiplier. Solid-state detectoris the usual detector used in SEM to collect electron. Secondary electrons and backscattered electrons coming from the specimen will reach and pass the the 'Faraday cage' pointing to the screen scintillator. The electrons are accelerated by the voltage +10kV will react with the photon-photon scintillator produce. Signal produced will be the'light-pipe' to the photo-multiplier to produce currents. Current output from photo-multiplier is used to modulate the intensity of the monitor display (CRT) and thus produce an image. Any signal processed by the electron detector will be sent to the display monitor. With this image in relation to the morphology of the specimen surface can be seen on the screen.

Table 2: Nuklear Malaysia Projects that uses SEM from 2004 to 2010

No.	Project Name	Responsible Officer
1.	Characterization of PVA/conducting polymer compound prepared by gamma radiation	Dr. Khairul Zaman Hj Mohd Dahlan
2.	Development of pharmaceutical film coating using sago starch derivatives as coating agent.	Dr. Kamarudin Hashim
3.	Chitosan derivative for wound dressing	Dr. Kamarudin Hashim
4.	Develop radiation protection glove	Pn. Noriza Md. Isa
5.	Hydro gel	Dr. Norimah Yusof
6.	Methanol fuel cell	Dr. Khairul Zaman Hj Mohd Dahlan
7.	Physicochemical properties of gamma Irradiated bacterial cellulose	Dr. Zulkifli Hashim
8.	Development of solder material for jewelry application	En. Hishamuddin Hussin
9.	Radiation grafting of fuel cell membrane	Dr. Khairul Zaman Hj Mohd Dahlan
10.	Verification & assessment of aluminum alloy SACP	Cik Siti Radiah Kamarudin
11.	Development of wound management product from chitosan characters	Pn. NorzitaYaacob
12.	Investigation of hydro gel as scaffolds for cartilage tissue engineering	Dr. Norimah Yusof
13.	Rawatanpermukaandenganmenggunakantekniksinaran	En. Mohd. Sufian Alias
14	Development of a pilot plant for the treatment of Dehpa/waste	Dr. Syed Malik Syed Zain
15.	Conducting polymer composite film	En. Mohd. HamzahHarun
16	Utilization of pearl seal for radionuclide xxx	Dr. Zaidi
17.	Carboxyl methyl cellulose from kenaf core	Dr. Kamarudin Hashim
18.	Effect of fly ash and electron beam irradiation on NR/LLDPE Composite	Dr. Dahlan HJ. Mohd.
19.	Develop of nano composite for LAM	En. Meor Yahya Razali
20.	Synthesis of nano structural organic-inorganic hybrid material from palm oil.	Dr. IshakManaf
21.	Development of bone graft substitute	Dr. Idris Besar
22.	Bio-form	Dr. Zulkifli Ghazali
23.	Palm oil acrylics	En. Mohd. HelmiMahmood
24.	Thermoplastic Elastomeric	Dr. Chantara Thevy Ratnam
25.	Bone cements	Dr. Yusof Abdullah
26.	PVC-ENR blend	Pn. Marina Talib
27.	Isotope technique for ground water . Contamination studies in urbanism and industrial areas	Dr. Mohd. Tadzaabd. Rahman
28.	Development of nano HA (Hydro cepatite) ceramics	Dr. Yusof Abdullah
29.	Penghasilan Amnion- bank tisu	Dr. Norimah Yusof
30.	Development of PVC/ENR compound for automation industrial paths.	Dr. Chantara Thevy Ratnam
31.	Preliminary study on development of environment friendly and biodegradable EFB fiber composite using sago starch as binder	Dr. Khairuzaman
32	Development of radiation cross linked biodegradable foams.	Dr. Zulkifli Ghazali
33.	Polymer/clay nano composite	Dr. Jamaliah Shariff
34.	Pembangunan hidrosapetiteporossebagaitulanggrafgantian	CikRohaidaChekHak
35.	Development of Aluminum composite incorporating fly ash reinforcement material using stir casting technique.	Dr. Mohd. Bin Harun
36.	Bone graft development	Pn. RusnahMustaffa
37.	LENRA sebagaipenserasidalampolimeradunan (NR/HDPC)	Dr. Dahlan Hj Mohd
38.	Elastomer Termoplastik (TPE) berasaskangetahasli	Dr. Dahlan Hj Mohd

