Materials Science and Engineering Instruments

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This document considers instrumentation requirements for Engineering Diffractometers and for a Radiography/ Tomography station at the ESS. Substantial gains are possible in performance for an Engineering Diffractometer for Strain Measurement at the ESS, compared with either existing reactor or pulsed sources. In the case of an optimally designed engineering strain scanner the overriding requirement of the instrument is the accurate measurement of a lattice parameter, d_{hkl}, at a known location within the material under study. The instrument is thus essentially a powder diffractometer, modified to meet these specific requirements. Secondary issues include the requirement for considerable space and flexible setup around the instrument to allow for large samples and complicated sample environments.

Gain over existing instrumentation is of the order of 30-60 over instrumentation presently under construction at ISIS and ILL, and from 50-600 over presently operational instrumentation, depending on type of experiment.

The Engineering diffractometer would require a high resolution moderator (decoupled / poisoned). It is felt that neither the proposed H_2 or H_2O moderator options so far presented fully met the needs of a strain measurement diffractometer, and that the possibility of obtaining higher resolutions using methane or hybrid moderators should be investigated. Further gains in performance could then be achieved.

Such an instrument would ideally be situated on the 50Hz short pulse (SP) target. It would lose considerable flexibility and some performance in operating on the 10Hz SP target described. An instrument could be built on the long pulse target, running in a 'reactor mode' with a monochromator, but there would be a reduction in range and performance.

Gains for an ESS traditional radiography instrument compared with siting a traditional radiography instrument at ILL are negligible. However Bragg edge radiography would allow totally new types of science which are unfeasible on a reactor source.

Introduction - Engineering Diffractometer:

Not long after the publication of the Bragg equation in 1912 the potential of diffraction based techniques for the measurement of lattice strain (ε_{hkl}) was appreciated [1]. The measurement of stress with neutrons has grown in importance over the last decade, with the European academic community taking a lead in this development. This has led to the construction of dedicated stress measurement diffractometers at many neutron facilities. Industrial interest and usage has grown similarly as it has become clear that the technique has matured. This is evidenced by an ISO international standard, developed under the Versailles Project on Advanced Materials and Standards (VAMAS) Technical Working Area 20, for stress measurement using neutrons [2] which is presently in draft form, with initial issue expected in 2001/2.

Strong European academic community

Growing industrial usage

Draft ISO / CEN standards submitted.

Two broad classes of experiment make use of these techniques, on a wide range of structural materials. Firstly, measurement of changes in lattice separation as a function of position within an actual component provide maps of the stresses remaining after production, joining or use. Such residual stresses affect fatigue resistance, fracture toughness and strength of materials, and hence influence safety, component lifetime, costs and speed of the design cycle. The second class of experiments studies the effect of stress, temperature and other environmental variables on the deformation of materials, thus providing a fundamental understanding of the mechanics of materials. Both types of experiments provide information for process modelling, and materials development.

Irrespective of the source or method, accurate strain or stress measurement thus relies upon the accurate location of diffraction peaks, in order to determine the lattice parameter. Secondly, in many experiments, this must be carried out at a precisely known position within the sample. Finally, many engineering materials must operate at considerable stresses or temperatures, and the modern engineering diffractometer must have the capability of applying realistic environmental variables to samples *in situ.* This is likely to become more important in the future as the drive to optimise processing parameters requires the investigation of material production routes (e.g. rolling) *in situ.*

Instrument Optimisation - Engineering Diffractometer:

In the case of an optimally designed engineering strain scanner the overriding requirement of the instrument is the accurate measurement of a lattice parameter, d_{hkl}, at a known location within the material under study. To enable different instruments to be compared it is reasonable to define a FOM such that an increase of a factor of two in the source illuminating an instrument results in a factor of two increase in the FOM. It is also necessary to take into account the uncertainty of the result obtained. Hence the most useful high-level definition of a FOM for a strain measuring instrument will be *'the inverse of the time taken to measure a d-spacing to a given uncertainty'*.

Definition of a Figure of Merit for determining peak position is the first requirement for instrument design.

d-spacings are obtained from the observed diffraction patterns by a 'least-squares' fitting procedure, and it has been shown by Sivia [3] that in the situation of an isolated Gaussian peak the time (t) taken to measure (with an uncertainty of σ) the position of a peak is:

$t \propto w^2 / I\sigma^2$ (1)

where w is the width of the peak, and I the (integrated) intensity within the peak recorded in unit time. Hence the FOM required for an instrument concerned solely with measuring the peak position may be written :

$FOM = I\sigma^2/w^2$ (2)

if the peaks were Gaussian in shape and well separated. The correctness of equation 2 when an *arbitrary* not necessarily symmetric peak shape is fitted by the least squares method is derived in [4]. The veracity of this result has also been demonstrated empirically, using experimental data from a number of sources and on a number of different materials [5].

