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Shielded EPMA from CAMECA to JEOL Preliminary Performance Comparison

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1. Introduction

Electron Microscopy is widely used in post-irradiation examinations for material characterisations. The most common methods are Scanning Electron Microscopy (SEM) and Electron Probe Microanalysis (EPMA).

KAERI has operated for more than 20 years a shielded EPMA CAMECA SX-SX50R for the surface examination of irradiated specimens. This method allows the characterization of a sample surface morphology by scanning electron microscopy (SEM) and both the high-resolution elemental composition and its spatial distribution by wavelength dispersive X-ray spectroscopy.

This instrument has been replaced this year by a standard JEOL JXA-8230 EPMA, which has now been modified to enable the investigation of highly Radioactive samples including fuel rods segments. This induces a different arrangement of the electronics for remote control and the addition of a tungsten alloy shielding both at the specimen stage and between the spectrometers and specimen chamber.

The objective of the acquisition of this new instrument is to widen the scope of the investigations from polished specimens to fractography samples
and to enhance the SEM and X-ray resolution.

2. Performance

2.1 JXA-8230 Modifications

The JXA-8530 operates on a Microsoft windows for data acquisition and analysis while maintaining the powerful hardware and vacuum system to achieve an ultra micro area analysis. The installation of the new EPMA began in Jun 2012 in the same lead shielded environment as before in IMEF. It had second to be electrically /electronically customized for remote control and measurement and is making the tungsten alloy shielding of the instrument and the sample holder capable for manipulator handling by JEOL. The main shielding is positioned between the spectrometers and specimen chamber with a notch in the upper level for the crystal drive toward the poleshoe, as shown in Fig. 1. Two stacked W-plates encase the end piece of the poleshoe to shield the upper part of the chamber and partly the electron optics.

They have only a 140 mm Rowland circle so they are smaller than those of the CAMECA machine (180 mm). Therefore the crystals and counters are nearer to the specimen. This should be a benefit for the signal intensity, but can be impaired by the closeness of the active sample, which implies a higher radioactive background on the counter

2.2 SE and BSE image

The nominal specifications of the EPMA with respect to SE- and BSE imaging, as well as X-ray signal intensities and peak/background ratios were tested and obtained with inactive standard materials. The shielding concept was assessed by KAERI but no guarantee on the part of the manufacturers could be given with respect to the quality of examination for highly active materials.

The quality of the electron microscopy imaging of a high g-active specimen was very good, as shown in Fig. 4. The image quality of the open specimens has to be checked.

The important features for a microanalysis are the spectral resolution, signal intensities and peak/background ratio. The first one has not yet been checked in detail and is anticipated to be marginally worse than on the old machine owing to the small size of the spectrometers. Some P/BG ratios have been measured and found to be very high for standard materials. The chemical composition of the surveillance specimen from Ulchin Unit No.5 RPV is reported in table 1.

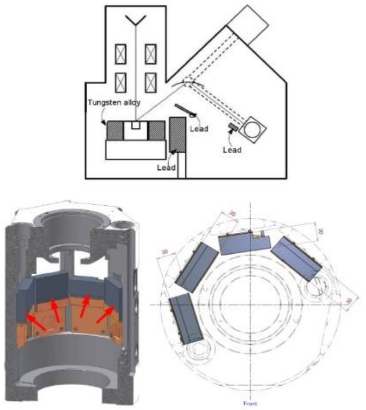

Fig.1. Schematic of the lead-shielding.

