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Nondestructive residual strain measurement using high energy x-ray diffraction

by

Mohammad Yousef Al-Shorman

A dissertation submitted to the graduate faculty
in partial fulfillment of the requirements for the degree of

DOCTOR OF PHILOSOPHY

Major: Condensed Matter Physics

Program of Study Committee:
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Iowa State University
Ames, Iowa
2008

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DEDICATION

To my parents, my wife and all of those who endured rough times to help me complete this work.
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ABSTRACT

A nondestructive high energy (60 keV) x-ray diffraction method to measure the internal strain depth-profile in materials was developed using a standard 320 kV p laboratory x-ray source. Traditional x-ray strain measurements are limited to few microns of depth due to the limited penetration of Cu $K_{\alpha}$ and Mo $K_{\alpha}$ radiation if a synchrotron or neutron source is not used. The high energy used allows for greater penetration without a synchrotron or neutron facility. Results for aluminum with penetration depths of 1000 $\mu$m and for titanium with penetration depths of 300 $\mu$m are demonstrated. The spatial resolution of this depth-profile is 50 $\mu$m to 125 $\mu$m depending on the collimation and attenuation of the sample. Sensitivity to a lattice parameter change of 0.001 Å is demonstrated. An energy dispersive HPGe detector is used to perform fixed-angle diffraction measurements. The strain depth-profile measurement is performed by observing the change in the position of the diffraction peaks in the energy dispersive spectrum. A simulation program of the diffraction system for modeling and validating the experimental setup is developed. A new technique to measure the strain using the natural width of the tungsten $K_{\alpha_1}$ line and the diffraction peak normalized intensity is presented. The results of the energy dispersive technique and the tungsten $K_{\alpha_1}$ line width technique are compared to the results obtained using high energy XRD $\theta$-2$\theta$ scans technique. Finally we present a preliminary study of the thermal relaxation of residual stress.
CHAPTER 1. Introduction

There are two types of stress that are realized in materials: Applied stresses which exist because of external forces acting on the material and residual stresses which remain even after all applied forces have been removed. Residual stress is a compression or a tension that exists in the material without applying an external load. Generally compressive or negative residual stresses delay crack initiation and propagation while tensile or positive residual stresses reduce the durability of the materials.

The basic cause of the residual stress is the nonuniform plastic flow due to some previously performed operations. Welding, casting, bending, twisting, grinding and shot peening are examples of such operations (1–4).

Residual stresses can be classified by the scale over which they self-equilibrate. Macroscopic residual stresses operate over large distances (few millimeters) while microscopic residual stresses operate over the grain scale of the material (3–5).

1.1 Contributions of this thesis

Residual stresses can be measured using a variety of techniques. Each technique has its own advantages and disadvantages. A summary of most of the currently available techniques is shown in table 2.1. The currently available techniques can measure residual stresses in almost all types of materials with a reasonable success. However, most of those techniques are destructive if a stress depth profile is to be constructed. Among all of the methods discussed in chapter 2, only neutron and synchrotron diffraction experiments and some of the electric/magnetic methods can construct stress/strain depth profiles nondestructively. Even those nondestructive methods have their own limitations that make them impractical in many cases. Namely, synchrotron and neutron diffraction experiments are limited to a handful of facilities. The beam time is very limited and doing routine measurements on a large
number of samples is not viable. In this thesis we present a new technique that provides a similar or even a better depth profile resolution than the currently available techniques, not only by using a more tightly collimated beam, but also by presenting a more detailed understanding of the various instrument functions such as the beam divergence and the detector resolution. It should be noted here that most of the currently published measurements that utilize a synchrotron source rely on a position sensitive detector by performing angle-dispersive diffraction scans around the sample. The experimental technique we are presenting in this thesis relies mainly either on an energy dispersive detector or a new technique that uses a normalized intensity to calculate the strain. Some of the synchrotron work such as the one in reference (6) used the energy dispersive transmission diffraction technique. The work we are presenting here utilizes a backscattered diffraction, that provides a strain depth profile with a good spatial resolution, using a lab based setup which means a better accessibility and facilitates the process of doing routine measurements on a large number of samples.

Electric/magnetic methods also have their own limitations. The methods that utilize magnetic techniques such as magnetoelasticity suffer from lack of spatial resolution. Most surface treated metals have the compressive or tensile stresses regions within few hundreds of microns from the surface. A measurement resolution of 1 mm is not practical in these cases. Other techniques that utilize eddy current suffer from their sensitivity to the plastic deformations and surface roughness in the material. In some materials, such as titanium, measuring stress depth profiles using eddy current techniques is not possible because of the measurement sensitivity to the cold work (8).

In this thesis, a new nondestructive lab based residual strain depth profile measurement technique is presented. An industrial high energy x-ray source with a tungsten target material that generates x-rays with energies up to 320 keV is used. This high energy x-ray tube power leads to a high tungsten characteristic lines intensity. The tungsten $K_{\alpha_1}$ line at 59.318 keV is used in most of the measurements. This high energy allows for a penetration depth of more than 1 mm in aluminum and about 300 $\mu$m in titanium. The resolution of the measurement depends on the type of the material and many other factors discussed in section 3.6.1. A measurement resolution of 50 $\mu$m to 150 $\mu$m is typical depending on the x-ray linear attenuation coefficient of the sample material. Measurements on materials with

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1Cold work: permanent deformation caused by application of an external force to a metal below its recrystallization temperature (7).
high x-ray attenuation like Ti give better resolution as shown in section 3.6.1. Like any other diffraction technique, this technique is limited to materials with a crystalline structure. The geometry of the sample should not prevent the diffracted signal from reaching the detector. It is worth noting here that different characteristic lines from tungsten or even other target materials might be more appropriate depending on the type of the sample material. We show in sections 3.6.1 and 3.8 that the tungsten $K_{\alpha 1}$ at $E_{K_{\alpha 1}} = 59.318 \text{ keV}$ with a natural line width $FWHM = 43 \text{ eV}$ is appropriate for measuring strains that are less than $\pm0.01 \text{ Å}$. Uranium $K_{\alpha 1}$ line at $E_{K_{\alpha 1}} = 98.439 \text{ keV}$ with $FWHM = 100 \text{ eV}$, for example, is more appropriate for measuring more extreme changes in the lattice constant. More information about possibilities with other target materials can be found in references (9; 10).

The energy dispersive x-ray diffraction system (EDXRD) described in this thesis can measure strain-depth profiles using three different techniques. The first one is a new technique we are proposing that utilizes the width of the incident characteristic line and measures the strain, for the first time, using a normalized peak intensity value. Because of the characteristic line shape, different strains lead to different diffracted intensities. The second technique measures the strain by measuring the location of the diffraction peak in energy space at a fixed diffraction angle. The third technique uses the traditional angle dispersive XRD method but at very high energies and small angles. A detailed understanding of the various instrument functions is presented throughout this thesis and in many cases those instrument functions are used to our advantage. A strain depth profile of a 10A shot-peened aluminum surface measured using these three different techniques is shown in figure 1.1. Finally, this lab based technique is relatively cheap when compared to the cost of a neutron or a synchrotron facility. Depth profile measurements can be performed on a large number of samples, not only in materials like aluminum and titanium, but also in any material that has a lattice structure.

Finally we present a preliminary study of the residual stress thermal relaxation due to the exposure to a high temperature for different periods of time. Then we present preliminarily results of the residual stress thermal relaxation due to exposing the same sample to the same heat cycle multiple times.

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2Shot peening: a process used to produce a compressive residual stress layer in a metal surface by impacting the surface with small spherical media called shot with energy sufficient to create plastic deformation. Peening intensity is determined by exposing several (minimum of 4) Almen strips to the blast stream for increasingly longer times and plotting the arc-height vs. exposure time. Peening "Intensity" is the value of the arc-height curve at time T1 that is within 10\% of the arc-height curve value at twice the time or T2. An Almen strip is a thin strip of SAE 1070 steel used to quantify the intensity of a shot peening process. Shot peening is used throughout this thesis as a mean to create residual stress in the samples under investigation.
Figure 1.1 Change in [111] lattice planes spacing of a 10Å shot-peened aluminum measured using the three different methods using the EDXRD setup described in this thesis.
CHAPTER 2. Literature Review

2.1 Residual stresses measurement techniques

Residual stresses can be measured using a variety of techniques. Some of those techniques are destructive while others can be either very expensive and/or limited to the surface of the sample. In this chapter, a review of the already existing residual stress/strain measurement techniques is presented. Then we will discuss the various causes of the residual stresses.

2.1.1 Hole drilling

Hole drilling is a widely used simple, cheap, and portable technique for measuring macro residual stresses. It works on crystalline and amorphous materials. In this process a hole is drilled into the sample then subsequent measurements of the surface stress are then performed using strain gauges. The problem with this technique is that it is a destructive technique. The resulting stress gradient is difficult to interpret and has a limited sensitivity and the penetration depth is limited by the size of the hole (11–13). However, hole drilling measurements are usually in excellent agreement with XRD layer removal technique (14). Detailed comparisons between the hole drilling technique and x-ray diffraction can be found in the references (14–16).