It is further possible to include the effect of background in the optimisation [6]:

Background can be included in the Figure of Merit.

$FOM \approx I\sigma^2/(w^2(1+2v2B/P))$ (3)

where P is the peak height, and B the background signal.
A number of assumptions can be made to make full use of this expression in instrument design. These include matching of instrument resolution terms, an expression for the number of diffracting peaks in a given wavelength window (which is material dependent), intensity losses with distance, and a simple form for the incident intensity spectrum. Carrying out this calculation [4] produces the FOM shown in Figure 1 as a function of primary flight path. This clearly demonstrates the requirement for a 40-60m flight path instrument, somewhat of a departure from the philosophy of existing engineering instruments. However, it should be noted that the calculation above does not include contributions from sample resolution. When this constraint and a 50Hz running option is

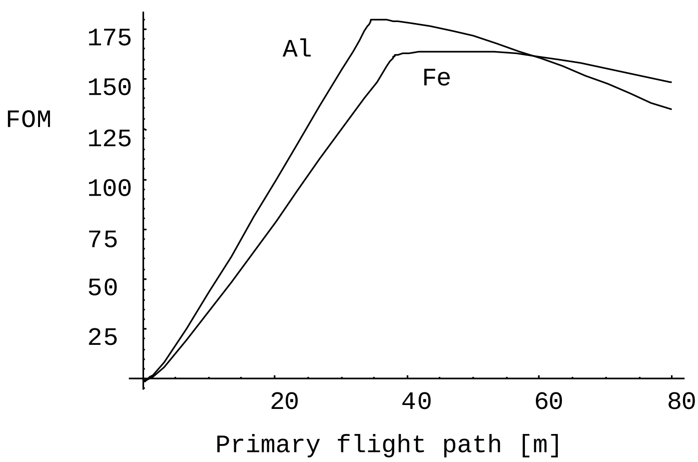

Fig.1: Figure of Merit as a function of primary flight path for 25Hz source [4], ignoring sample broadening contribution, for aluminium and iron.

included in [4], the optimum instrument instead has a primary flightpath ~50m, with the added requirement of tuneable resolution, i.e. the option to gain flux by coarsening the horizontal resolution. A number of scenarios can be imagined for achieving this flexibility, for instance by allowing the final sections of flight path to switch between guide and absorber [7].

The explosion of interest and demand for neutron strain measurement diffractometers has led to the construction of several new dedicated and optimised instruments at this time (early 2001). While specific design details follow from Monte Carlos modelling of the complete instrument with its particular source, all the 2nd generation strain diffractometers presently under construction or in the design phase make use of these ideas to a greater or lesser degree (at LANSCE, ISIS, ILL and SNS). The ESS instrument will be highly competitive with these instruments, not only because of the massively improved source, but also because of the further lessons which will be learnt over the next two to three years as these new instruments are commissioned.

Instrument Design - Engineering Diffractometer:

Based on the arguments above, the optimum instrument requires

- a variable resolution, from medium to high,
- a moderately large detector array centred on $2\theta = 90°$ with resolution matched to the best case intrinsic resolution
- backscattering and transmission detectors
- a variable gauge volume and
- at least 1-2m^3 space for large components and sample environments.

These requirements can only be properly achieved with a dedicated instrument, i.e. a shared powder diffractometer beamline will require unacceptable compromises for both engineering and powder diffraction communities. The optimised instrument would have a 50m flight path, with frame definition choppers at 6 and 9m, and curved with a 5km radius from 10m to 37m. Guide (m=3+) extends from within the primary shutter (~4m) to 48.5m (i.e. within 1.5m of the sample position). The top and bottom of the guide will always be in place [8], but the sides of the guide will be switchable for absorbing material from 38m to 48.5m. This allows horizontal divergence (and hence instrument resolution) to be improved, at the expense of neutron flux, 'tuning' the instrument resolution to match the sample resolution in order to maximise performance, as judged by the FOM (Eq. 3). Achievable resolution in the 90° detector banks would be $\Delta d/d \sim 2x10^{-3}$ to $7x10^{-3}$. The wavelength window would be ~1.5Å at 50Hz, corresponding to a lattice parameter window of ~1Å in the 90° detectors.

