Engineering at Large Scale:
Experience from Diamond Light Source

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Abstract— **Diamond Light Source is the new UK synchrotron facility located at the Harwell Science and Innovation campus near Oxford. It represents the biggest UK investment in science in 30 years, and is set to become the flagship laboratory for multi-disciplinary research. Engineering solutions of remarkable scale and complexity are required for the operation of this facility. On the other hand, engineering science is rapidly becoming an important theme in Diamond's research portfolio. This lecture will cover some aspects of engineering at the Diamond Light Source.**

Index Terms—**Engineering science; synchrotron light; X-ray diffraction; X-ray imaging.**

I. INTRODUCTION

Diamond Light Source is the biggest UK investment in a science facility over the last thirty years. This highlights the importance of this facility as a national centre for cutting edge research into all aspects of natural science, from physics, chemistry and engineering to biology, pharmacology and medicine.

Synchrotrons have gained prominence in modern science for a number of important reasons. The first such reason is probably the remarkable versatility that X-ray beams possess for the purpose of characterizing materials, tissues, processes across the scales. Ever since the discovery of X-ray by Konrad Röntgen in 1899 (for which he was duly awarded the first Nobel prize in Physics), scientists have been discovering ever more new modes and means by which the interaction of X-rays with matter can help shed light on nature's fundamental processes and structures. The remarkable ability of X-rays to penetrate solid matter has given rise to X-ray radiography, a technique for determining the density of materials by examining the "shadow" image obtained on photographic plate or film, or by recording an electronic image using a suitable detector. This technique is not only widely used for medical examination, but also as the basis for industrial non-destructive characterization, e.g. defectoscopy.

Modern developments in X-ray radiography concern the use of multiple angle projections of the same object that provide additional sampling of the same density distribution. These "rich" data sets make it possible to "step out of the picture" and visualize the three-dimensional internal structure of the object of investigation. With the help of modern computing power and mathematical algorithms, such as inverse Radon transform (filtered back projection) and ART (algebraic reconstruction technique), it is possible to perform tomographic reconstruction of 3D structure. An important role here is also played by the so-called phase contrast that arises from interference between the X-ray beams that are transmitted and refracted through the object.

Another important development in X-ray physics took place by 1914, when Max von Laue was awarded Nobel prize for demonstrating experimentally that X-rays diffracted on the atomic crystal lattice. The idea for the experiment grew in his mind as a consequence of discussions of the structure of crystals with Ewald.

Laue's setup used a single crystal and a polychromatic (white) beam of X-rays. In the Bragg experiment, on the other hand, the sample had the form of polycrystalline powder, while the beam was monochromatic, possessing a well-defined wavelength. This allowed Braggs to formulate the celebrated Bragg's law that connects the interplanar lattice spacing d to the X-ray wavelength λ and the sine of half of the scattering angle 2θ:

$$2d \sin \theta = \lambda . \qquad (1)$$

For this development, the Braggs (father and son) were awarded Nobel prize in 1915.

Diffraction represents an example of the broad phenomenon known as X-ray scattering, that differs substantially form that of X-ray transmission, which serves as the basis for X-ray radiograms. As Bragg's law (1) suggests, diffraction allows the determination of very short periods d present within atomic lattices, provided the wavelength λ and the scattering angle 2θ are known with sufficient accuracy. Stringent requirements have to be placed on the geometric precision needed to evaluate the angles, and hence, the incident X-rays must be made as parallel as possible. Good statistics is also necessary, and since only a fraction of the incident beam undergoes scattering, high flux is also a requisite. In other words, bright, parallel beams of X-rays are required in order to undertake diffraction experiments.

The development of X-ray diffraction can be thought of as the first step towards nano-science and nanotechnology, since the length scales under investigation lie in the range of nanometers and below.

The third major realm of application for X-rays is spectroscopy. This concerns the processes of X-ray absorption and re-emission that allow the energetics of atomic systems to be probed. Particularly in those cases when the atoms of interest are present in small quantities and their

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distribution within samples or on their surfaces are of interest, the requirements once again arise of high parallelism and flux.

Originally, the most widespread devices for X-ray generation were similar to Röntgen's tubes: electrons are accelerated through applied voltage and impinge upon the material of the target, producing Bremsstrahlung and characteristic radiation. However, the efficiency of such devices is relatively low, and X-ray beams produced in this way show high divergence and relatively low flux.

In the process of developing particle accelerators, the emission of X-rays by the charged particles travelling along a curved path was discovered, and originally thought of as a parasitic effect. It was soon established, however, that the X-ray beams produced in this way had exceptionally high flux and parallelism, thus answering the requirements for scientific experiments. After several generations, synchrotron sources started to be built with the specific purpose of producing X-ray beams that could be used for studies in physics, chemistry, materials science, biology and medicine.

