

Time-Resolved Synchrotron X-ray Techniques for Experimental Mechanics

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Abstract. The availability of high brightness synchrotron light sources, and advances in detector technology, mean that time-resolved X-ray experiments to measure strain in materials and components are routinely possible. Beamline I12, the Joint Engineering, Environmental & Processing (JEEP) beamline [1] at Diamond Light Source (DLS) is a facility offering diffraction and imaging techniques to a wide user community, including researchers interested in the evolution of strain with time. Here, we introduce the techniques available, referencing examples of how they have been applied in the field of experimental mechanics.

Introduction

Strain measurement using X-rays, while primarily perceived as a diffraction technique, can use real space, imaging methods in 2-dimensions (radiography), 3-dimensions (tomography) and 4 dimensions (3D plus time). At synchrotrons, it is possible to combine diffraction and imaging techniques in the same experiment. Experiments can be performed in-situ, subjecting samples to changing load, temperature or other environmental conditions while collecting data for strain measurement. Beamline I12 and other facilities have sample environments available for in-situ work, or users can bring their own test equipment and rigs. The X-ray source brightness and detector speed mean that time-resolved experiments are possible. Like any technique, there are limitations and experiments must be designed to account for them. However, synchrotron X-ray techniques are being employed by researchers for innovative strain measurement experiments.

X-ray Diffraction

Diffraction of X-rays by crystalline materials, which obeys the well-known Bragg's Law, enables measurement of the atomic interplanar spacing d , which changes when a material is strained. If a strain-free interplanar spacing d_0 , is known, then the elastic crystal lattice strain can be determined. Calculation of stress requires knowledge of the diffraction elastic constants for the material being studied.

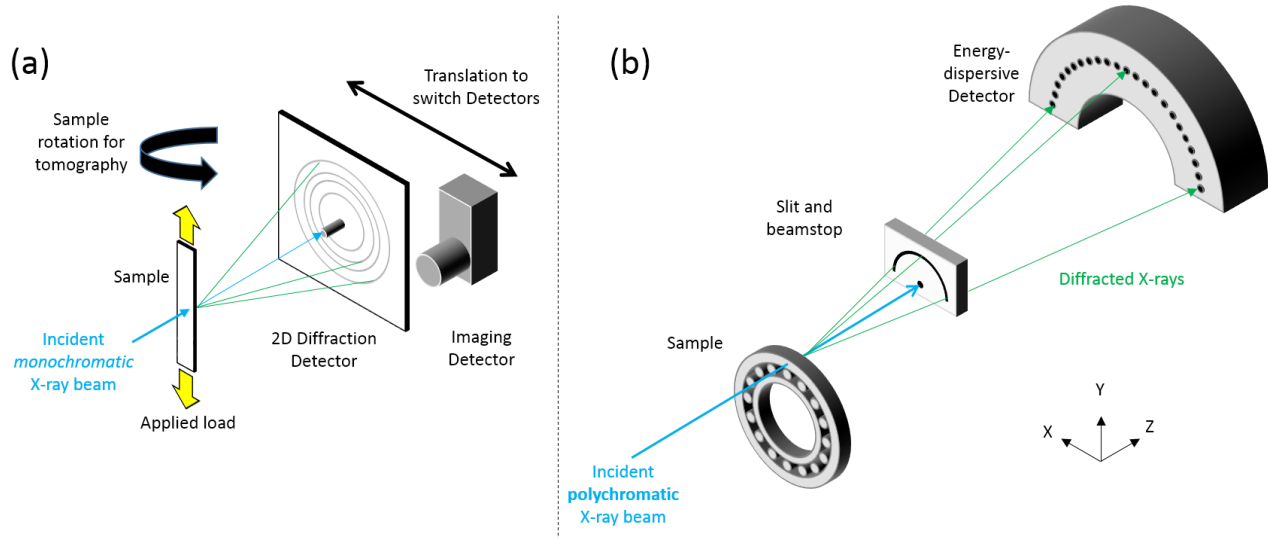


Fig. 1. (a) Typical 2D monochromatic diffraction & imaging setup. (b) Energy-dispersive diffraction setup

Powder Diffraction on Beamline I12, uses a 2D, flat panel detector to collect diffraction patterns over a range of scattering angles θ , Fig.1(a). The technique uses a single wavelength λ of X-rays from a monochromator, and works in transmission *through* the sample. Characteristic peaks in the diffraction pattern arise when the Bragg condition for momentum transfer q is fulfilled, Equation (1). Momentum transfer q is simply calculated as 2π divided by d . Expressing Bragg's Law in terms of q is useful because it is generic and independent of the type of radiation used, thus allowing comparison of results obtained from different methods.

$$q = \frac{4\pi \sin(\theta)}{\lambda} \quad (1)$$

Time resolution of less than a second is possible, so that strain can be measured in real time during mechanical tests or materials processing. An example in-situ experiment was the study of microstrain evolution during the biaxial deformation of steel sheet samples [2].

