Development of Laser- Raman Microscopy and Microhardness Testing capability for Post Irradiation Examination as part of UK wide capability

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1. Introduction

Windscale Laboratory, based at the Sellafield site in the UK, is a heavily shielded facility which handles highly active (HA) materials. Constructed in the 1960's for post irradiation examination (PIE) of uranium metal (Magnox) fuel, and subsequently extended to provide capability for water reactor and gas reactor fuel, the facility consists of thirteen heavily shielded cells. Each cell has 5 separate workstations and is connected to the rest of the facility via a fuel transport corridor, which allows delivery of fuel or other HA materials via the transfer system. A number of extensions and refurbishments were carried out in the late 1980's.

As part of UK government funded research (UKRI), a new capability has been formed across a number of laboratories and hot cell operators within the UK. The Henry Royce Institute (HRI) is open to researchers, academics and industry alike, and provides a unique capability comprised of facilities, equipment and researchers.

As part of the HRI, the UK National Nuclear Laboratory (NNL) has purchased a hot cell triple laser raman and microhardness microscope. This microscope will be used for analysis of irradiated fuels, cladding (UK and PWR systems) and graphitic materials. The following paper gives an overview of the micrscope, the design process and the proposed areas of interest for research.

2. Modified microscope

The Olympus optical microscope, Figure 1, has been modified for remote handling and characterisation of highly active samples (e.g. irradiated fuel and cladding). The microscope has a laser confocal analysis system incorporating 3 lasers. Raman spectroscopy can be performed on highly active materials and features of interest via the use of the optical microscope. In addition to this, microhardness testing (Vickers) can also be performed at chosen sites, allowing a more complete characterisation of features. Microhardness testing (Vickers type indenter) can be carried out with a load range of 0.1N to 4N.

The microscope operates in one of three modes; 100% Microscope (for imaging / microhardness testing); 100% Raman (for spectroscopy) or 20% microscope / 80% Raman (for laser spot positioning on the sample). The laser Raman spectroscopy system is supplied by Horiba and operates in confocal mode. Three lasers are remotely operated with signal transmission via optical fibres:

- 532 nm /100mW air cooled laser (spatial resolution 4μm)
- 660 nm /100mW air cooled laser (spatial resolution 4μm)
- 785 nm /90mW air cooled laser (spatial resolution 8μm)

The multichannel cooled CCD detector,1024x256 pixels has a spectral range 220 – 1050 nm, pixel size : 26 x 26µm, and in conjunction with the LabSpec 6 software, has an autofocus capability using spectrometer signal analysis, and a >4-8 micron spatial resolution. Specially designed sample holders (Figure 2) ensure that samples are a consistent distance from the Raman confocal head, even if there is a variation in sample geometry. This allows traditional metallography to be undertaken on samples at the same time as Raman spectra are acquired, and this can be done at a number of positions along the length of the sample.

3. Future Spent Fuel and Cladding Examination

The current UK spent fuel storage strategy requires an extended period of interim wet storage. In order to assist in experimental modelling for storage and disposal scenarios, long stored and water exposed PIE offcuts have been recovered from historic storage containers for further examination. The oxide fuel will be characterized to assess the likelihood of oxidation. Elemental characterisation of the bond between the stainless; steel cladding and fuel will also assist with modelling storage scenarios.

In addition to advanced gas cooled reactor characterization, oxidation depths and phenomena in water reactor (zircaloy) type cladding will also be explored.

Figure 1. Triple Laser Raman Microscope (left) and control system (right).

Figure 2. Sample holder (left) and hardness reference block (right).