Methods of XRD Diffraction

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PROTO

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About Proto Manufacturing LTD

PROTO Manufacturing LTD is one of the fastest growing x-ray diffraction companies worldwide, specializing in many avenues like powder diffraction, residual stress measurement, and Laue single-crystal orientation. They pride themselves on making scientific and engineering advancements possible by using their intelligent staff as well as highly accurate and precise x-ray diffractometers. PROTO currently has products that work with XRD residual stress measurement, powder diffraction, Laue single crystal, and many more methods of data collecting. The division I was working in, XRD, creates x-ray detectors and is responsible for fixing, optimizing, and overall testing these machines.

<u>Many methods of XRD were used,</u> and all have their pros and cons. One of my many jobs was to test these devices and collect data to help the company decide which methods were best for certain avenues and price points.

What is XRD?

X-ray Diffraction (XRD) is a technique used to determine the crystallographic structure, structural properties and atomic arrangement of materials. This non-destructive method can be done in many ways, but all use the same principle of irradiating a material with incident X-rays, and then measuring the scattering angles and intensities that leave the material. This method works using Bragg's law,

$$2d\sin\theta = n\lambda$$

Where θ is the incident angle, d is the space between diffracting planes, n is an integer and λ is the wavelength of the beam.

At Proto Manufacturing LTD, many machines use XRD methods to collect data, such as Laue single crystal, residual stress, high-resolution XRD and single crystal crystallography machines.

My Role at Proto Manufacturing LTD

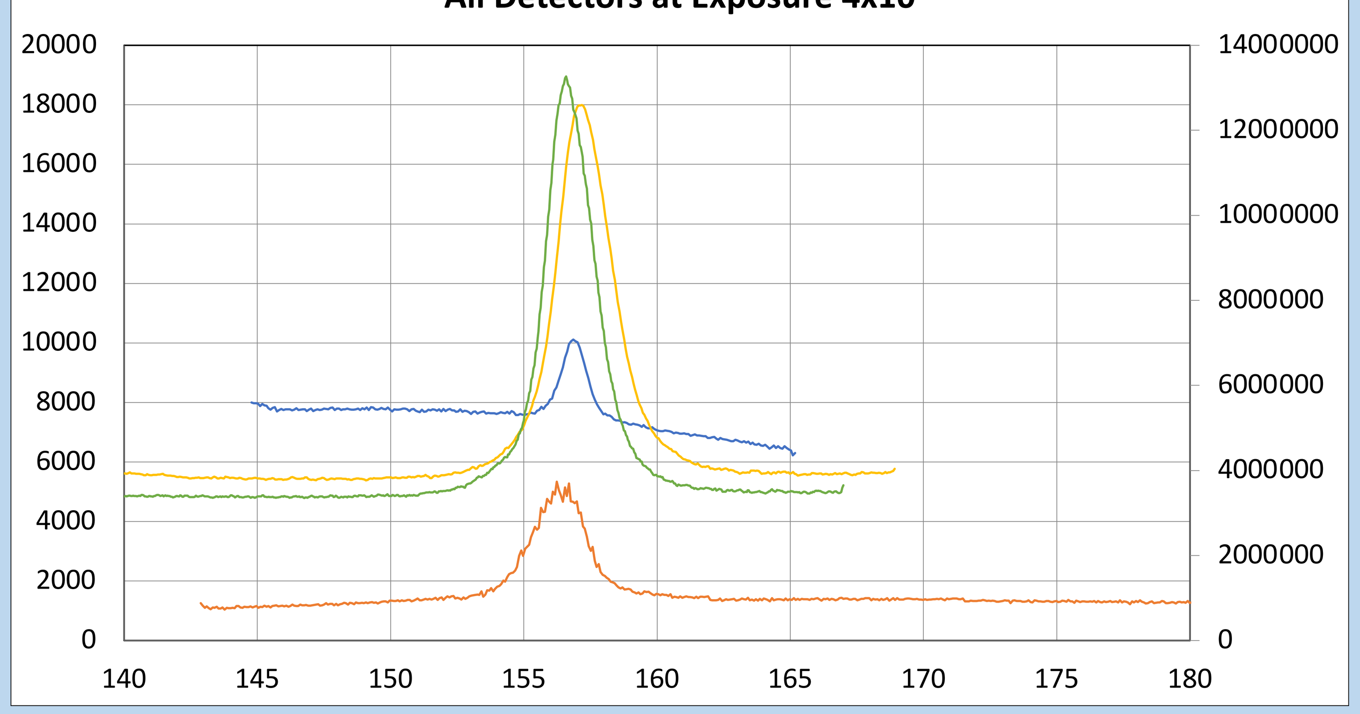
In my role I worked alongside Brian Simpson using a residual stress XRD machine to collect data on multiple detectors and x-ray tubes. For this data collection I used 4 detectors, the Current Wide Proto, the Dectris, the MiniPix and the New Wide Prototype detector. Different X-ray tubes and materials were used to test the data collection capabilities of each detector to find the best detectors to use for specific elements. The new wide system was also used for another project in which I collected specific data at separate gain values to determine the optimal settings for the detector itself.