2.1.2 X-ray diffraction

This method depends on the fact that the spacing of the atomic planes in a crystalline material is altered by the stress. The fractional change in the planes spacing is the strain from which the stress can be calculated. Traditionally $Cu \, K_\alpha \, 8.0 \, keV$ and $Mo \, K_\alpha \, 17.5 \, keV$ are used in such a measurement. Using these energies, the x-ray method is nondestructive only if the stress at the surface is to be determined. If information about subsurface stress is required then material must be removed to expose the subsurface
area, but then the measurement becomes destructive. In this thesis using higher energies is proposed so
that nondestructive subsurface measurements can be performed.

XRD was applied to stress measurement in 1925 (17). In those days the diffracted beams were
recorded on photographic films. This method is suitable only for materials that give sharp diffraction
lines, but it does not work on the materials that produce broad diffraction lines. In 1953 a notable
advance was made (18). Stress measurement using x-ray diffraction became easier and could be used
on a wider range of materials using the new diffractometers. Since then this method became a routine
measurement in many labs (19). In this method, the sample is irradiated with mono-energetic x-rays,
such as \( \text{Cu } K_\alpha \) 8.0 keV or \( \text{Mo } K_\alpha \) 17.5 keV, the crystal planes diffract some of these x-rays, then a
detector moves around the sample to find the angular position where diffracted x-rays are located. The
locations of the peaks enable the user to calculate the stress on the surface.

XRD has the advantage of being fast and cheap. However, this technique is limited to crystalline
materials. The penetration depth is limited to 50 \( \mu m \) in aluminum and 5 \( \mu m \) in titanium if \( \text{Cu } K_\alpha \) or
\( \text{Mo } K_\alpha \) radiation is used. The resolution is limited to 20 \( \mu m \) depth and 1 mm laterally (20). It can be
used to measure micro and macro stresses. One disadvantages of XRD is the limitation on the size and
geometry of the sample. It has to be such that the x-ray can hit the measurement area and still diffract to
the detector without getting obstructed. Problems may also occur if the surface is too rough, so surface
condition is a consideration in traditional XRD measurements (21; 22).

2.1.2.1 Basic stress/strain relationships

Consider an infinitesimally small cube in a body under stress. There are three components of stress
acting on each face as shown in figure 2.1. These nine components can be represented by the tensor:

\[
\sigma = \begin{pmatrix}
\sigma_{xx} & \sigma_{xy} & \sigma_{xz} \\
\sigma_{yx} & \sigma_{yy} & \sigma_{yz} \\
\sigma_{zx} & \sigma_{zy} & \sigma_{zz}
\end{pmatrix}
\]

Under equilibrium there is no rotation. Thus \( \sigma_{xy} = \sigma_{yx} \) and \( \sigma_{xz} = \sigma_{zx} \) and \( \sigma_{yz} = \sigma_{zy} \). The stress tensor
will then be reduced to six independent components. Those six independent components can further be
reduced to three components by transforming the stress tensor to the diagonal state in another rotated
Hooke’s law states that:

\[ \sigma_x = E \varepsilon_x \]

where \( E \) is the modulus of elasticity and \( \varepsilon_x \) is the strain. A stress in one direction will not only produce a strain in that same direction, but it will also introduce strains in directions perpendicular to the strain original direction:

\[ \varepsilon_y = \varepsilon_z = -\nu \varepsilon_x = -\frac{\nu \sigma_x}{E} \]

where \( \nu \) is Poisson’s ratio. Combining the strains introduced by Hooke’s law with the strains introduced by Poisson’s ratio, the three principal stresses can be related to the three principal strains in the same system of coordinates by the following set of equations:
\[ \varepsilon_1 = \frac{1}{E} [\sigma_1 - \nu (\sigma_2 + \sigma_3)] \]
\[ \varepsilon_2 = \frac{1}{E} [\sigma_2 - \nu (\sigma_3 + \sigma_1)] \]
\[ \varepsilon_3 = \frac{1}{E} [\sigma_3 - \nu (\sigma_1 + \sigma_2)] \]

Some of the special cases for this set of equations can be useful. For example one can set \( \sigma_3 = 0 \) when calculating a planer stress or \( \varepsilon_3 = 0 \) in the case of a planer strain.

Usually it is more convenient to calculate the stress/strain in spherical coordinates. See figure 2.2.

![Ellipsoids of stress/strain using units employed in XRD stress analysis.](image)

The strain in the general direction \((\psi, \phi)\) can be written as:
\[ \varepsilon_{\psi\phi} = \varepsilon_1 \alpha_1^2 + \varepsilon_2 \alpha_2^2 + \varepsilon_3 \alpha_3^2 \]

where
\[ \alpha_1 = \cos \phi \sin \psi \]
\[ \alpha_2 = \sin \phi \sin \psi \]
\[ \alpha_3 = \cos \psi \]

similarly
\[ \sigma_{\psi\phi} = \sigma_1 \alpha_1^2 + \sigma_2 \alpha_2^2 + \sigma_3 \alpha_3^2 \]

Substituting \( \varepsilon_1, \varepsilon_2, \varepsilon_3 \) values in \( \varepsilon_{\psi\phi} \) equation above leads to:
\[
\varepsilon_{y\phi} = \frac{1+\nu}{E} \left[ \sigma_1 \alpha_1^2 + \sigma_2 \alpha_2^2 + \sigma_3 \alpha_3^2 \right] - \frac{\nu}{E} (\sigma_1 + \sigma_2 + \sigma_3)
\]

If energies such as Cu K\(\alpha\) and Mo K\(\alpha\) are used in the diffraction setup, then it is reasonable to assume that only surface stresses exist because the penetration of those x-rays is usually less than 10 \(\mu m\) to 20 \(\mu m\). If \(\sigma_3 = 0\) the strain equation above then reduces to:

\[
\varepsilon_{y\phi} = \frac{1+\nu}{E} \sigma_0 \sin^2 \psi - \frac{\nu}{E} (\sigma_1 + \sigma_2)
\]

The equation above is the main equation in calculating the stress using x-ray diffraction method. Now that the basic formulation needed to calculate the stress from the strain is known, the different methods that utilize x-ray diffraction to measure the residual stress will be discussed next.

1. The single-angle method:

This method is generally less sensitive than the two-angle or \(\sin^2 \psi\) methods that will be discussed later, primarily because the possible range of \(\psi\) is limited by the diffraction angle \(\theta\). In this method, a collimated beam hits the sample at a known angle from the sample surface normal. X-rays diffract from the sample, forming a cone of diffracted radiation. The diffracted x-rays are recorded using a film or a position-sensitive detector. The presence of a stress in the sample surface varies the lattice spacing slightly between the diffracting planes. This will result in slightly different diffraction angles on either side of the x-ray beam. The strain can then be calculated using the width and location of the diffraction peak on each side of the beam.

2. The two-angles method:

In an equation that we derived above, the strain is related to the three principal components of the stress by the equation:

\[
\varepsilon_{y\phi} = \frac{1+\nu}{E} \left[ (\sigma_1 \cos^2 \phi + \sigma_2 \sin^2 \phi) \sin^2 \psi + \sigma_3 \cos^2 \psi \right] - \frac{\nu}{E} (\sigma_1 + \sigma_2 + \sigma_3)
\]

If the x-ray used does not penetrate more than few microns below the surface then a surface stress can be assumed, i.e \(\sigma_3 = 0\). The equation above becomes:

\[
\varepsilon_{y\phi} = \frac{1+\nu}{E} \left[ (\sigma_1 \cos^2 \phi + \sigma_2 \sin^2 \phi) \sin^2 \psi \right] - \frac{\nu}{E} (\sigma_1 + \sigma_2)
\]
the surface stress can be written as:

$$\sigma_\phi = \sigma_1 \cos^2(\phi) + \sigma_2 \sin^2(\phi)$$

and the strain, which is not necessarily a plane strain, can be written as:

$$\varepsilon_{\psi\phi} = \frac{\Delta l}{d_0} = \frac{d_{\psi\phi} - d_0}{d_0}$$

The lattice planes spacing for the sample $\psi \phi$ orientation can now be written as:

$$d_{\psi\phi} = \left[\left\{\frac{1+v}{E}\right\}_{hkl} \sigma_\phi d_0\right] \sin^2 \psi + \left[\left\{\frac{v}{E}\right\}_{hkl} d_0 (\sigma_1 + \sigma_2) + d_0\right]$$

The equation above is a linear relation between $\sin^2 \psi$ and $d_{\psi\phi}$. The slope of the line is:

$$\text{Slope} = \left[\left\{\frac{1+v}{E}\right\}_{hkl} \sigma_\phi d_0\right]$$

By measuring $d_{\psi\phi}$ at two different orientations, the surface stress $\sigma_\phi$ can be calculated.

3. $\sin^2 \psi$ method:

This method is similar to the two-angle method, except that $d_{\psi\phi}$ is determined for multiple $\psi$ angles. The least squares fitting method is then used to find the slope of the line from which the surface stress is calculated. This method is the standard stress measurement in Japan and Germany. It does not offer more accuracy compared to the two-angles method, but it shows that using XRD is possible on the sample of interest by showing the linearity of the relation between $d_{\psi\phi}$ and $\sin^2 \psi$.

4. Full-tensor determination:

The lattice planes spacing at a given point in the sample can be calculated as a function of $\psi$ and $\phi$ without assuming a plane stress (23; 24). However, the required extensive data collection exceeds what is acceptable for routine testing. Subsurface measurements can be done destructively and corrected for the presence of subsurface stress gradients (25).
2.1.3  Synchrotron

Synchrotrons provide high energy very intense x-ray beams. Those high intensities allow for 20 mm penetration in aluminum using 80 keV beam (26) and 3D strain maps can be constructed for depths up to 100 µm in most practical materials using 5 keV to 14 keV synchrotron source (27). White synchrotron beams with energies ranging from 10 keV to 200 keV were used to perform energy dispersive fixed angle strain diffraction measurement through several millimeters of steel (6). More measurements on steel showed penetration depths up to 300 µm using energies up to 70 kev and up to 30 µm using 25 keV (28–31).