II. DIAMOND LIGHT SOURCE, UK

The design, construction and operation of Diamond Light Source are a major engineering undertaking. The nature of experiments undertaken requires utmost positional accuracy and stability. In order to achieve it, the specification was developed for the concrete floor requiring the stability of one micron over ten metres per hour, and ten microns over ten metres per day. This is necessary for the reliable operation of the bending magnets and insertion devices that control the passage of the electron beam, and of the optical elements and detectors that condition and monitor the X-ray beams.

The experimental hall of Diamond Light Source rests on a concrete slab that is a ring about 650 metres in circumference and about 30 metres wide. The ring was built on 1500 piles that were driven down 12-15 meters deep. This engineering solution was necessitated by the nature of the chalky clay soil that the facility was built on. The soil swells and shrinks readily depending on the moisture level, depending on the water table varying by as much as 8 metres each year. Piled foundations ensure that the slab rests on deeper, more solid layers of rock, thus providing the flat and stable platform required.

On top of this large scale, high precision civil engineering structure, some of the most sophisticated and refined engineering instruments and devices are housed. These are advanced high power electronic and magnetic systems, including cryogenic superconducting devices; monitoring equipment and control apparata; and thousands of translation and rotation axes and stages. A key parameter of any synchrotron is the stability and reliability of the stored electron beam, and this can only be achieved by employing fast feedback systems and controls. Dozens of specialized engineering companies were engaged to deliver specific solutions, while the integration of these systems into the new facility was the principal responsibility of Diamond engineering team.

III. THE JOINT ENGINEERING, ENVIRONMENT AND PROCESSING (JEEP) BEAMLINE

A. Engineering research at Diamond

Historically, synchrotron laboratories were mainly the preserve of physicists, chemist and crystallographers. However, the development of ever more powerful and highly energetic beams led to the realisation that they could be used to probe the internal structure and state of substantial, bulk solid objects, such as engineering components and assemblies. Examples are provided by railway rails, jet engine blades and casings, aircraft wings, power generation turbines, etc.

The requirements for the study of such objects, however, differ substantially from those for scientific research. In physics and chemistry research the sample is often relatively small and needs to be studied in reciprocal space, i.e. by relative rotation of the sample and detector(s) with respect to the beam. In contrast, in engineering research the sample itself is extended in real space, heavy, and often requires large items of ancillary equipment to manipulate it, e.g. large capacity positioning tables, mechanical presses and loading devices, furnaces and reaction cells, etc.

The variety of modern engineering is such that it is simply impossible to envisage all the applications. For this reason it seems logical to identify *flexibility* and *available space* as the principal requirements for construction.

The Joint Engineering, Environment and Processing (JEEP) beamline I12 on Diamond was conceived as the first synchrotron beamline designed and dedicated to engineering research.

B. Beamline description and functionality

The Joint Engineering, Environment and Processing (JEEP) beamline I12 at Diamond Light Source near Oxford (UK) is a multi-purpose high energy instrument based on a 4.2 T superconducting wiggler that produces a broad, smooth spectrum of X-rays with a useable range of energy from 50keV to 150keV. The beamline was designed to address the wide range of problems in engineering science where synchrotron X-rays provide special, often unique insight. The two principal modes are (i) the study of deformation in single crystal and polycrystalline materials by monochromatic or white-beam diffraction, [1]; (ii) the imaging of the internal structure of materials and objects using X-ray radiography and tomography [2]. A third mode, small angle X-ray scattering, will be possible once the beamline is fully commissioned, and can be used to study amorphous materials and longer-range periodic structures.

The beamline consists of two in-line hutches. The first experimental hutch (EH1) is located at 54m from the source and receives the beam up to 50mm(H)×15mm(V) in cross-section. A series of slits allows beam collimation, typically down to 100 microns by 100 microns for energy dispersive diffraction. The sample positioning table allows rotation and *x-y-z* translation across the beam. A versatile detector table provides precise positioning for imaging cameras and diffraction detectors. The second experimental hutch (EH2) provides a sample position at 95m from the source and the full beam with the spot size up to 100mm(H)×30mm(V). It is used for studying large and/or heavy samples under static conditions or for different types

of in-situ processing experiments. Setting up these experiments can be done whilst EH1 is operational. The positioning table in EH2 has a capacity of 2 tonnes. It provides translation ranges of 1 metre in both, horizontal and vertical directions, and full rotation.