Based on the criteria of Eq. 3, the instrument will perform ~30 times better than best in class instruments presently under construction, and 50-300 times compared to existing instruments. By including backscattering and transmission detectors, and extending the main detector banks, extra strain components will be obtained for some types of experiments allowing rapid mapping of strain tensors. While only providing improvements in some types of

Main components of the instrument

- ***Large 90°detector array***
- ***Tuneable resolution***
- ***Variable gauge volume***
- ***Backscattering and transmission detectors***
- ***Large, flexible sample space***

Parameters of the instrument

- ***50m curved flight path***
- ***Frame definition choppers***
- ***Swappable m=3 guide / absorber for end section***

Performance of the instrument

- ***Resolution Dd/d ~ 2x10^{-3}***
- ***d-spacing window ~1Å at 90° and 50Hz***

- ***FOM x30 compared to best in class under construction***
- ***FOM x 90 for some experiments***

experiment, this would improve performance by a further factor of 2 to 3.
On the 50Hz source, the flexibility exists to run the instrument at 50, 25 and 16.6Hz – all options which would be used depending on the material and type of experiment. If moved to the 10Hz source, this flexibility would be lost, without any concurrent gain in flux (such as would be achievable on the planned ISIS 10Hz target). While the instrument (moved to a ~80m primary flight path) could be built on the 10Hz target, it would suffer around a 1/3 performance for the majority of experiments compared with siting on the 50Hz target. The long pulse (LP) target could be used, running with a monochromator, but would provide poor performance for this instrument.

Preferred target is 50Hz short pulse.

10Hz target is possible, with loss of performance. Long pulse target not suitable.

The instrument requires a high resolution moderator, hence a thermal, decoupled / poisoned moderator is appropriate. It is felt that neither the H_2 or H_2O moderator options so far proposed fully meet the needs of a strain measurement diffractometer; the best case moderator provides a pulse nearly twice as wide as achievable with the ISIS CH_4 moderator. Hence the possibility of obtaining higher resolutions using methane or hybrid moderators should be investigated for ESS. Further gains in performance could be achieved under this scenario.

Requires sharp thermal moderator.

Options such as methane or hybrid moderators should be investigated.

Technical feasibility - Engineering Diffractometer:

The instrument is highly feasible. The novel feature is the requirement for a tuneable resolution diffractometer, however the engineering requirements for accurate switching of guide/absorber sections is certainly achievable.

Instrument is feasible with present technology.

Further gains can be envisaged from future improvement in performance of high *m* guides, improvements in detector efficiency and use of in-shutter guides. It is likely that experience gained in the commissioning of the 2nd generation instruments presently under construction will influence the detailed design of this instrument, though not the main features of the design.
Finally, considerable opportunities and improvement in throughput and achievable science, will be available with improvements in experimental setup (use of coordinate measurement machines), advanced sample environments and development of integrated software.

Radiography / Tomography station:

The primary requirements for traditional radiography are for high thermal or cold neutron flux, with a reasonably parallel beam. However, there is the considerable potential advantage while doing radiography on the 50Hz SP target of carrying out Bragg edge discrimination during radiography measurements, allowing simultaneous identification of the material present.
Gains for an ESS traditional radiography instrument compared with siting a traditional radiography instrument at ILL are negligible. However Bragg edge radiography would allow totally new types of science which are unfeasible on a reactor source. Gains compared to an ISIS Bragg edge radiography instrument would be 30.

New type of science possible at ESS source.

50Hz SP or LP target and high flux moderator useable for traditional radiography

50Hz SP target and moderate resolution moderator required for Bragg edge radiography

For traditional radiography, either 50Hz SP or LP targets would be appropriate, using an intensity optimized moderator (cold or thermal). For Bragg edge radiography, the 50Hz SP target is required, using a moderate resolution moderator.

Introduction - Radiography / Tomography station:

Neutrons have been used as a tool for non-destructive evaluation of materials and components for many years. The quality of the images obtained and the sample properties which can be measured depend strongly on the quality of the neutron source and the detector performance and efficiency. One of the main applications of neutron radiographs is the study of materials distribution in macroscopic samples. Due to the neutron cross section of hydrogen it can be measured very precisely, to some mg per g. Thus the study of time and space dependent moisture distribution has been studied in civil engineering, biology and hydraulic engineering. Further, hydrogen in materials is of much more widespread interest, for instance in the investigation of adhesive joints or the precipitation of hydrides.

The high depth penetration of neutrons even in heavy materials makes it highly relevant to industrial applications, in particular the monitoring of internal defects in components. It is these two areas, monitoring of hydrogen in materials, and viewing of defects in components that neutron radiography has been most utilised.

An added benefit is provided when carrying out radiography at a pulsed neutron source. In crystalline solids the coherent neutron scattering cross section varies abruptly at the 'Bragg edge' (Fig. 2). Since the spacings of the Bragg edges are characteristic of a diffraction pattern of the material, one can imagine making measurements at energies above and below major Bragg edges, providing radiographs in which material show up with different scattering intensities, thus helping to selectively identify the

Use of information from Bragg edges, combined with traditional radiography, would provide a unique facility, exploring new areas of science.