Synchrotron radiation is only available in few central facilities around the world. The beam time is limited typically to few weeks around the year. Such an expensive and limited facility that needs a high level of expertise is not a viable option for routine measurements or a large number of samples.

2.1.4  Neutron diffraction

Like x-ray diffraction, neutron diffraction relies on measuring the diffraction planes spacing within a polycrystalline material. There are two neutron diffraction techniques: θ-2θ scans around the sample and the time-of-flight approach. These two methods were developed because of the two forms in which neutron beams are available: continuous beams from a reactor source and pulsed beams from a spallation source (1; 32). It is a nondestructive technique and its greatest advantage is its high penetration ability. Neutrons can penetrate a bulk of aluminum up to 100 mm and can penetrate steel for up to 25 mm. The resolution of this measurement method is around 500 µm. This high penetration allows constructing 3D maps of the stress inside the materials. Unfortunately, neutrons diffraction has a very high cost and is not practical for routine measurements. Beam time is also very limited as there are a limited number of neutron diffraction facilities around the world.

In this case of a spallation neutron source, a pulse of neutrons has a large range of neutron energies. The diffraction angle is typically fixed to $2\theta = 90^\circ$. Since different neutrons with different energies will have different speeds, the energy of each detected neutron will depend on its time of flight. The strain is given by $\Delta \varepsilon = \frac{\Delta t}{t}$. The flight path is usually large (about 100 m) (1). This method in principle is similar to the energy dispersive x-ray diffraction technique.
2.1.5 Curvature and layer removal

Unlike diffraction techniques, this method works on all types of materials. Successful measurements using this method were done on polymeric composites and coatings (33; 34). It is usually used when the sample has a simple geometry. When a layer is removed from a plate the stresses become unbalanced and the plate bends. It is quick and requires only simple calculations to relate the curvature to the stress. By removing successive layers, a stress profile can be constructed. Curvatures can be measured using strain gauges or laser scanning or microscopy and many other methods. However, it is a destructive method and cannot measure the stresses near the surface successfully.

2.1.6 Magnetic and electrical methods

Ferromagnetic materials properties change their shape when subjected to an external magnetic field. On the other hand, if a ferromagnetic material is deformed, its state of magnetization will be affected. This is called the magnetoelastic effect. This basically means that there is an interaction between magnetic and elastic processes/properties (35).

The magnetic methods have the advantage of being nondestructive, cheap, simple and fast. The resolution of such measurements is typically around a millimeter and they penetrate 5 mm to 10 mm in the material, but they suffer from being limited to ferromagnetic materials and the measurement is very sensitive to other properties such as the hardness, the texture and the grain size. They also require calibration against known stress levels (20).

Another method that falls under this category utilizes eddy current. This method is based on inducing eddy currents in the material and then detecting any changes in the conductivity and/or the permeability through changes in a test coil impedance. The penetration depth can be changed by changing the excitation frequency. Penetration depths around 1 mm are typical. Eddy current is a quick and cheap method, but it is not suitable for many materials because of its sensitivity to the plastic deformations, heat treatments and the microstructural changes in the material of interest (1; 36).

2
This phenomenon is called the magnetostriction. It was discovered by James Joule in 1842. The magnetoelastic effect is a result of this phenomenon.
2.1.7 Ultrasonic

This method utilizes the sensitivity of the ultrasound wave velocity to the stress. The resolution of such a measurement is poor, typically around 5 mm, and surface measurements are very sensitive to the surface roughness (37; 38). However, the penetration depth is usually high: more than 100 mm.

2.1.8 Raman/Fluorescence

Raman effect involves the inelastic interaction of light with matter. Analysis of the scattered spectrum reveals vital information about the sample physical state and chemical structure. The fluorescence lines shift linearly with variations in stress (20). It is a nondestructive technique that has a high spatial resolution, around 0.5 µm. It applies to both crystalline and amorphous materials. However, only surface measurements can be done using this technique. It also requires calibration with known stresses and applies only to materials that exhibit Raman effect (39).

2.2 Causes of residual stresses

Residual stresses can be introduced in materials using: mechanical, thermal, chemical processes, or even using a combination of such processes (1–4; 41–43).

2.2.1 Mechanical processes

Generally all cold working mechanical processes introduce residual stresses, including:

- Surface working such as shot-peening, rolling, polishing etc. These processes create a compressive stress on the surface while the less worked deeper region in the sample will create an undesirable tensile stress. It is well known that the compressive stress on the surface increases the reliability of parts like springs, shafts, gears etc. while tensile stresses have the opposite effect.

- Drawing and rolling: These processes can result in either compressive or tensile stresses on the surface depending on the details of the manufacturing process.
Table 2.1 Comparison of some of the most common residual stress measurement methods.

<table>
<thead>
<tr>
<th>Method</th>
<th>Destructive?</th>
<th>Equipment Type</th>
<th>Material Type</th>
<th>Resolution</th>
<th>Penetration</th>
<th>Additional Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hole Drilling</td>
<td>Yes</td>
<td>Portable or lab based</td>
<td>Any</td>
<td>50 µm depth increment</td>
<td>Hole diameter</td>
<td>Quick and portable. Limited resolution.</td>
</tr>
<tr>
<td>X-Ray Diffraction</td>
<td>No</td>
<td>Portable or lab based</td>
<td>Polycrystalline</td>
<td>20 µm depth 1 mm lateral</td>
<td>&lt;5 µm in Ti</td>
<td>Surface preparation is critical.</td>
</tr>
<tr>
<td>Synchrotron</td>
<td>No</td>
<td>Central facility</td>
<td>Polycrystalline</td>
<td>20 µm depth 1 mm lateral</td>
<td>100 mm in Al 3 mm in Steel</td>
<td>Limited access.</td>
</tr>
<tr>
<td>Neutron Diffraction</td>
<td>No</td>
<td>Central facility</td>
<td>Polycrystalline</td>
<td>500 µm</td>
<td>100 mm in Al 25 mm in Steel</td>
<td>Limited access.</td>
</tr>
<tr>
<td>Curvature and Layer Removal</td>
<td>Yes</td>
<td>Portable or lab based</td>
<td>Any</td>
<td>depends on material</td>
<td>N/A</td>
<td>Limited to simple shapes.</td>
</tr>
<tr>
<td>Ultrasonic</td>
<td>No</td>
<td>lab based</td>
<td>Polycrystalline</td>
<td>5 mm</td>
<td>&gt;100 mm</td>
<td>Limited resolution.</td>
</tr>
<tr>
<td>Magnetoelasticity</td>
<td>No</td>
<td>lab based</td>
<td>Ferromagnetic</td>
<td>1 mm</td>
<td>&lt;300 µm</td>
<td>Sensitive to microstructure. Poor resolution.</td>
</tr>
<tr>
<td>Eddy Current</td>
<td>No</td>
<td>lab based</td>
<td>Polycrystalline</td>
<td>10 µm (36)</td>
<td>&lt;1 mm in Ni (36)</td>
<td>Measurement on some materials is not possible because of sensitivity to plastic work.</td>
</tr>
<tr>
<td>Raman Spectroscopy</td>
<td>No</td>
<td>lab based</td>
<td>Ceramics and Polymers</td>
<td>0.5 µm</td>
<td>Surface only</td>
<td>Requires calibration. Limited range of materials.</td>
</tr>
</tbody>
</table>

*a*Information compiled from (20) and (40) and (1) and other sources cited in chapter 1.

*b*Using Cu or Mo kα radiation

c*Destructive if used to create stress depth profiles

*d*Destructive if used to create stress depth profiles
• Grinding and machining: Gentle grinding and good machining practices will generate compressive stress on the surface because of the cold working processes. On the other hand, abusive grinding and machining practices such as heavy cuts and dull wheels will generate thermal effects that will result in tensile stress on the surface (44).

• Assembling: e.g. shrink fitting of a die assembly. The mismatch between the two components will result in a tensile stress in the outer ring and a compressive stress in the insert.

2.2.2 Thermal processes

Residual stresses resulting from thermal processes fall in two categories:

• Stresses resulting from the thermal gradient only: Examples of such processes include quenching, casting and welding.

• Stresses resulting from thermal processes accompanied by phase transformations: Steel hardening heat treatments are important examples of such processes (45).

2.2.3 Chemical processes

Chemical processes are sources of residual stresses just as are mechanical and thermal processes. Examples of such processes include:

• Oxidation and Corrosion: Residual stresses arise here mostly because the resulting oxide, for example, has a larger volume compared to the original material. If the oxide maintains crystallographic coherency with the underlying metal then a compressive stress will be introduced on the surface and the underlying metal will have a tensile residual stress (46).

• Electroplating: This process may result in either compressive or tensile stresses in the underlying material if the electroplating maintains crystallographic coherence with the underlying material.