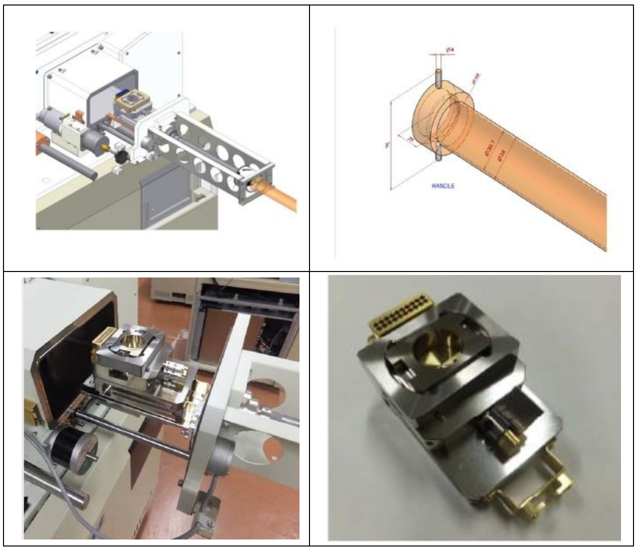

Fig.2. Specimen Exchange & Mount/Unmount Detachable Holder by Tung

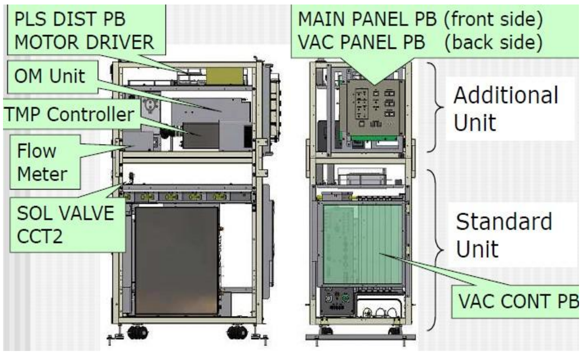

Fig.3. Remount connection box to avoid radiation damage.

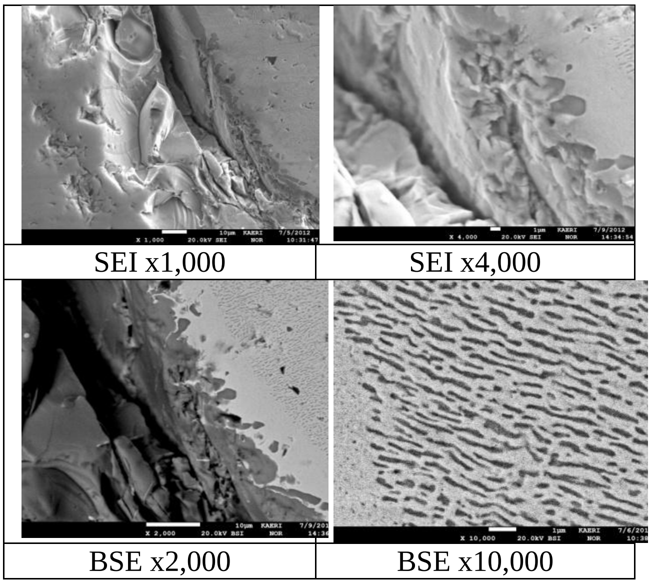
SEI x1,000
SEI x4,000
BSE x2,000
BSE x10,000

Fig.4. SEI, BSE image of an irradiated SFR specimen.

Table1. Chemical composition of surveillance specimen from Ulchin Unit No.5 RPV.

System	Chemical		CAMECA		JXA-8230	
Mater	Basic	Weld	Basic	Weld	Basic	Weld
Ni	0.9	0.1	0.855	0.098	0.831	0.08
Cu	0.03	0.02	0.037	0.018	0.026	0.024
Cr	0.19	0.04	0.295	0.059	0.195	0.047
Mo	0.46	0.5	0.437	0.467	0.478	0.493

3. Conclusion

A new shielded EPMA demonstrates a high versatility with respect to the examination capabilities and specimens that can be investigated.

It has advanced SEM and X-ray imaging with a high lateral resolution. The first test of the machine with active specimens shows the following:

- The imaging performance for low b,g-active samples is very good for highlyy active
- The spectrometer efficiencies of the CAMECA SXR and JEOL 8230 are comparable with the advantages for the surveillance specimen. Also the spectral resolutions are at first sight comparable despite the small JEOL spectrometers.
- The sample loading for embedded flat samples is convenient. The use and loading of a rotation and tilt sample holder must yet be adjusted.
- Further adjustments on the light microscope, automatic aperture and infrastructure.