39.	Synthesis of zeolite from kaoline	Julie AndriyaniMurshidi
40.	Degradable of packaging plastics	Dr. Kamarudin Hashim
41.	Metal recovery from waste	En. Megat Harun Al Rashid Megat Ahmad
42.	Polyethylene –oil palm trunk composite	Dr. Khairul Zaman Hj Mohd Dahlan
43.	Preparation of hydro gel from carboxyl methyl cellulose	Dr. Kamarudin Hashim
44.	Pembangunan selulosa bacteria tersinaralur electron sebagai system penyampaiandadahterkawal.	PN. Nadia Halib
45.	Reinforcement of PCL by synthetic polymer grafted OPEFD	Dr. Khairul Zaman Hj Mohd Dahlan
46.	Sago starch hydro gel, chitosan hydro gel	Dr. Kamarudin Hashim
47.	Recycling of rubber by using Elektron Beam Radiation	Dr. Chantara Thevy Ratnam
48.	Application of nuclear Analytical Technique	Dr. SuhaimiHamzah
49.	Dib lock copolymer	Dr. Dahlan Hj Mohd
50	Development of a method for radium removal from industrial waste water using humid acid fixed onto immobilized materials.	Zalina Laili
51.	Polymer blends of polycaprolactone / poly vinyl chloride composite	Dr. Khairul Zaman Hj Mohd Dahlan
52.	The effect of irradiation on nylon 66 with copper filler reinforcement	Dr. Khairul Zaman Hj Mohd Dahlan
53.	Development and the effect of radiation on microbial fuel cell in generating bio energy from agriculture waste xxx	Dr. Jong BorChyan
54.	Radiation processing of nano materials	Mohd. Hilmi Mahmud
55.	Preparation of cat ion exchange resin	Norhasimah
56.	Industrial organic wastewater treatment using electron beam machine	KhomsatonabuBakar
57.	Effect of crosslink agent on CMC hydrogel	Dr. Kamarudin Hashim
58.	Development of conducting paste for LTCC application	Dr. Che Seman Mahmood
59.	Development of TLD using Nano size do pant material	Dr. Wan Shafiee Wan Abdullah
60	Menghasilkan resin getahasliberpengisi silica teknik sol-gel	Dr. Dahlan Hj. Mohd.
61.	Development of sheet and paste from biomedical grade chitosan derivatives for wound management application.	Pn. NorzitaYaacob.
62.	Preparation of NR based nano sized materials using sol gel technology.	Mahathir Mohamad
63.	PenggunaanTeknologysinaranuntuksalutankeatas MIDF	Dr. Nik Ghazali Nik Salleh.
64.	Synthesis of titania Nano tube from local waste	Dr. Meor Yusof Meor Sulaiman
65.	Development of sorption material for removal of radium from wastewater	Cik Zalina Laili
66.	High purity alumina from aluminum can	Dr. Meor Yusoff Meor Sulaiman.
67.	Development of scaffold porous HA	Pn. RusnahMustaffa
68.	Investigation of hydro gel as ECM scaffold for cartilage tissue engineering	Dr. Norimah Yusoff
69.	Mechanism of wound healing using hydro gel based bio resources	Dr. Norimah Yusoff
70.	Development of nano HA hydroxyl apatite ceramics	Dr. Yusofabdullah
71.	Dielectric characterization of perouskite materials	Pn. Norhayati Alias
72.	Catalyst assisted growth of Si Nanowires	Dr. Ng Inn Khuan
73.	Characterization and enhancement of natural barrier for landfill	Dr. TadzaAbd. Rahman
74.	Polyurethane oil palm trunk composite	Dr. Khairul Zaman Hj Mohd Dahlan
75.	Degradability of packaging plastics	Dr. Kamarudin Hashim.
76.	Preliminary study on polymer for radiation grafti9ng membrane	Ting Teo Ming
77.	Bio- material amnion	Dr. Norimah Yusoff
78.	Biomass refinery industrial complex project D-functional food	En. Ahmad Sahali Mardi
79.	Development of Vanila product/gaharu product	Dr. Seri CempakaMohdYusof
80.	Development of fly-ash reinforcement automotive friction material	Dr. Mohd. Bin Harun
81.	Study on the damage mechanism in fire flare boom at ambient and elevated temperature.	Dr. Mohd. Bin Harun