2.2.4 Combined processes

When some of the processes mentioned above are combined together, predicting the type or level of the introduced residual stress may not be readily interpretable. Abusive grinding is an example of
such processes. Development of grinding cracks is well known. In this case a mechanical effect from the cold working and a thermal effect exist. If the generated heat is excessive then a significant tensile stress will exist on the surface. More example of such processes are discussed in the the references (47; 48).
CHAPTER 3. The Experimental Setup and Results

As mentioned in chapter 2, standard XRD methods can measure the stress/strain-depth profiles in materials that have a crystal structure. However, those measurements are destructive. Our goal while designing this system is to construct such stress/strain-depth profiles nondestructively. In this chapter, the details of the experimental setup and the rationale behind many of the details will be presented.

3.1 Problem statement

To achieve our design goal, especially for metallic samples like Ti, one can either increase the intensity or the energy of the incident beam to achieve the higher penetration. The required high intensities currently can only be achieved using synchrotron sources. Even those high intensities as obtained from synchrotron are not capable of penetrating 100 $\mu$m to 300 $\mu$m in metals at 8 keV, for example. As already mentioned, synchrotrons are not practical for routine cost effective measurements. The inability to use sources with such high intensities means that the only way to achieve the higher penetration is to use a higher energy x-ray source.

Typically, a metal lattice parameter ranges from 2 Å to 5 Å. Using 50 keV to 100 keV energies means that $\theta = 10^\circ$ to $1^\circ$. Usually residual strain in metals is $\leq 0.005$ Å. These facts and the fact that most stress profiles of interest fall within the first few hundreds of microns under the metal surface (e.g shot peened materials) impose some requirements on the detector and the sample positional accuracy and the collimation of the incident and diffracted beams. A spatially broad beam means higher intensity but poorer strain depth profile spatial resolution, while a more tightly collimated beam means higher strain profile resolution at the cost of reduced penetration and intensity. The collimation is also limited by the sample grain size. A finely grained material can be probed using tighter collimation while coarsely grained materials require wider collimation.
Figure 3.1 Lab picture: X-ray source and the incident beam collimators.
Figure 3.2  Lab picture: Diffracted beam collimators and the HPGe detector.
Figure 3.3 Lab picture: The sample holder and its two linear stages and rotary stages.
Figure 3.4  Incident 270 kV p x-ray white beam from a tube with a tungsten target.
3.2 Setup components

3.2.1 Instrumentation

The system consists of the following components:

- High energy Comet MXR-320/23 industrial x-ray tube with a tungsten target.
- Nitrogen-cooled high-purity energy-dispersive germanium detector and Ortec 927 16k USB multi-channel analyzer.
- Parker-Daedal 210RT and 206RT rotary stages for the sample and the detector.
- Incident and diffracted beam collimators made of 6 mm thick tungsten plates.
- Four Parker-Daedal MX80S miniature linear positioners for the collimators.
- Two Parker-Daedal 404XR sample linear positioners.
- Compumotor 6K8 motion indexer and eight Compumotor E-DC drives and stepper motors.
- Data acquisition and control software.

The first main component of this diffraction setup is a typical 320 kVp industrial x-ray tube with a tungsten target in our case. The tube we are using is a Comet MXR-320/23 closed tube. This tube x-rays has the characteristic lines $K_{\alpha 1}$ at 59.3182 keV and $K_{\alpha 2}$ at 57.9817 keV and $K_{\beta 1}$ at 67.2443 keV and $K_{\beta 3}$ at 66.9514 keV (10; 49). Figure 3.4 shows the energy distribution of the tube generated x-rays when the tube is operating at 270 kV. As we can see in this figure, the tungsten $K_{\alpha 1}$ characteristic line has the highest intensity. Operating the x-ray tube on its highest rated power will result in the most intense characteristic lines. The combined high intensity and energy of this line is what makes performing such a nondestructive strain-depth profiles on materials like titanium possible. In fact, the brilliance of this x-ray tube at 59.3 keV is estimated to be about $10^{14}$ photons/sec/0.1%BW/mrad²/mm². This beam brilliance is estimated by collecting the energy dispersive spectrum of the direct beam using a known collimation and counting time after passing through a block of aluminum with a known x-ray attenuation. This intensity is comparable to the intensity of a low-end synchrotron source at
that energy. The exact energy distribution shape of the incident spectrum is very important especially around the chosen characteristic line (more details about this later in this section and sections 3.7 and 3.8). As we already know, the x-ray linear attenuation coefficient $\mu$ is a function of the incident energy. In the case of a tube with a tungsten target, for example, $K_{\alpha 1}$ line intensity is about twice the intensity if $K_{\alpha 2}$ and about four times the intensity of $K_{\beta 1}$ line. See figure 3.4. In the case of an aluminum sample, $\mu_{K_{\alpha 1}} = 0.737 \text{ cm}^{-1}$ and $\mu_{K_{\alpha 2}} = 0.760 \text{ cm}^{-1}$ and $\mu_{K_{\beta 1}} = 0.636 \text{ cm}^{-1}$. The advantage of the higher intensity when using $K_{\alpha 1}$ in this case outweighs the advantage of the lower linear attenuation coefficient of the more energetic characteristic lines. This expected result is shown in figure 3.5 which shows an energy dispersive $\theta-2\theta$ XRD of an aluminum sample. The highest [111] peak intensity is at $\theta = 2.562^\circ$. On the other hand, in the case of a nickel sample, $\mu_{K_{\alpha 1}} = 13.283 \text{ cm}^{-1}$ and $\mu_{K_{\alpha 2}} = 14.122 \text{ cm}^{-1}$ and $\mu_{K_{\beta 1}} = 9.66 \text{ cm}^{-1}$ and $\mu_{K_{\beta 3}} = 9.552 \text{ cm}^{-1}$. In this case $K_{\beta 1}$ line is a better choice than the other characteristic lines, depending on the diffraction angle and the initial diffraction peak intensity on the surface of the alloy under investigation, as the intensity changes exponentially with the penetration distance inside the sample for a given $\mu$.

Another factor that affects the choice of the characteristic line, especially when small angle of diffraction is used, is its natural width. Some x-ray target materials have characteristic lines that are as narrow as few electron volts, while for higher Z materials, $K_{\alpha 1}$ lines might be as wide as 100 eV. The used characteristic line should not be too narrow when a large angle of diffraction, $\theta \geq 5^\circ$, is used. For example, a hypothetical 60 keV characteristic line with $FWHM = 5 \text{ eV}$ can barely be used to measure strains as small as $\pm 0.0005 \text{Å}$ when $\theta \approx 5^\circ$ in an aluminum sample, while a hypothetical 60 keV line with $FWHM = 50 \text{ eV}$ can be used to measure strains that are three times that value at the same angle. These numbers were estimated using the experimental setup modeling program that will be introduced in section 3.7.2. The reasoning behind these estimates is that we want some of the characteristic line to diffract within the divergence of the diffracted beam collimators. We want to have a diffraction peak with reasonable intensity that can be located by counting for only few minutes rather than hours. Other characteristic lines from other target materials can be a better choice for some samples depending on the value of $\mu (E)$ for that line. Higher Z materials can produce $K_{\alpha 1}$ lines with energies close to 100 keV and about twice the natural width of the tungsten $K_{\alpha 1}$. For very small angles of diffraction,
Figure 3.5 Energy and angle dispersive aluminum diffraction pattern. White incident beam as the one shown in figure 3.4. The highest diffracted signal intensity is obtained when the angle \( \theta \) is such that \( K_{\alpha 1} \) meets the diffraction condition for \( d_{111} \).
the width of the characteristic line becomes dominated by the divergence of the diffracted beam. The choice of the characteristic line also dictates the diffraction angle for a given sample. Smaller angles of diffraction mean that wider range of energies will make it to the detector for a given collimation-divergence of the incident and diffracted beams even when the used characteristic line is very narrow. Although the shape of the characteristic line adds an extra level of complication to understanding the data, it is actually an important factor that we will use to our advantage. This width means that different strains will have different intensities at a fixed angle. Unlike most synchrotron sources which do not provide such a high energy very narrow intensity distribution\(^1\), the diffracted intensity can be used as a strain gauge when the incident beam is dominated by a characteristic line. The importance of the characteristic line width will be discussed in more details in section 3.8.

The x-ray tube has two spot size settings. A large 3.6 mm spot size setting and a smaller 1.9 mm spot size setting. Although the small spot setting has a lower maximum power rating, its intensity is distributed in significantly smaller area than the large spot setting, which allows for a significantly higher intensity per unit area compared to the large spot setting. Since we will be working with collimations that are significantly less than both spot settings, the setting with the tighter intensity distribution will serve better. Figures 3.6(a) and 3.6(b) show pin hole images of the tube hot spot in both cases.

Another crucial component in the setup is the x-ray detector. We are using an energy-dispersive nitrogen-cooled Ortec High Purity Germanium detector. Traditional x-ray diffraction setups use an incident monochromatic beam and find the diffraction peak in \(\theta\) space by rotating the detector around the sample. This setup option is shown in figure 3.7. Another diffraction setup option is to use a white incident x-ray beam and find the diffraction peak in energy space using an energy dispersive detector (see figure 3.8). Because of this HPGe energy dispersive detector, this energy dispersive diffraction setup is now a practical option. Figures 3.7 and 3.8 show these two possible diffraction setups.