Energy-Dispersive X-ray Diffraction (EDXD), Fig. 1(b) is a technique in which conditions fulfilling Bragg's Law are determined by measuring the momentum transfer q between photons of different wavelengths λ , at a fixed diffraction angle θ . A spectroscopic detector is used, which counts photons as a function of the photon Energy E . If h is Planck's constant and c is the speed of light, Equation (1) becomes:

$$q = \frac{4\pi E \sin(\theta)}{hc} \quad (2)$$

Collimation of the incident and diffracted X-rays with slits defines a three-dimensional gauge volume, which is used to probe inside thick or complex samples. Strain mapping is done by scanning the sample through the gauge volume. The EDXD technique is commonly used for in-situ loading experiments, for example mapping of the changing strain field around propagating cracks [3, 4].

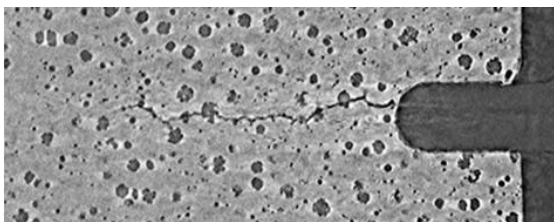
Stroboscopic Diffraction is a development of the EDXD technique, in which data acquisition is gated by an external signal generated by a sensor on a rotating or reciprocating piece of machinery. The gate signal ensures that the detector only counts X-rays when a signal is received from external hardware. This is used to perform measurements on repeated cyclic processes, such as a cyclic load applied to an engineering component. The X-ray counts for one cycle may not be sufficient for data analysis, but by summing up counts collected at the same point in the cycle over many cycles, an analysable dataset for that point in the cycle is collected. At Beamline I12, the stroboscopic technique has been used to measure elastic strains in a running engine [5] and the outer race of a rotating bearing [6].

X-ray Imaging

Imaging is attractive to researchers because of the intuitive ability humans have to detect features and patterns in images. Quantitative measurement of strain from images has been greatly improved by Digital Image Correlation (DIC) techniques. It is feasible to alternate X-ray imaging with diffraction by switching detectors, see Fig.1(a). The new K11 DIAD beamline at DLS will be able to perform diffraction and imaging concurrently, improving time resolution.

Radiography is the original X-ray technique. It is frequently used to support X-ray diffraction measurements, enabling quick sample alignment and identification of features such as a pre-existing crack or notch. Two dimensional radiographs can be analysed using DIC to obtain displacement and strain information.

X-ray Computed Tomography (XRCT) is probably most familiar to us from medical imaging. The principle



500 μm

Fig. 2. Tomography slice showing crack growing from notch in cast iron.

is to take a large number of radiographs of an object from different angles, which are then processed with a computer algorithm to generate a 3-D virtual reconstruction of the object. The virtual object can be sliced and rendered with visualisation software. With synchrotron X-rays, scan times are short enough for many scans to be acquired, for example during a loading or crack growth experiment. If the object contains trackable features, then Digital Volume Correlation (DVC) techniques can be used to measure displacement and strain between successive scans. Fig. 2 is a vertical slice through notched cast iron sample containing a fatigue crack. The graphite nodules in the cast iron provide suitable features for DVC. A good example of

work combining X-ray diffraction, tomography and DVC is a study of by Bharli et al. of crack strain fields in polygranular graphite [7].

Conclusion

Synchrotron X-ray facilities offer researchers a variety of techniques for experimental mechanics. Time-resolved studies are possible, and combining diffraction and imaging is becoming common. New facilities will further enhance the science infrastructure available to researchers.

References

- [1] M. Drakopoulos, T. Connolley, C. Reinhard et al.: J. Synchrotron. Rad. Vol. 22 (2015), p. 828-838.
- [2] D.M.Collins, M.Mostafavi, R.I.Todd, et al.: Acta Materialia Vol. 90 (2015), p. 46-58
- [3] H. E. Coules , G. C. M. Horne , M. J. Peel and T. Connolley: Int. J. Mech. Sci. Vol. 150 (2019), p. 103-111
- [4] C. A. Simpson , S. Kozuki , P. Lopez-Crespo et. al.: J. Mech. Phys. Solids Vol. 124 (2019), p. 392-410
- [5] N. Baimpas , M. Drakopoulos , T. Connolley et al.: J. Synchrotron Rad. Vol. 20 (2013), p. 316-323
- [6] M. Mostafavi , D. M. Collins , M. J. Peel et al.: Strain Vol. 53 (2017), e12221
- [7] S. M. Barhli, L. Saucedo-Mora, M. S. L. Jordan et al.: Carbon, Vol. 124 (2017), p. 357-371