Table 3: List of Nuklear Malaysia's uses SEM service from 2004 to 2010

No.	Officers	No. of samples
1.	Dr. Kamaruddin Hashim	260
2.	Dr. Norimah Yusoff	120
3.	Dr. Dahlan Mohamad	110
4.	Dr. KhairuzamanMohdDahlan	100
5.	Dr. Jamaliah Shariff	102
6.	Dr. chantara	145
7.	Dr. Meor Yusoff Meor Sulaiman	260
8.	Dr. Kok Kuan Ying Dr. Ng Inn Khuan	289
9.	Nur Ubaidah Saidin	130
10.	YusmaazranYusof	150
11.	Siti Radiah Kamaruddin	80
12.	NorzitaYaakob	110
13	Rusnah Mustaffa	90
14.	Megat Harun al Rashid	80
15	Masliana Muslimin	70
16.	Hishamuddin Hussin	60
17.	Che Rohaida Che Hak	100
18.	Julie andrianyMursidi	60
19.	Dr. Yusof Abdullah	65
20	Dr. Muhamad Daud	50
21.	Dr. KamarudinSamuding	55
22.	Wilfred	52
23.	Dr. Wan shaffie Abdullah	45
24.	Pn. Zainon Othman	40
25.	Pn. Marina Talib	30
26	Nadia Halib	23
27.	Norhashimah	25
28.	Hafizal Yazid	30
29	Noorhayati Alias	23
30	Dr. Zulkifli Ghazali	20
31	Dr. IshakManaf	40
32	ChooThye foo	18
33.	Dr. Zainuddin Othman	26
34	Siti Selina	20
35.	Zalina Laili	25
36.	Mohd. Iqbal	20
37	Zaifol	26
38.	Fahmi	45
39.	Nurazila Mat Zali	20
40.	Dr. SuhaimiHamzah	20
41.	Dr. Che Seman Mahmood	30
42.	Dr. Mohd. Bin Harun	200
43	Dr. Zaidi	10

Table 4: List of companies that requested SEM services

No.	Companies	No. of sampels
1.	Wembley Rubber Product (M) Sdn. Bhd., SalakTinggi, Sepang, Selangor	50
2.	TenagaNasionalBerhadRemaco Sdn. Bhd.	60
3.	TenagaNasionalBerhad Research	250
4.	UNITEN	85
5.	PCO lite	20
6.	Universiti Putra Malaysia	120
7.	UniversitiKebangsaan Malaysia	65
8.	Petronas Research Services Sdn. Bhd.	30
9.	UniversitiTeknoloji Malaysia	20
10.	Universiti Malaysia Sarawak	4
11.	Universiti Malaya	20
12.	ESTOP	8
13.	PERMINTEX	4
14.	Muzium Negara	22
15.	Universiti Islam Antarabangsa Malaysia	10
16.	Vinyl Chloride (M) Sdn. Bhd.	25
17.	Ethylene-Polyethylene (M) Sdn. Bhd	24
18	Petronas gas	20
19	PetronasCarigali (M)	19
20	SGS	15
21	Pengkalan Offshore Sdn. Bhd.	25
22	Total Sterling (M) Sdn. Bhd	23
23	Telekom Malaysia	80