One advantage for using the energy dispersive detector is the fact that we do not have to use a monochromating crystal to create a monochromatic incident beam as this process will cause a significant loss of intensity for the incident beam. It is also important to note that since we are using an

\(^1\)At the time of writing this thesis, out of the currently available ten synchrotron sources in the United States, only the Advanced Photon Source in Argonne National Lab can provide such a high energy (\(\geq 60\) keV) beam that is as narrow as a tube source characteristic line.
energy-dispersive detector and an incident white beam, we can perform diffraction measurements in energy space as well as in $\theta$ space by discriminating against some energies using the energy dispersive detector. The energy dispersive detector enables us to discriminate against energies that are not necessary for the measurement such as very low energies or very high energies. It can also exclusively collect energies that fall within a defined range of interest such as the data in figures 3.22 and 3.24(a) and the energy dispersive data that will be presented later in section 3.8.2.

The energy-dispersive detector is very helpful in many other situations such as defining the location of the sample surface in a measurement depth profile scan. A material that gives a known energy dispersive diffraction signal at a specific energy can be placed on the sample surface. When the sample is scanned, detecting that known diffraction energy at a specific location along the depth profile indicates that the sample surface is in that location. This way we can define the sample surface location without affecting the energy dispersive signal that will help study the sample itself. Defining the sample surface is important in the strain depth profiles uncertainties analysis as will be shown later in section 3.8.

The detector we are using has a 0.4 keV FWHM resolution at 60 keV. Although this number may
seem high, we should emphasize here that we will be mainly interested in finding the precise location of one peak rather than trying to resolve two peak locations. An energy-dispersive diffraction peak location can be measured within $\pm 0.01 \text{ keV}$ depending on the count rate using this MCA-detector system. We also made sure that the calibration of the MCA-detector is not affected by the count rate. However, the detector calibration is significantly affected by its temperature if it is not left to cool down for about six hours after filling it with nitrogen. We always made sure that the detector was cold enough while collecting all of the data presented in this thesis. The detector is connected to the data acquisition and control computer via Ortec 927 16k USB multichannel analyzer. More information about the detector-MCA system calibration in appendix A.

The incident and diffracted beams are collimated using 6.35 $\text{mm}$ thick tungsten plates. The sides of these plates were polished and have less than 1 $\mu\text{m}$ average surface roughness. The opening of each collimator can be fixed using a foil of known thickness. Typically the opening of the collimators is 135 $\mu\text{m}$ and the distance between the incident beam collimators is around 1.0 $\text{m}$. The same setup applies to the diffracted beam collimators. Each collimator is mounted on Daedal MX80S miniature linear stage that has 1.5 $\mu\text{m}$ bi-directional repeatability and 50 $\text{mm}$ travel distance. These collimation dimensions mean that the divergence of the incident and diffracted beams is about 0.008°. In Bragg’s law, this divergence corresponds to about 0.2 keV at 60 keV. After adding this beam divergence to the detector 0.4 keV $\text{FWHM}$ energy resolution, the total effective $\text{FWHM}$ energy resolution becomes 0.45 keV. It should be emphasized here that in all of the work we are presenting in this thesis, we are mainly interested in finding a specific peak location, not resolving two peaks. This means that our energy measurement resolution is mainly dictated by the intensity of the peak, i.e. the statistics in the curve fitting process. The collimation imposes an upper limit on the spatial resolution of the measurement. The collimation of the incident and diffracted beams defines a volume from where the diffracted signal is collected. When this volume is partially inside the sample, the measurement spatial resolution becomes better (For more details see sections 3.6.1 and 3.8).

The detector and the sample holder are mounted on Daedal 210RT and 212RT precision grade rotary tables. The sample can be moved linearly across the beam and vertically using two Daedal 404XR precision grade linear tables. The rotary tables and linear tables and the collimators are controlled us-
ing Compumotor 6K8 indexer and Compumotor E-DC stepper motors drives and VS23 stepper motors. The rotary tables can rotate in steps as small as 0.0004° while the linear stages can move in steps as small as 1 µm. The motion control system was tested to provide enough torque for moving the detector with its filled Nitrogen dewar without slipping.

Finally, unlike lab based traditional θ-dispersive XRD experiments or synchrotron based EDXRD experiments, the incident beam we are using is mainly one of the tungsten high energy characteristic lines. This beam, although collimated tightly, has a non-negligible divergence. These characteristic lines have Lorentzian shapes with non-negligible energy spread compared to the beam angular divergence. They even have a non-negligible bremsstrahlung radiation. On top of these issues, when the diffraction signal is collected from a region very close to the sample surface, when the volume defined by the intersection of the incident and diffracted beam collimators is not fully inside the sample, the possible range of diffracted energies and diffraction angles becomes different when compared to the case when this probing volume is fully inside the sample. These complications mean that the same energy will correspond to a different lattice parameter at a fixed system angle for different probing volume locations. To gain a better understanding of the collected data, a program that models the experimental setup was developed. This modeling program is an important part of this work. It significantly helps untangle the complications mentioned above and serves as a very rigorous data analysis tool. It reliably helps convert the diffraction measurements to strain measurements while taking the issues mentioned above into account. This program is discussed in more details in section 3.7.

3.2.2 Instrument issues

An important factor that affects the accuracy of the motion control components is the backlash in the gears of the rotary and linear stages. We measured the backlash in θ and 2θ axes to be 0.005° and 5 µm in the linear stages (see figures 3.9(b) and 3.9(a)). An uncertainty of 0.005° in 2θ, for example, will result in uncertainty of 0.003 Å in measuring the lattice constant if 59.32 keV beam is used.

To overcome this problem we always approach the motion destinations from the negative to the positive directions only. To move down from $x_2$ point to $x_1 < x_2$ we always move first back to a position $x_0$ that is less than $x_1$ by at least the amount of the backlash of that axis, then move from $x_0$ to
Figure 3.7 Traditional XRD setup in which a specific energy is chosen from a white spectrum using a monochromator. The incident collimated beam on the sample is a monochromatic beam. The diffraction peak is found by performing \( \theta - 2\theta \) detector scan around the sample.
Figure 3.8 Energy dispersive XRD setup. A white collimated beam hits the sample. The diffraction peak is found in energy space at any angle $\theta$ using energy dispersive detector.
This will eliminate the effect of the backlash in the linear stages and keep the positional accuracy of the rotary stages around ±0.001° or less. While we can completely ignore the positional accuracy of the linear stages without affecting the strain profile measurements, we still need to keep the rotary stages accuracy in our minds during the measurements uncertainties analysis process that will be discussed later in section 3.8. It should be emphasized here that even though the linear stages accuracy and repeatability is 1.6 µm, there are other uncertainty sources in determining the sample position. The sample is typically scanned at steps of 5 µm to 25 µm. Pinpointing the sample and the detector locations is limited by the step size of the subsequent measurements as will be shown later. All of these sources of the measurement uncertainty and many other possible causes of measurement uncertainty will be discussed later in section 3.8 in more details.

The incident and diffracted beam collimators are mounted on linear stages as mentioned in section 3.2.1. Although these miniature linear stages have a 1.5 µm positional accuracy, the actual accuracy of the position of the collimators is affected by many other factors such as the collimator alignment scan step size and the accuracy of defining the center of rotation of the sample and the detector rotary stages. More about this issue will be presented in section 3.8 and appendix B.

Another factor that affects the quality of the data is the background level. Figure 3.10 shows an example of an HOPG\(^2\) [002] diffraction peak with a high background level. To minimize the background, the following measures are taken:

- The source is shielded with lead so that x-rays leave the source only through the collimators.
- The incident and diffracted beams travel through lead-shielded pipes to lower the chance of scattering from other objects in the lab.
- The detector is wrapped with a layer of copper underneath its tungsten casing to prevent natural radiation from the detector tungsten casing, cosmic rays, and the detector casing tungsten fluorescence from reaching the detector Germanium crystal.

After taking these measures, clean data such as the one shown in figure 3.18 can be routinely collected.

\(^2\)HOPG: Highly Ordered Pyrolytic Graphite crystal. Its c axis angular spread is less than 1 degree. HOPG diffracts x-rays with a very high efficiency.
Figure 3.9 Scanning the same intensity distribution starting from two opposite directions results in a shifted intensity vs position distribution.
Figure 3.10 HOPG [002] diffraction peak with a high level of background radiation. Proper shielding to the incident and diffracted beams resulted in a cleaner data as shown in figure 3.18. $2\theta = 3.571^\circ$. 
3.3 Data acquisition and control software

A program was written in Visual C++ to communicate with the various components of the experimental setup. The program can collect the energy dispersive data from the MCA-detector system and at the same time it can control the eight motion axes of the setup:

- The sample $\theta$ axis (210RT rotary stage).
- The sample-detector $2\theta$ axis (212RT rotary stage).
- The sample vertical and horizontal linear positioners (two 404XR linear positioners).
- The four collimators miniature linear positioners (four MX80S linear positioners).

By synchronizing the various components of the setup properly, the program can perform the following tasks:

1. Simple energy dispersive data collection for a given setup and period. The data can be collected, viewed and/or saved for a pre-selected range of energies. Example of such results are shown in figures 3.18 and 3.19. Figure 3.19 demonstrates not only the basic ability of collecting energy dispersive spectrum, it also demonstrates the ability to perform diffraction experiments at very small angles and very high energies.

2. One dimensional scans using any selected axis out of the eight axes: in this case the energy dispersive spectrum is collected while a specific axis is scanning a selected range of positions at fixed intervals. Figure 3.24(a) shows an example of energy dispersive intensity depth profile in an HOPG crystal. By integrating intensity of [002] peak in figure 3.24(a), we get the integrated intensity depth profile of [002] peak as shown in figure 3.24(b).