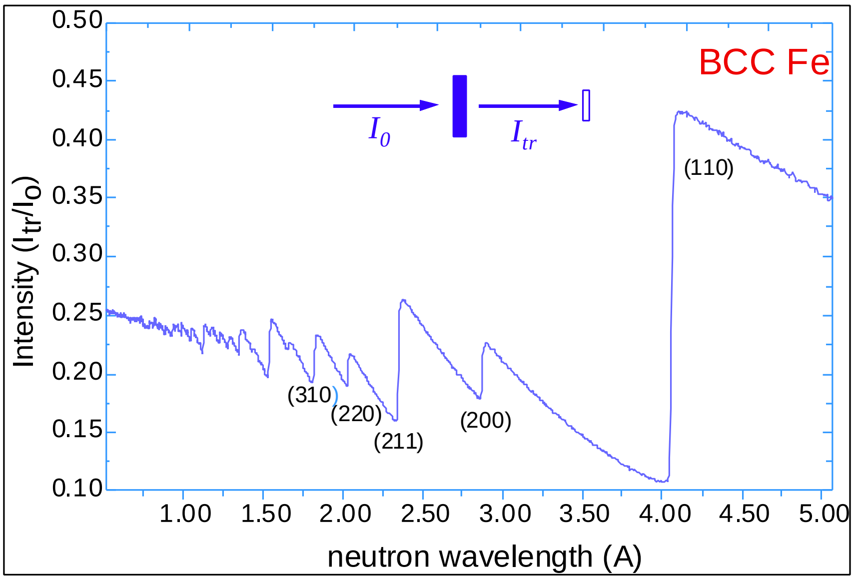

Fig. 2: Spectrum of transmitted intensity through bcc iron, showing clear Bragg edge spectrum.

distribution of particular phases. Further, if a true Bragg edge diffraction pattern could be collected with both the fine spatial and time resolution required, true 3D maps could be produced of both phase and stress.

Instrument Design - Radiography / Tomography station:

A detailed design study of this instrument has not been carried out, however the following aspects are noted:

- Depending on the type of experiment, the spatial resolution (pixel size) required for radiography varies between 0.02mm and 0.2mm, with typically requested sizes of 0.05mm to 0.1mm. Active areas of 20mm x20mm to 250mm x250mm are used, again dependent on the type of science.
- The instrument characteristics, most importantly beam divergence, must match these requirements. It is possible to achieve the required divergence either by extending the flight path, or through the use of soller type collimation.
- Many detector systems are sensitive to fast neutrons and gamma rays. The use of a t0 chopper or curved guides followed by soller collimation needs to be considered.
- The instrument requires space for manipulation and rotation of samples, variable sample-detector distance and variable incident beam size/divergence.
- Traditional radiography requires only high total flux (in either the thermal or cold neutron range, dependent on type of science). Hence the 50Hz SP or LP targets are both acceptable, using high flux moderators. The gain in performance compared with constructing such an instrument at the ILL however, is small.
- Bragg edge radiography has been proved in concept, but to become truly feasible and useful it will require detector developments.

Traditional radiography
- ***15 – 50m flight path***
- ***50Hz SP or LP targets***
- ***High flux thermal or cold moderators***

Phase specific radiography / tomography would provide a unique facility at the ESS. This technique is not presently possible, and to be truly useful requires the high pulsed fluxes which will be achieved at the ESS. At least moderate resolution in the pulse shape would be required; the decoupled hydrogen moderator is a good choice. The instrument would require a short flight path combined with soller collimators, to allow both a large wavelength window and the highly parallel beam required for high spatial resolution.

Bragg edge radiography
- ***New science***
- ***15m flight path***
- ***Soller collimation***
- ***50Hz SP target***
- ***Moderate resolution thermal moderator***

Technical feasibility- Radiography / Tomography:

The instrument is highly feasible in its simplest form. To take advantage of the pulsed nature of the source, detector developments are required.
In the past few years a number of detector developments have been made, providing high sensitivity detectors with good dynamic range and linearity, in the fields of imaging plates and scintillator screen-CCD cameras. These detectors are limited in their readout

Traditional radiography / tomography is technically feasible now.
Bragg edge radiography /tomography requires detector development, thought to be achievable in 5 year time frame.

speed and are sensitive to gamma radiation. However, it is newly emerging technologies which hold great promise; amorphous silicon arrays and micro-strip gas counters.

With rapid readouts and fast data acquisition, the step from neutron radiography to neutron tomography becomes feasible, allowing a 3D reconstruction of the object, providing far greater spatial sensitivity. The software and techniques for combining multiple radiographs to produce tomographs are well established from work in the x-ray community.

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