SAMPLE PREPARATIONS

For conventional imaging in the SEM, specimens must be electrically conductive, at least at the surface, andelectrically grounded to prevent the accumulation of electrostatic charge at the surface. Metal objects require little special preparation for SEM except for cleaning and mounting on a specimen stub. Nonconductive specimens tend to charge when scanned by the electron beam, and especially in secondary electron imaging mode, this causes scanning faults and other image artifacts. They are therefore usually coated with an ultrathin coating of electrically-conducting material, commonly gold, deposited on the sample by low vacuum sputter coating.Gold has a high atomic number and sputter coating with gold produces high topographic contrast and resolution. Coating prevents the accumulation of static electric charge on the specimen during electron irradiation. There are two reasons for coating, even when there is enough specimen conductivity to prevent charging, are to increase signal and surface resolution, especially with samples of low atomic number (Z). The improvement in resolution arises because backscattering and secondary electron emission near the surface are enhanced and thus an image of the surface is formed.

For SEM, a specimen is normally required to be completely dry, since the specimen chamber is at high vacuum. Hard, dry materials such as wood, bone, feathers, dried insects or shells can be examined with little further treatment, but living cells and tissues and whole, soft-bodied organisms usually require chemical fixation to preserve and stabilize their structure. The chemically dried specimen is usually mounted on a specimen stub using an adhesive such as electrically-conductive double-sided adhesive tape, and sputter coated with gold before examination in the microscope.

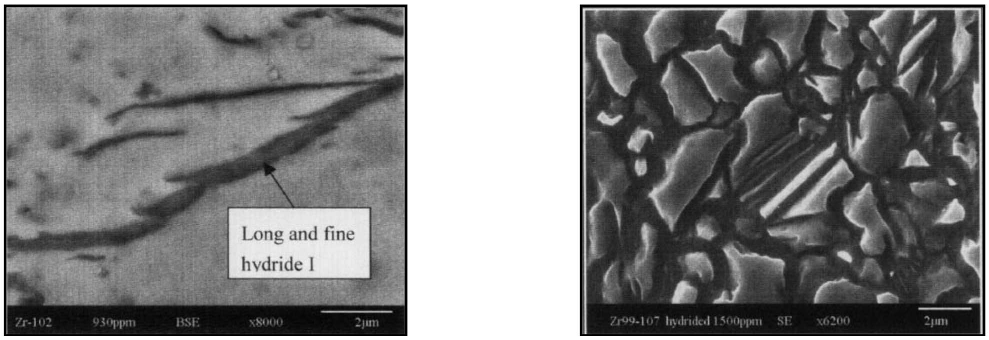
An alternative to coating for some biological samples is by using specialized SEM instrumentation that is "Environmental SEM" (ESEM) that operated at low voltage. Environmental SEM instruments place the specimen in a relatively high pressure chamber where the working distance is short and the electron optical column is differentially pumped to keep vacuum condition adequately low at the electron gun. The high pressure region around the sample in the ESEM neutralizes charge and provides an amplification of the secondary electron signal. Low voltage SEM of non-conducting specimens can be operationally difficult to accomplish in a conventional SEM and is typically a research application for specimens that are sensitive to the process of applying conductive coatings.

Back scattered electron imaging, quantitative X-ray analysis, and X-ray mapping of geological specimens and metals requires that the surfaces be ground and polished to an ultra smooth surface. Metals are not generally coated prior to imaging in the SEM because they are conductive and provide their own pathway to ground. Study of fractured surfaces can be done on a light microscope or on an SEM. The fractured surface is cut to a suitable size, cleaned of any organic residues, and mounted on a specimen holder for viewing in the SEM.Metals, geological specimens, and integrated circuits all may also be chemically polished for viewing in the SEM.Embedding in a resin with further polishing to a mirror-like finish can be used for both biological and materials specimens when imaging in backscattered electrons or when doing quantitative X-ray microanalysis.