Another example of such a scan type is shown in figure 3.11 where an energy-dispersive intensity depth profile is shown. [111] and [200] peaks are shown in that figure. The energy-dispersive intensity depth profile of [111] peak is shown in figure 3.12 while [200] energy-dispersive intensity depth profile is shown in figure 3.13. Integrating the energy-dispersive intensity depth profile of [111] peak in figure 3.12 yields the total intensity depth profile of [111] peak shown in figure 3.14.
3. Two dimensional scan using any two selected axes. Although we never used this feature extensively, but it is helpful in the system alignment procedure as shown in appendix B. This scan type can be used to construct two dimensional strain maps. Extending this scan type to perform three dimensional scans should be straightforward.

4. Angle-energy-dispersive XRD $\theta$-2$\theta$ scans around the sample. An example of such a scan is shown in figure 3.15. Angle-dispersive XRD for a given energy can by found by integrating the intensities of a narrow range of energies at different angles as shown in figure 3.16 where an angle-dispersive XRD spectrum of HOPG, when $E = 59.32$ keV, is shown. This figure is obtained by integrating the intensities between the two horizontal lines in figure 3.15. A vertical slice from figure 3.15 will give the energy-dispersive spectrum at a given angular location. For example, integrating the counts between the two vertical lines around $2\theta = 3.571^\circ$ in figure 3.15 will yield the energy-dispersive spectrum shown in figure 3.17.

5. Time scans by observing the total intensity and the energy dispersive spectrum at any selected location as a function of time. This scan type can be helpful, for example, in investigating the stability of the x-ray source and monitoring the detector calibration stability/drift as a function of the count rate.

Before collecting any data, all of the system components must be aligned properly. The detailed steps of the alignment process are discussed in Appendix C. Once the system components are aligned we can start testing the system operation.
Figure 3.11 Al EDXRD intensity depth profile showing [111] and [200] peaks. \(K_{a1}\) line tuned at [111]. \(\theta = 2.562^\circ\). [200] is not intense enough using this color scale. A zoom-in on [200] is shown in figure 3.13.

Figure 3.12 Experimental EDXRD intensity depth profile of unstrained Al. Figure 3.14 shows the integrated intensity depth profile of this data \(\theta = 2.562^\circ\).
Figure 3.13  Al EDXRD intensity depth profile showing [200] peak. $\theta = 2.562^\circ$.

Figure 3.14  Non-peened Al [111] integrated intensity depth profile. $\theta = 2.562^\circ$. The x-axis is the position of the center of the probing volume with respect to the sample surface.
Figure 3.15  \( \theta-2\theta \) scan example: Energy and angle dispersive diffraction pattern of HOPG crystal. The data slice between the two horizontal lines represent an angle dispersive \( \theta-2\theta \) scan data when the incident beam is 59.32 keV. This horizontal slice is shown in figure 3.16. The data slice between the two vertical lines is the energy dispersive spectrum when \( 2\theta = 3.571^\circ \). This vertical slice is shown in figure 3.17.
Figure 3.16  HOPG angle-dispersive XRD pattern. \( E = 59.32 \, keV \).

Figure 3.17  HOPG [002] XRD peak. \( 2\theta = 3.571^\circ \). \( E_{[002]} = 59.32 \, keV \).
Figure 3.18  HOPG [002] XRD peak. $2\theta = 5.126^\circ$. $E_{[002]} = 40\, keV$.

Figure 3.19  HOPG [002] XRD peak. $\theta = 0.424^\circ$. $E_{[002]} = 250\, keV$. 
3.4 EDXRD basics

X-ray diffraction basics are well established. In this section we will provide a quick overview of the energy-dispersive and angle-dispersive diffraction concepts, then we show how our system compares to them.

The basic principle behind x-ray diffraction is Bragg's law:

\[ 2 \, d_{hkl} \sin \theta = n \lambda \]

In a traditional angle-dispersive x-ray diffraction experiment, such as the one shown in figure 3.7, the incident x-ray beam is a monochromatic beam. The detector is rotated around the sample and the diffraction peak is found in \( \theta \) space. In this case, Bragg's law can be rewritten as:

\[ d_{hkl} \sin \theta = constant \]

an example of such a diffraction result is shown in figure 3.16. However, in an ideal energy-dispersive x-ray diffraction (EDXRD) experiment, see figure 3.8, the diffraction angle \( \theta \) is kept constant and the incident beam will have a wide range of energies. The diffraction peak can then be found in the energy space for fixed \( \theta \). Bragg's law can be rewritten as:

\[ d_{hkl} \, E = constant \]

where \( E \) is the center of the diffraction peak in the energy space. This type of XRD experiment can already be done at high energies using a synchrotron source. An example of such an XRD experiment result, collected using our diffraction setup, is shown in figure 3.18.

Both of these diffraction techniques provide means to measure the sample lattice constant. However, in an EDXRD, unlike the traditional angle-dispersive method, the diffraction peak can be obtained without the need to keep accurate track of the sample angular position using a goniometer while collecting the data. A "white beam" hits the sample, then a diffraction pattern is collected using an energy-dispersive detector located at \( 2\theta \). There is no need to keep rotating the sample and the detector while collecting the data which leads to a significantly faster data collection.

Although our system is meant to be an energy-dispersive XRD system, it should be emphasized here that our system is different from an ideal energy dispersive XRD system in many ways:
1. We will be dealing with small diffraction angles most of the time ($\theta < 5^\circ$). The divergence of the incident and diffracted beams is not negligible as illustrated in figure 3.20. In this figure, x-rays that take path 1 will diffract at an angle that is smaller than the nominal angle $\theta$ of the diffraction setup. This will result in a higher diffraction energy compared to x-rays that follow path 2. Path 3 will have the highest possible diffraction angle for a given collimation which will result in lower diffracted energies at the detector site. This energy spread is usually negligible in traditional diffraction setups because of the higher diffraction angles. This divergence is not negligible for small diffraction angles as it will result in a larger energy spread in the diffracted signal.

![Figure 3.20 A diagram illustrating the different diffraction angles for the divergent x-rays enveloped within the collimators.](image)

2. Ideally, in an EDXRD setup, the incident beam has a uniform intensity distribution. The incident beam in our system is one of the tungsten characteristic lines. These lines have a very narrow intensity distribution, but the width of any of those characteristic lines ($\sim 50$ eV) is on the order of the divergence of the incident and diffracted beam in Bragg’s law. A different lattice plane spacing will result not only in a different energy peak location, it will also result in a different intensity. Even the location of that energy peak can be different from what is expected from a simple direct calculation using Bragg’s law.

Throughout all of this thesis we will be dealing with the full Bragg law $d_{hkl} E \sin \theta = \text{constant}$. We will be using a modeling program that will take all of these factors into account. This program is basically an integration of all of the possible incident energies and intensities and diffraction angles. To conduct an EDXRD experiment using our system, we set the diffraction angle $\theta$ to meet the $K_{\alpha 1}$ diffraction condition for the unstrained $d_{hkl}$. This will result in a maximum intensity for the diffracted
signal in the strain-free regions in the sample. The strain profile can then be measured using the changes in the diffracted energy and intensity. For example, the dashed line in figure 3.21 shows the expected location of the energy dispersive diffraction peak when $\theta = 2.562^\circ$ for a uniform incident beam. However, when the incident beam is a Lorentzian characteristic line, the relation between $d$ and the location of the EDXRD peak is expected to look like the solid line in that figure. More details about this figure and the system modeling program are presented in section 3.7.2.

![Figure 3.21](image)

**Figure 3.21** A comparison between the ideally expected $d$ vs $E$ at a fixed angle ($\theta = 2.562^\circ$) when the incident beam is uniform white beam (the dashed line) and when the incident beam is the tungsten $K\alpha_1$ line. More details about how we got this figure in section 3.8.2.

![Figure 3.22](image)

**Figure 3.22** Al angle-dispersive XRD pattern. $E = 59.32$ keV.
Figure 3.23  Energy and angle dispersive aluminum diffraction pattern. White incident beam as the one shown in figure 3.4. The highest diffracted signal intensity is obtained when the angle \( \theta \) is such that \( K_{\alpha 1} \) meets the diffraction condition for \( d_{111} \). The data between the two horizontal lines around \( E = 59.32 \, keV \) is shown in figure 3.22.
Figure 3.24  HOPG crystal intensity depth profile. $2\theta = 3.571^\circ$, $E = 59.32$ keV. More information about how the intensity depth profiles are collected can be found in section 3.6.
3.5 Initial diffraction results

3.5.1 HOPG crystal diffraction results

After building the system, and before proceeding to doing strain-depth profile measurements, some tests need to be performed to make sure that the system can make basic XRD measurements such as getting XRD peak at the expected accurate energy dispersive or angular locations. We also need to check the accuracy of the various positioners and the overall system alignment as well as the detector calibration accuracy. The intensity gain due to tuning a diffraction peak to a characteristic line will also be demonstrated in this section. The simplest test for the system is to get an EDXRD spectrum at a fixed angle from a material with a known lattice constant. HOPG crystals have the advantage of being very efficient in diffracting x-rays. They also have a large mosaic spread that makes them easy to align the crystal in the beam. Figure 3.18 shows HOPG [002] diffraction peak at $E_{[002]} = 41.35$ keV when $2\theta = 5.126^\circ$ as expected. Moving the sample and the detector to $2\theta = 3.571^\circ$ moves the peak to $E_{[002]} = 59.32$ keV as shown in figure 3.17. A 250 keV HOPG [002] peak is shown in figure 3.19. The diffraction peak in figure 3.19, along with the one in figure 3.25, demonstrates the ability of the system to perform very high energy XRD measurements at very small angles as well as higher angles. The energies of those peaks meet what is expected from Bragg’s law.

