Strain scanning (in-situ tensile test)

The aim of strain measurement is to characterise the response to applied loads, particularly those corresponding to in-service loading, and to observe the response of objects so as to predict the integrity of various designs. The particular significance of neutron and X-ray diffraction methods is that they offer a direct method of measuring the elastic component of strain deep within crystalline materials through the precise characterisation of the interplanar crystal lattice spacing. Diffraction uses the atomic lattice itself as a deformation gauge. The principle of diffraction strain measurement in polycrystalline alloys relies on Bragg's law that establishes the relationship between the average interplanar lattice spacing *d* within the measurement gauge volume.

The volume of material contributing to the diffraction pattern corresponds to the intersection of the incident and diffracted beams, typically defined by slits and collimators, respectively (Fig. 1). The centroid of this gauge volume (typically of the order of cubic millimetres) defines the location of the measurement. In a neutron strain scanning instrument (e.g. ENGIN-X), the gauge volume is fixed at a position in the laboratory, so the strain variation across the sample is explored by moving the sample using a translation stage. The measured strain gives the component of the strain tensor along the direction of the neutron scattering vector, which bisects the incident and diffracted beams.



Fig. 1 Schematic diagram of a time-of-flight neutron strain scanner with in-situ loading setup

ENGIN-X commands

This lists Open Genie and EX-OG commands commonly used at ENGIN-X, but is not an exhaustive list.

Open Genie instrument control commands:

begin - start recording diffraction pattern

end - finish recording diffraction pattern

updatestore - save a temperature file while still recording data

scan/y centre=1.0 range=20.0 nstep=21 ctime=0.1 - perform wall scan along y axis.

file_scan - run script file for strain scanning

stressrig/file - run script for in-situ loading test

loadscript "c:\\scripts\\xxx.isc" – load script from file c:\\scripts\\xxx.isc and then run script with file name, e.g. xxx.isc

EX-OG / Open Genie data analysis commands:

xfocus – focus raw data and create .his file. For example: w=xfocus(123456,1) focus run number "123456" from bank "1" data. And then assign the data to variable "w".

rfocus – read .his file or focus raw data if .his file not exit. For example: w=rfocus(123456,2) check whether 123456_1.his file exits, if not create 123456_1.his; otherwise read 123456_1.his. And then assign the data to variable "w". Both xfocus and rfocus will give you the same data at the end. The difference is that if the .his file already exists, using rfocus to read data will be quicker.

set/ext – set file extension. Using "updatestore" will save temperature data file. The first saved file will have extension of "s01" and the second saved file will have extension of "s02", and so on. In order to view the temperature file, you have to use this command to change the file extension. For example, set/ext "s01"

display - display .his file. For example: w=rfocus(123456,2); display w

unit/d - change display unit to d-spacing. For example: w=rfocus(123456,1); u/d w; display w

analyze_scan - whole pattern refinement using Pawley refinement

analyze_scan/peaks - single peak fitting

batch_focus - create .his files

batch_gsas_file - create GSAS files

Practical Exercise

- To quantify the evolution of microstrain as a function of mechanical deformation, one approach is to measure the lattice strain response by diffraction as the sample is subjected to increasing uniaxial load. Setup a tensile test experiment and record diffraction pattern during loading. Please read Appendix A for setup tips.
- 2. Display the data and change the unit to d-spacing.
- 3. Analyse the first diffraction data and index the pattern.

When a polycrystalline aggregate is deformed elastically, the inter-planar spacing within its constituent grains changes. Within a set of planes that have similar orientation with respect to the stress direction, the inter-planar spacing is essentially similar between one grain and another. This grain-set-specific strain causes observable shifts of diffraction peaks. As only those grains that possess orientations that fulfill the criteria of Bragg reflection will contribute to the measured reflection, the strain obtained will be representative of the average strain from grains within the irradiated volume.

The Single peak fitting method normally fits to the experimental data using the profiles such as Gaussian, Pseudo-Voigt peak type, etc. Each single peak is characterized by position, amplitude and peak width; there's no need to input the crystal structure information into the program. Since Single peak fitting analyse individual *hkl* reflection, the elastic lattice strain for each lattice plane is determined. The lattice strain can be found from the deformed lattice spacing *d* as

$$\varepsilon = \frac{(d - d_0)}{d_0} = \frac{\Delta d}{d_0}$$
 (Eq. 1)

where d₀ denotes unstrained lattice spacing.

In addition to fitting the single peak, it is possible to perform a whole pattern refinement on the data. Pawley refinement is a similar approach to Rietveld refinement that accommodates the variation in peak intensities by allowing the intensity of individual reflections to vary freely, while the peak positions are determined in the usual manner from the unit cell dimensions. This approach provides an empirical average of the different reflections and potentially includes physics that describes the overall deformation of the polycrystal. Lattice parameter *a*, *b* and *c* are determined, hence lattice strain is normally calculated from lattice parameters. The lattice *a* strain is therefore readily found as

$$\varepsilon = \frac{a - a_0}{a_0}$$
 (Eq. 2)

where a_0 is the reference value from unstrained lattice.

Practical Exercise

- Use Open Genie Single peak fitting routine to analyse individual *hkl* reflections in all the collected data. Use Eq. 1 to calculate strain for each lattice plane. Plot the Stress vs. Lattice strain to show the ansisotropic response of lattice planes during the uniaxial tensile test.
- 2. In solid mechanics, Young's modulus (*E*) is a measure of the stiffness of an isotropic elastic material. Young's modulus, *E*, can be calculated by dividing the stress σ by the strain ϵ (Eq. 3). Use Eq. 3 to calculate the embedded stiffness for the individual lattice plane.

$$E = \frac{\sigma}{\varepsilon}$$
 (Eq. 3)

3. Use Open Genie whole pattern fitting routine to analyse all the collected data. Use Eq 2 to calculate lattice *a* strain. Plot Lattice a strain against the applied stress.