NUKLEAR MATERIALS STUDIED USING SEM

The need for better performance of nuclear plant material assemblies and structure, mainly regarding to their corrosion resistance and irradiation embrittlement has led to extensive research and development efforts in order to improve the properties of nuclear material especially Zr alloys. This nuclear material has widely being investigated through advance composition and thermomechanical processing to optimize the microstructure within the corresponding specification [1]. Zircaloy –IV for example is use as cladding material for fuel elements in the nuclear industries [2]. SEM contributes towards a better understanding of the zircaloy characteristic after being tested with a nuclear plant operating conditions.

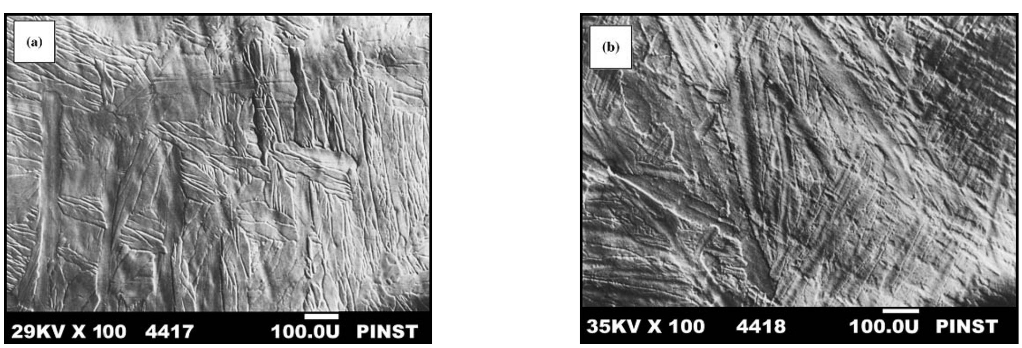
Milena Velevaet. al. had studied the microstructure of a stress-relieved annealed (SRA) and recrystallizedZircaloy. The study of morphology of the hydrides in two alloys at different hydrogen contents were also been done using SEM. The deformation process of these hydrides samples was also using SEM in-situ tensile testing [1]. In the study of SRA material done by Milena Velevaet. al, two types f hydrides were observed: thick, short hydrides and fine long hydride, there was no subplatelet structure observed, due to the preferential etching of the other hydride particle. The proportional of these hydrides increases with increasing hydrogen content. Several configuration of subplatelet structure could be found in the case of fine and long hydrides as shown in Fig. 1(a). In the recrystallized material the thick and short hydrides are in general located at the grain boundaries and follow their curvature. The hydrides platelets become thicker and more often intergranular for higher hydrogen contents. Observation n the highly hydride sample showed that the hydride form a continuous network along the grain boundaries (Fig.1 (b)).



(a) (b)

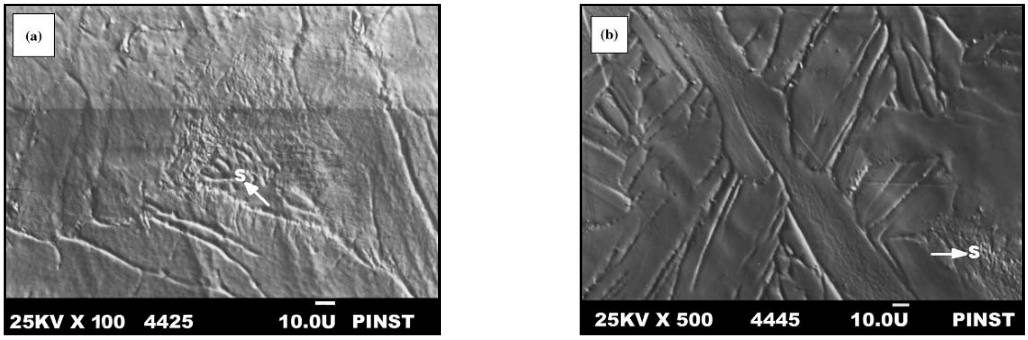
Figure 1: (a)Morphology of hydrides in SRA material, (b) Morphology of hydrides in recrystallized material