Figure 3.16 shows HOPG $\theta$-2$\theta$ energy dispersive diffraction pattern. The darkest curved line is [002] diffraction peak for different angles. The light curved line under the darkest line is the detector germanium escape peak that results from [002] HOPG peak (11.103 keV below) \(^3\). The three lighter curved lines above the darkest one are [004] [006] and [008] HOPG diffraction peaks respectively. All diffraction peaks are in agreement with what is expected from Bragg’s law of diffraction.

Looking at the total intensity of a very narrow range of energies at the detector site is similar to using an incident monochromatic beam. Figure 3.16 shows HOPG $\theta$-2$\theta$ diffraction pattern if the incident beam is the tungsten $K_{\alpha1} = 59.318$ keV. The three peaks from left to right are [002] [004] and [006] respectively.

Figure 3.23 shows an energy dispersive aluminum $\theta$-2$\theta$ XRD pattern. This figure demonstrates

\(^3\)Escape peak is a peak produced when the incident photons interact with the detector via the photoelectric effect and produce Ge characteristic photons which escape from the detector without depositing their energy.
the fact that the highest diffracted intensity is obtained when $\theta$ meets the diffraction condition at $K_{\alpha 1}$ for $d_{111}$. It offers the highest diffraction intensity not only compared to the bremsstrahlung, but also compared to any other characteristic line. For this reason, throughout this thesis, we will be working around the tungsten $K_{\alpha 1}$ line while doing all strain measurements. By taking horizontal slices from figure 3.23 at any energy, one can obtain the angle-dispersive diffraction pattern at that energy. For example, figure 3.22 shows aluminum XRD pattern at $E = 59.32\, keV$. The data in this figure is the result of integrating the counts between the two horizontal lines in figure 3.23.

Figure 3.25 shows a 160 $keV$ [731] diffraction peak at $\theta = 3.058^\circ$ from a high purity germanium crystal. This figure and figures 3.17 and 3.18 and 3.19 and 3.16 demonstrate the ability of the system to perform EDXRD measurements over a very wide range of energies and angles for low and high order diffraction peaks.

![Graph](graph.png)

Figure 3.25 Ge crystal high energy EDXRD peak.
3.6 Intensity-depth profile probing volume

The intersection of the collimation of the incident beam with the collimation of the diffracted beam defines the probe volume where the lattice parameter is measured. By moving the sample across the probing volume, a diffraction signal from a selected region inside the sample can be collected. Intensity depth profile, and from this a map of the lattice parameter, can be constructed by moving the sample at regular intervals across the probing volume using the sample horizontal 404XR linear positioner, see figure 3.26.

When we start a scan, the whole probing volume should initially be outside the sample as shown in figure 3.27(b). Ideally, we do not expect any counts to reach the detector at this point. However, there will always be some background radiation levels. By moving the sample a little farther, the tip of the probing volume will start hitting the sample surface as illustrated in figure 3.27(c). By moving the sample farther and farther into the probing volume we will start picking up more and more intensity until the whole volume is fully inside the sample. After this point, further penetration into the sample will result into a lower total intensity because of the longer x-ray path length inside the sample. Figure 3.24(b) shows a depth scan through HOPG crystal. The intensity depth profile behaves as expected. Once the probing volume starts hitting the sample surface, we start getting some intensity. We reach the maximum intensity from HOPG when the whole volume is inside the sample. After that the intensity starts decaying with depth as expected. Note that in figure 3.24(b) the intensity basically does not change with depth after about 150 µm. The reason for that is the small size of the crystal we used. After about 150 µm the incident beam enters the crystal from its side rather than the surface; and the diffracted beam exits the crystal from the other side rather than from the surface. This means that the x-ray path inside the crystal becomes constant after that point, as illustrated in figure 3.28, which means the x-ray attenuation will stay the same.

To get the intensity-depth profile for a given sample, the system diffraction angle is fixed such that it meets Bragg’s diffraction condition for the unstrained lattice parameter value when the incident beam is a characteristic line with a well known energy. Given the collimation and sample surface geometry, the intensity-depth profile will be predictable as already discussed. Any strain in the sample at a given location will result in moving the energy dispersive diffraction peak location off the center.
of the characteristic line. This will result in a drop in the intensity of the diffraction peak. Since the depth and the strain are the only factors that may cause any change in the intensity in a finely grained sample, correcting the intensity depth profile for the depth attenuation will result in an intensity depth profile in which the change in the intensity is solely due to the change in the lattice parameter. This concept is illustrated in figure 3.29. In this figure, unstrained aluminum [111] intensity-depth profile and 9A shot-peened aluminum intensity-depth profile are shown. The calculated intensity-depth profile of ideally unstrained aluminum sample is shown to demonstrate the agreement with the experimental unstrained sample intensity-depth profile. The strain depth profile of the shot-peened sample can then be calculated using this corrected intensity depth profile. More details about converting the intensity-depth profile to a strain-depth profile can be found in section 3.8.1. The strain-depth profile calculated from the 9A shot-peened intensity-depth profile in figure 3.29 is shown in figure 3.30.

We would like to emphasize here that close to the sample surface, when the probing volume is partially inside the sample, the exact shape of the intensity-depth profile of the unstrained sample depends on the shape of the incident beam as the energy and intensity distribution inside the probing volume is not uniform. The shape of the intensity-depth profile gets even more complicated in the case of a strained sample and an incident Lorentzian characteristic line that has a non-negligible bremsstrahlung radiation. Combining these facts with the fact that the collimators divergence is not negligible because of the small diffraction angles, a comprehensive simulation program that models the experimental setup and takes all of these details into account was developed. More details about the simulation program can be found in section 3.7. Later in section 3.7.4 we will present a more detailed study of the intensity and energy distribution inside the probing volume as they affect the strain measurements obtained using the various techniques. We will also discuss the shape of the probing volume and how it affects the spatial resolution of the strain measurements in section 3.6.1.

Figure 3.26 A diagram illustrating the probing volume concept: The volume defined by the incident and diffracted beams collimators.
Figure 3.27 A demonstration for moving the sample into the probing volume. At the beginning of a depth profile scan, the probing volume should be completely outside the sample as shown in figure 3.27(a). No XRD signal is expected at this point. The sample is then moved until the tip of the volume starts hitting the sample as shown in figure 3.27(b). At this point some XRD signal should start reaching the detector. Moving the sample further into the probing volume will increase the XRD signal because the effective probing volume gets larger as shown in figures 3.27(c) and 3.27(d). The exact point at which the intensity starts going down depends on the x-ray linear attenuation coefficient of the sample. Moving the sample further as shown in figure 3.27(e) will result in a lower XRD signal intensity.
Figure 3.28 Probing deeper into the sample will not result into a lower intensity if the incident beam enters from the side of the sample and exits from the other side rather than entering and exiting from the same surface.
Figure 3.29 Al [111] intensity depth profiles of 9A shot-peened aluminum sample and non-peened sample and a simulation of an ideally unstrained aluminum. Correcting intensity depth profiles for the sample attenuation results in corrected intensity depth profiles from which the strain-depth profiles can be measured. The x-axis is the position of the probing volume with respect to the sample surface.
(a) 9A shot-peened Al-6061 intensity depth profiles. $\theta = 2.562^\circ$. The x-axis is the position of the center of the probing volume with respect to the sample surface.

(b) 9A shot-peened Al-6061 strain depth profile. The x-axis is the distance from the sample surface inside the sample (location of center of the effective probing volume).

Figure 3.30 9A shot-peened Al-6061 intensity and strain depth profiles. The strain is calculated using the intensity-change method.
3.6.1 Probing volume dimensions and the system resolution

In all of the data presented in this thesis, we were working with incident and diffracted beams that are collimated to 135 \( \mu m \) over 1 m. Although initially it may seem that this is a very coarse collimation to study stresses that are only 100 \( \mu m \) to 500 \( \mu m \) below the surface, there are many factors that enhance the measurement resolution even when the collimation is not that tight:

- While probing the sample surface, a fraction of the probing volume will be inside the sample. In such a case the resolution will depend on where the probing volume is located. An example of such a situation is shown in figures 3.27(b) and 3.27(c).

- Different angle of diffraction means different geometry for the probing volume. Small angles of diffraction give more weight (i.e. more intensity at the detector site) to the points inside the probing volume that are close to the sample surface because of the shorter x-ray path length compared to x-ray that diffract from the opposing tip of the probing volume which will travel more inside the sample, while higher angles will tend to give the same weight for all points inside the probing volume because of the less pronounced difference of the different path lengths inside the probing volume.

- The resolution when the whole probing volume is inside the sample depends heavily on the x-ray linear attenuation coefficient of that sample. X-rays that diffract from the region of the probing volume that is closer to the sample surface will be attenuated less than the x-rays that diffract from the region that is farther from the surface. For example in the case of an Aluminum sample, almost all of the x-rays that make it to the detector are diffracted from the 100 \( \mu m \) around the center of the probing volume. While in the case of a Titanium sample it will be closer to 80 \( \mu m \) for low order diffraction planes. In a nickel sample, for example, the resolution will be around 20 \( \mu m \) for low order planes using that same 135 \( \mu m \) collimation.