Another project I worked on was with Robert Drake, where I maintained and positioned the machinery for the single crystal diffraction XRD machine. As this was a prototype machine, it was entirely done manually.
Other projects I worked on were the rebuilding of a preowned SAXS machine and a research project about CRL's and if they are optimal to use for the company.

Residual Stress Data Collection

Throughout this first project we used the residual stress machine to measure data on multiple detectors and x-ray tubes to determine which were best to use and which we could optimize to a greater potential. The four detectors used were Current Wide and New Wide from PROTO, Dectris, and a MiniPix. As each detector had its own degree of detection, I created a excel macro which allowed all 4 to be measured and aligned onto one field of degrees. This allowed us to compare the data received, such as their maximum, their background noise, FWHM, etc. Each detector was used on Cu, Cr, Mn, and V x-ray tubes, and were placed into a handmade algorithm which sorted all the data into tables to help compare each detectors result. Macros were used to help align, straighten, and properly scale the data. This project was completed in 3 months and overall proved that the New Wide system, a new prototype created by my supervisor, was by far the best detector out of the four.

Below you will see one of over 100 graphs produced comparing all detectors at the exposure time 4x10 (4 seconds, 10 frames), as well as the table displaying a sample of the collected data. The graph is showing the profile of Fe powder using a Cr X-Ray tube.



Dectris = ▢ Current Wide = ▢ New Wide = ▢ MiniPix = ▢

All data was taken at the exposure 4x10

Exposure	Max	Background Std.dev	FW/HM	SNR	Peak to Background
4x10	17993.89	710.80	2.33/11900.07	8.16	3.09
4x10	5337.4	1320.07	2.17/3328.74	7.54	4.04
4x10	10112.46	552.03	1.15/8719.8	13.27	1.38
4x10	13268285.07	6250.53	2.05/ 8416016.42	10.61	3.72