M. Ahmad et. al. has use SEM attached energy dispersive system (EDS) with in their study the role of segregation on microstructure and mechanical properties of Zircaloy-4 [3].SEM examination of the samples, taken from the ingots designated as A and B, revealed a marked difference betweenthe grain structure of the two alloys as shown in Fig.3(a) and(b). Alloy A has generally a parallel plate type structure withthe “basket-weave” pattern in some localized areas, whereas alloy B has mostly the “basket-weave” type structure throughout the material. The size of the plate in alloy B is moreuniform than that in alloy A.Another difference between the microstructures of the two alloys is the presence of localized areas of segregated phases. These areas are small and rare in alloy B, whereas large segregated areas are found in alloy A. Most of the segregation areas were observed at triple points and at the grain boundaries in both the alloys. Such areas in A and B are shown in Fig. 2(a) and (b). SEM studies of the fractured surfaces revealed the cleavage lines and flutes in alloy A (Fig. 4(a), (b)), while only the dimples were observed in alloy B (Fig. 5). Cleavage fracture is a low energy fracture that propagates along well-defined low-index crystallographic planes, while flutes are ruptured halves of tubular voids, which are formed by the planar intersecting slip mechanism.The absorbed energy of alloy A is lower than that of alloy B, which is confirmed by the presence of cleavage lines and flutes. In Fig. 3(a), cracks and cleavage lines were seen. It was also observed that the cracks were initiating from the segregated areas highly enriched in Ni, Fe, and Cr, this spot is label with “S” in the micrograph.



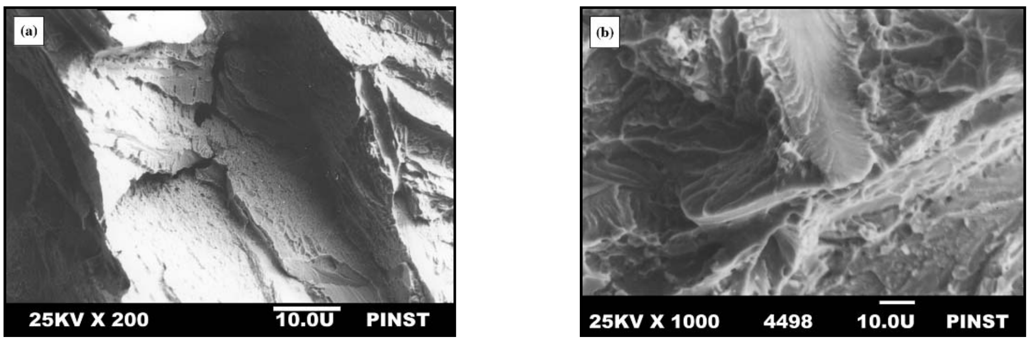
(a) (b)

Figure 2. (a) Basketweave type structure in alloy B; (b) parallel plate type structure in alloy A



(a) (b)

Figure 3: Segregation areas in (a) alloy A; (b) alloy B



(a) (b)

Figure 4 (a) Cleavage fracture along with cracks in alloy A; (b) formationof flutes in alloy A