The example experiment described below used one particular diffraction mode (white beam energy dispersive). The setup is illustrated in FIG.1 and described in the following section. The experiment also provided a demonstration of the capabilities of the new instrument [3].
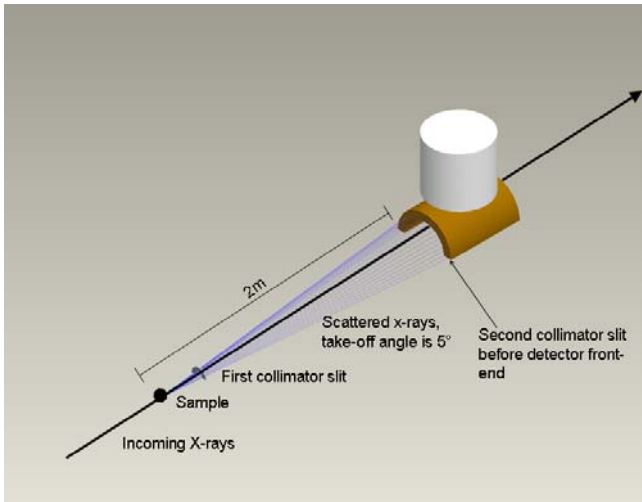
C. Experimental setup

Figure 1. Experimental setup for white beam, energy dispersive diffraction at JEEP. The incoming beam scatters from the sample (indicated by the solid dot), passes through sample and detector collimators and is registered by the 23-cell energy-discriminating horseshoe detector [3].

The example engineering experiment described in [3] was devoted to the interrogation of grain interaction and deformation hardening in polycrystalline metallic alloys. The importance of this theme is defined by the fact that most alloys used for structural applications are in polycrystalline form, i.e. aggregates of many individual grains. The cohesion between these constituent gives rise to internal stresses (inter-granular and intra-granular) that play a crucial role in determining the deformation response. Steady progress is being made in the understanding of the nature and evolution of internal stresses in polycrystals [4,5]. A fruitful line of attack on this complex problem involves the combination of deformation modelling with experimental data obtained non-destructively and at the appropriate scale. Diffraction of penetrating radiation, such as the high flux, high brightness synchrotron X-rays, provides a uniquely powerful tool for the purpose of experimental characterisation. The strains associated with each phase can be quantified separately, allowing strain partitioning between phases to be assessed.

The state of strain in a pre-deformed sample of polycrystalline alloy Ti-6Al-4V was investigated, and the results interpreted the results of the deformation response of individual phases and grain groups. An additional result of the study was the initial evaluation of the new energy-dispersive X-ray diffraction (EDXD) setup at Diamond Light Source.
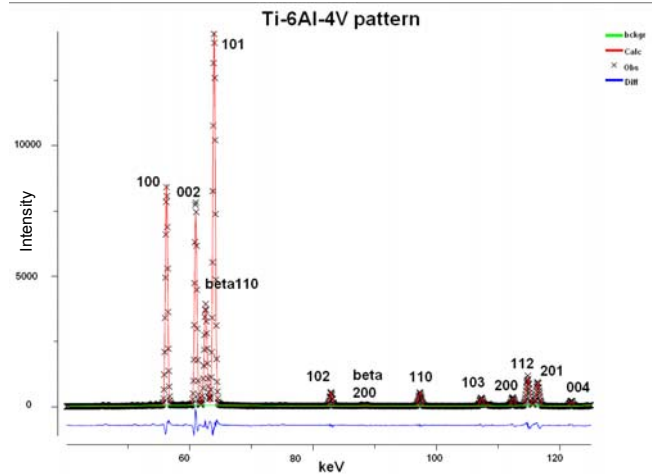

Figure 2. Energy-dispersive diffraction pattern of the Ti-6Al-4V alloy. The data was collected using one cell of the horseshoe detector. The pattern was analysed by Pawley refinement [6] (from (3)).

Aerospace grade titanium alloy Ti-6Al-4V (BS EN 3456:2009), is a two-phase alloy that contains the majority (~95%) hexagonal close-packed (HCP) α-phase and minority (~5%) body-centred cubic (BCC) β-phase. The particular interest for the present study was to analyze inter-phase strains (α vs β) and orientation-dependent strains (within α-phase). This can be accomplished by Pawley refinement of energy-dispersive diffraction patterns [2,6,7] and the consideration of phase- and orientation-specific lattice parameter variation. FIG.2 shows an example of the indexed and fitted Ti-6Al-4V energy-dispersive diffraction pattern obtained.

In hexagonal close-packed (HCP) crystals of α-Ti, the elastic and plastic properties exhibit a large degree of anisotropy, with a 30% difference between single crystal Young's moduli perpendicular and parallel to the basal plane [4]. Easy slip in HCP crystals is associated with the close-packed directions in the basal plane, resulting in the easiest yield and highest ductility when loaded perpendicular to the c-axis, compared to loading along the c-axis. This strong directional dependence of the elastic and plastic properties of single crystals of α-Ti is also reflected in the macroscopic behaviour of polycrystals.