Single Crystal Data Collection

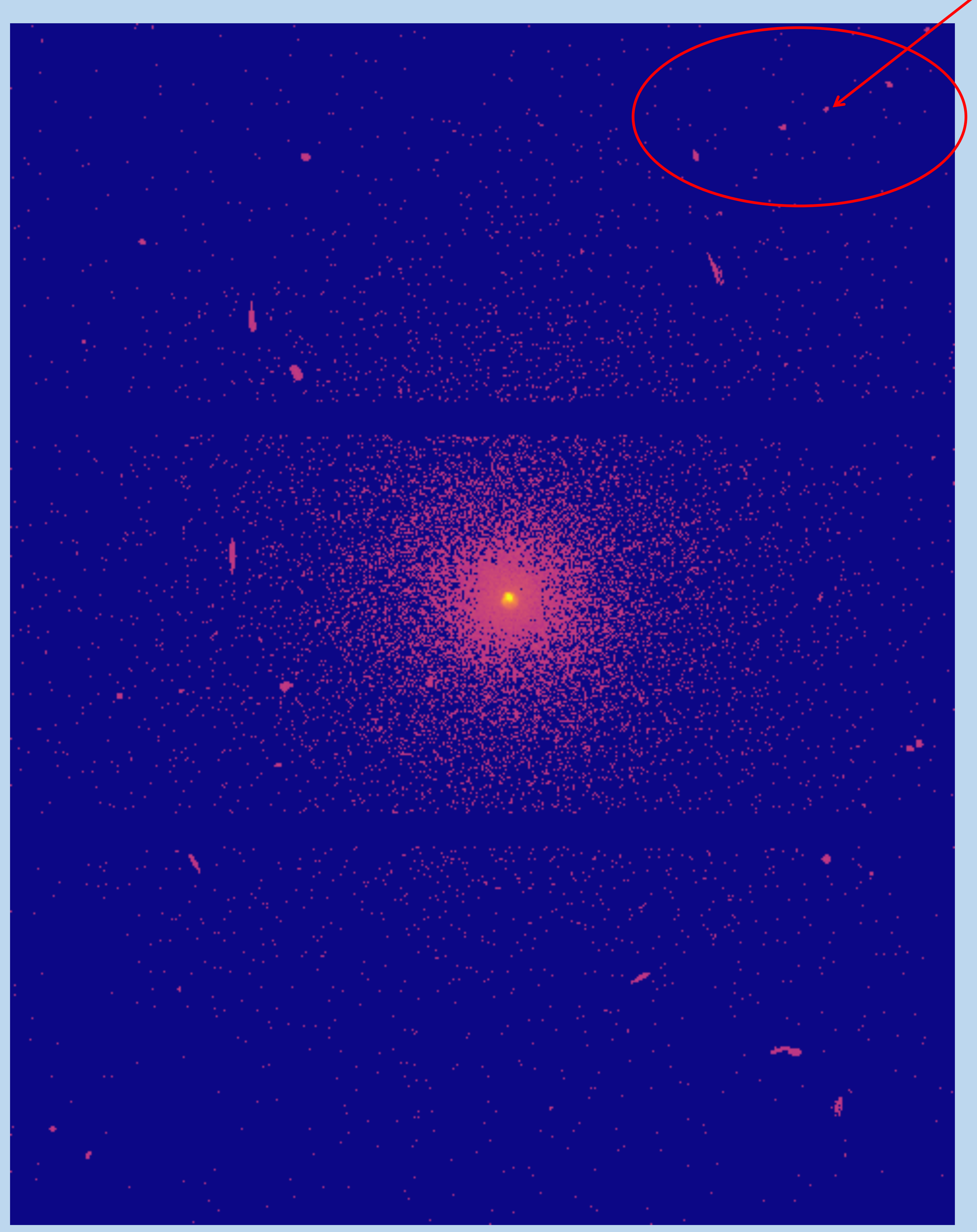
The second project I worked on during my placement was the building and calibration of the Single Crystal Data collection machine. This machine was a prototype-tester for the company, so it was not fully motorized. Based on this, I had to personally calibrate it by hand, to millimeters of accuracy. Using a Two-mirror Kirkpatrick–Baez system, the beam was focused to a small point, and I positioned two pinhole collimator to allow for just the beam itself to hit the detector, a PILATUS3 R 300K.

We used single crystal rubies, as well as sugar crystals, to calibrate the machine and properly align everything to allow single crystal diffraction to occur. One of my other roles during this project was to mount the crystals to goniometers, using resin and microscopes.

The diffraction of the single crystal was done using monochrome radiation, collimated to concentrate and focus on the sample, which in this case was ruby. This diffraction could occur as the conditions would satisfy Bragg's law.

The image to the right is collected data from when the sugar crystal was being used to calibrate the machine. The dots are from the diffraction that occurred while the crystal was rotated on a goniometer on the phi axis.

These dots are occurring while the crystal is rotating on the phi axis, while mounted on a goniometer. The angle the crystal is mounted on, as well as the monochromatic radiation, allow for Bragg's Law to be fulfilled.



Other Methods of Data Collection

There are many other methods of data collection done at PROTO besides Residual Stress measurements and Single Crystal Diffraction. Some of the other methods used in the XRD department are:

Powder diffraction uses a powder diffractometer. This is similar to single crystal diffraction, but the distinction between powder and single crystal diffraction is the degree of texturing in the sample. While single crystals are said to be anisotropic, in powder diffraction samples, every crystalline orientation is represented equally, which is considered isotropic.

Laue single crystal is a method of data collection like single crystal diffraction but uses chromatic radiation and can investigate the structure and orientation of single crystal material. This can be used to identify the crystal's growth direction as well as what type of crystalline structure is present.

MetalJet Integration is a method of diffraction involving the world's brightest microfocus x-ray tubes. This method provides superior spot quality in x-ray applications. This system can show phase contrast, crystallography, diffraction at high energies, and small angle x-ray scattering.

Conclusion/Future Work

Projects 1 and 2 were both complete within the 4-month period of this co-op. Project 1 was able to aid the XRD department in deciding which detectors are more optimal for use in machines given to clients, as well as giving information on what settings the New Wide detector should be used on to get the best and most optimal results. My portion of Project 2 was completed and has been taken over by the programming department who are aiming to code more types of data collection for the system. My only recommendation is to motorize all the new machines as this machine took over two weeks to calibrate by hand through trial and error.

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Information

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