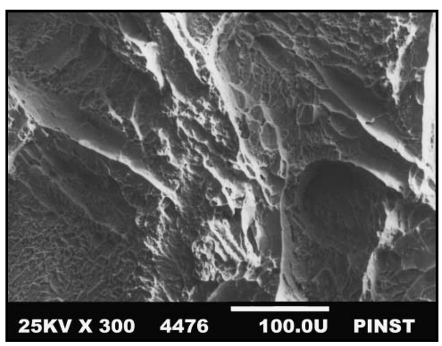
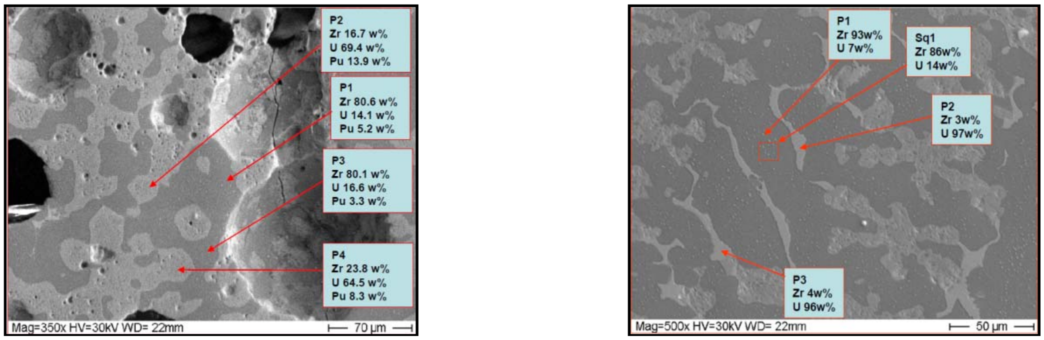


Figure. 5Ductile fracture in alloy B

D. Bottomleyet. al. have investigated high temperature irradiated fuel-liquefied Zircaloy interactions in support of severe accident safety studies. SEM and EDS has been used to find the cracked condition of the fuel and the fissiongas release during these interactions are major factors for fuel break-up, dispersion anddissolution in the melt under temperature transients [4]. In their study SEM micrographs of irradiated MOX in Figure 6(a) that the melt freezes into twophases with a dark, metallic phase and a light, ceramic phase (in contrast to optical micrographs). TheEDS analysis confirms that the ceramic phase is dominated by U and Pu with 70 - 90 wt% U+Pu. Bycontrast the dark (metallic) phase is dominated by zirconium and is ~80 - 90 wt% Zr. The brightlustrous appearance of the Zr-rich phase seen in the optical micrographs indicates it is metallic instructure and therefore it is likely to be α-Zr(O) containing U in solid solution. The ITU tests were intended to indicate the effects ofirradiation on the Zr-U-O system compared to natural materials and so to improve the modelling forsevere accidents.From the energy-dispersive X-ray analysis in ITU's tests it is seen that approximately 10 wt% ofthe 2 main elements - U(+Pu) and Zr - have diffused into the other phase during the interaction.Therefore, the ceramic phase is $(U, Zr)O_2$ mixed oxide with typically 10 wt% ZrO2 in the oxide. Theextent of the interaction can be gauged by the proportion of U in the Zr melt from the cladding. Theinteraction is dependent on the time and temperature, but it is seen to vary with the position analyzed.The interaction is seen in the natural UO2/Zircaloy cladding test in Figure 6 (b), the dark phase hastypically 85 - 90 wt% Zr while the U-based white ceramic phases have ~95 wt% U and although themelt composition is similar (~10 wt% exchange of U, Pu and Zr) the interaction distance is much morelimited. Thus in natural UO2 the effective solid-state inter-diffusion is much slower. Here the stabilityof the fuel is certainly a dominant factor and results in linear interaction zones.



(a) (b)

Figure 6.SEM-EDS of (a) irradiated MOX (b) natural UO2 sample(5 mins / 2000 °C / He).

CONCLUSION

Electron microscopy facilities are essential to assist research activities and services in the Malaysian Nuclear Agency. Microscopy facility costs are high and require a competent and experienced operators and maintenance engineer who can provide a high commitment to improve the experience any damage. This facility is the most common and important equipment in studying any most nuclear material degradation and due to irradiation. Study of microscopic cracks in nuclear material other objects formed by this irradiation degradation can be done for safety assessment of a nuclear plant.

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