Four point bending imposes a simple linear variation of total longitudinal strain within a beam. The onset of plastic flow occurs within the sample when the maximum strain at the surface exceeds the yield strain $\varepsilon_Y = \sigma_Y / E$, where σ_Y is the macroscopic yield stress, and E the macroscopic Young's modulus. Upon further loading the bent beam sample experiences progressively increasing tensile and compressive plastic strains that persist upon the removal of the external bending moment. The permanent plastic strain (eigenstrain) acts as the source of the residual stress state in the sample. The total strain consists of the sum of plastic strain (eigenstrain) and the equilibrating residual elastic strain, the latter being related (via Hooke's law) with the residual stress [8,9].

The samples considered in this study were 1.7×4×60 mm³ bars of Ti-6Al-4V alloy machined from the middle of a plate produced by vacuum arc melting above the phase transus temperature followed by cross-rolling and annealing

to minimise the crystallographic texture. The samples were plastically deformed in the four-point bending configuration and unloaded prior to diffraction measurement, creating a locked-in residual stress-strain state. The bending arrangement created strain variation across the 4mm dimension of the sample.
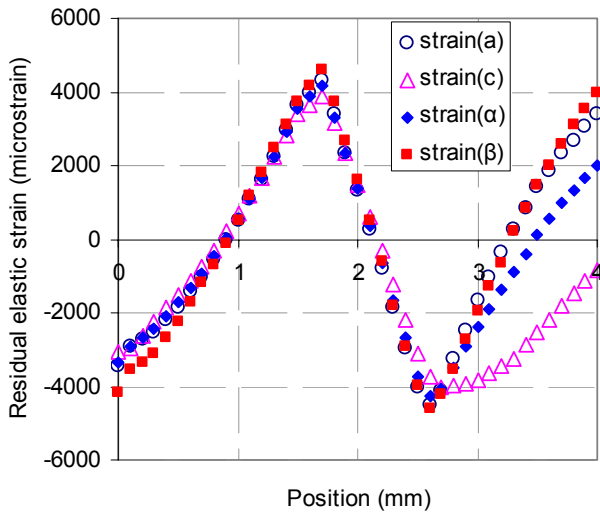

Figure 3. Measured residual elastic strain profiles in the two phases (α and β) and in the crystallographic directions a and c within the HCP α-phase [3].
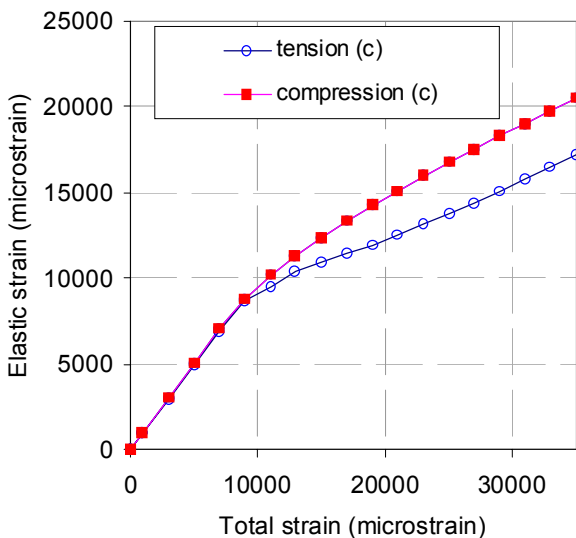

Figure 4. The illustration of the hardening asymmetry observed in the grains of c-group (HCP a-phase grains of particular orientation).

The measured residual elastic strains are illustrated in FIG.3. It is apparent that distinct differences are observed between the response of the two phases, and also within the HCP phase depending on the orientation of the crystallites with respect to the scattering vector. It is apparent that strong asymmetry is also observed between the tensile and compressive senses of loading.

The results were interpreted [3] in order to extract the relationship between the total strain applied to the polycrystalline aggregate (horizontal axis) and the measured elastic lattice strain (vertical axis). The results are illustrated in FIG.4.

A significant tension-compression asymmetry of yield (in strongly textured Ti alloy polycrystals) has been reported before [10]. However, the present results are different in that they relate to the specific response of a particular pseudo-phase (c-group) embedded in a polycrystalline ensemble that is close to random in terms of orientation distribution. The effect reported here requires separate further analysis, but is likely to be related to the scarcity of slip systems that can be active in the grains of this orientation, and the difference in the crystal rotation associated with plastic slip under tensile and compressive loading [11].

The data interpretation described above required highly accurate measurement of residual elastic strains. The typical error of strain determination was below 50 microstrain (unit of 10^{-6}), as evidenced by the Pawley refinement error. The interpretation presented relies wholly on the availability of highly accurate strain measurement. Thus, it highlights the superior performance of the new I12 JEEP instrument for the purpose of advanced strain analysis in engineering materials and structures.

IV. CONCLUSION

Without seeking to be in any way exhaustive, this short report touched upon the different aspects of engineering at Diamond Light Source, from the design, construction and operation of the facility to the application of the facility for engineering research.

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