Because of the small diffraction angles we are dealing with in most cases, the probing volume dimension in a direction parallel to the sample surface is several millimeters depending on the exact value of the diffraction angle. An example when \( \theta = 2.562^\circ \) is shown in figure 3.31. The inner solid shape in figure 3.31 represents the ideally expected shape if the divergence of the beam is ignored, while
the outer black lines represent the borders of the probing volume when the divergence of the incident and diffracted beams is taken into account. This spread in the lateral direction is not necessarily a bad thing as the larger volume size means that the sample grain size is not an issue. It also means a stronger signal which leads to a shorter data acquisition time. At the same time, this lateral spread does not cause any resolution loss while measuring stress profiles that depend only on the distance from the surface, such as for shot peened materials. In all of the data shown in this thesis, we always made sure that the sample surface was aligned with respect to the probing volume as shown in figures 3.27, 3.28, and 3.43. This guarantees that the collected data has optimum resolution. That is why the extra spread in the lateral dimension of the probing volume due to the beam divergence, from 3.09 mm to 4.40 mm in figure 3.31, does not have any significant effect on the strain measurement. Later in section 3.7 we present a detailed study of the probing volume and the whole experimental setup with the aid of a comprehensive simulation program that was developed to help understand the various details of the setup including the measurement resolution. Later in that section we show that all points inside the probing volume that are at the same distance from the sample surface will essentially diffract the same energy. This means that the lateral spread of the probing volume due to the divergence of the beam will not only have no effect on the location of the diffraction peak, it will not even broaden that peak. It is the extra spread that is perpendicular to this lateral spread, from 0.13 mm to 0.19 mm in figure 3.31, that will affect the width and/or location of the diffraction peak. See section 3.7.4 for more details.

Figure 3.31  Actual dimensions of the probing volume for 135 \( \mu m \) collimated incident and diffracted beams. \( \theta = 2.562^\circ \).
3.7 Experimental setup modeling

3.7.1 Motivation

As we discussed in section 3.4, in an energy dispersive XRD setup, the diffraction angle \( \theta \) is set at a fixed angle which results in a different energy dispersive diffraction peak locations for the different lattice parameter values \( d_{hkl} \). Since we want to maximize the intensity of the diffraction peak, the diffraction angle \( \theta \) is chosen such that the location of the strongest diffraction peak from the unstrained sample is at the location of the strongest incident characteristic line. For example, to get the maximum possible diffraction signal intensity from an unstrained aluminum sample using an incident white beam generated using a tungsten target, the energy dispersive \([111]\) diffraction peak must be at \( E_{K\alpha_1} = 59.318 \text{ keV} \) for strain-free aluminum \( d_{111} = 2.3379 \text{Å} \). Using Bragg’s law, the diffraction angle must be \( \theta = 2.562^\circ \) in this case. In other words, \([111]\) diffraction peak is tuned to be on top \( K_{\alpha_1} \). At this point we would like to remind that x-ray characteristic lines have a Lorentzian shape. For example the tungsten \( K_{\alpha_1} \) line width is 43.2 eV. If a strain is introduced in the sample, the energy dispersive diffraction peak location will change according to \( d_{hkl} E \sin \theta = \text{constant} \). Consequently, a very small decrease in \( d_{hkl} \) will result in a slightly higher diffraction energy as shown in figure 3.32. This means that the diffraction peak will not be exactly tuned to the characteristic line and only part of this characteristic line will contribute to the intensity of the diffraction peak. More strain in the sample will result in more deviation for the diffraction peak from the characteristic line which will result in a lower intensity as shown in figure 3.35. Although it may seem that this is a disadvantage for using the x-ray characteristic line in an EDXRD experiment, it is actually a huge advantage as it allows for measuring the strain not only using the energy dispersive diffraction signal, but also using the percentage of drop in the diffracted intensity. This method is discussed in more details in section 3.8.1.

In order to understand the result shown in figure 3.32, some very important aspects of our diffraction setup need to be explained. It may seem from a first look that with 135 \( \mu m \) incident and diffracted beams collimation with about 1 m distance between each set of two slits, that the system has a tight collimation to the extent that we can safely ignore the divergence of the incident and diffracted beams.
However, a more detailed study of the problem reveals a different picture:

- A typical residual strain in metals is on the order of 0.001 Å to 0.005 Å.

- The angular difference between the positions of two diffraction peaks for the strains mentioned above, assuming 60 keV incident beam, is $\Delta 2\theta = 0.003^\circ$ to $\Delta 2\theta = 0.008^\circ$.

- The angular divergence of the incident as well as the diffracted beam, assuming 135 µm collimation with the collimators 1 m apart, is 0.015°. This angular distance corresponds to $\Delta d_{hkl} = 0.013$ Å in Bragg’s law assuming 60 keV incident beam. This effect is illustrated in figure 3.33. In this figure, x-rays that take path 1 will diffract at an angle that is smaller than the nominal angle $\theta$ of the diffraction setup. This will result in a higher diffraction energy compared to x-rays that follow path 2. Path 3 will have the highest possible diffraction angle for a given collimation which will result in lower diffracted energies at the detector site. This energy spread is usually negligible in traditional diffraction setups because of the higher diffraction angles. This divergence is not negligible for small diffraction angles as it will result in a larger energy spread in the diffracted signal.

- In a fixed angle diffraction setup, a strain of 0.001 Å to 0.005 Å will shift the diffraction peak in energy space by 25 eV to 125 eV.

- The incident beam is a "white" x-ray beam. However, we rely mainly on the tungsten characteristic lines to provide the intensity needed to conduct the measurements. These characteristic lines are naturally Lorentzian peaks. The tungsten $k_{\alpha_1}$ for example has a $FWHM = 43.2$ eV (9).

- In the case of a fixed-angle white-beam diffraction experiment, if we start with a diffraction angle that is tuned to give $k_{\alpha_1}$ when there is no strain in the sample, any strain in the sample will result in moving the diffraction peak in energy space off the center of $k_{\alpha_1}$ line. This will result in a significant drop of intensity at the detector position and a change in the energy of the diffraction peak. The overall shift in the position of the peak in energy space will actually not directly follow Bragg’s law of diffraction because of the items listed above. If we plot the location of the diffraction peaks in figure 3.34 versus the strain then the relation between the strain and the
Figure 3.32 The ideally expected shape for aluminum [111] energy dispersive diffraction peak for strain-free and strained samples. The simulated incident beam is similar to the one shown in figure 3.4. $\theta = 2.562^\circ$. When $\Delta d_{[111]} = 0.000 \text{Å}$ the diffraction peak is centered on the tungsten characteristic line $K_{\alpha1}$. Introducing some strain in the sample will result in moving the diffraction peak off-center the $K_{\alpha1}$ line which will result in a lower diffraction peak intensity.

Figure 3.33 A diagram illustrating the different diffraction angles for the divergent x-rays enveloped within the collimators.
location of the energy dispersive diffraction peak will become as shown in figure 3.36. This effect is illustrated in figures 3.32 and 3.34 and 3.35 and 3.36. This behavior will be discussed in more details in section 3.8.

![Figure 3.34 Aluminum [111] energy dispersive diffraction peak for various strain values. This simulated data is the same as the data shown in figure 3.32 after applying the detector resolution 0.4 keV to that data.](image)

As a result of the angular divergence of the beam and the non-negligible energy spread of the tungsten characteristic lines and the residual strain, the full Bragg law \( d_{hkl} E \sin \theta = \text{const} \) should be taken into account while trying to understand any experimental result from this setup. Without a detailed simulation of these factors, interpreting the data becomes very difficult.

3.7.2 The simulation program

3.7.2.1 The program target

The experiment simulation program evolved with time as new questions arose while developing the system. At this point the simulation can:
Figure 3.35  The relation between aluminum [111] diffraction peak intensity and the strain value as calculated from figure 3.34.

Figure 3.36  The relation between the location of aluminum [111] energy dispersive diffraction peak and the strain as calculated from figure 3.34. The dotted line represents the ideally expected value from Bragg’s law. The deviation from Bragg’s law value is due to the Lorentzian shape of the incident beam in energy space and the divergence of the incident and diffracted beams. This figure will be discussed in more details in section 3.8.2.
1. Calculate the integrated-intensity depth profile and the energy-dispersive intensity depth profile for a given strain-depth profile.

2. Calculate the spatial distribution of the intensity inside the probing volume for a given strain-depth profile.

3. Calculate the distribution of energies at a given point inside the probing volume.

4. Calculate the strain-depth profile using a given intensity depth profile.

5. Calculate the strain-depth profile using a given energy dispersive intensity depth profile.

### 3.7.2.2 Assumptions and more details about the simulation program

The following assumptions and details went into the program:

1. The sample is a very finely grained sample. No grain structure is assumed.

2. The x-ray source is a uniform source that emits the same intensity at all radial directions. However, the energy-intensity distribution can be any user-given distribution.

3. The sample is an infinite flat surface tilted at an angle that may or may not change depending on the type of diffraction experiment being simulated.

4. The position of the sample \( x_s \) is defined as the distance between the sample surface and the point of intersection of the collimators center lines.

5. The sample linear attenuation coefficient \( \mu \) can either be provided by the user as a constant for all possible energies. Otherwise the program will calculate \( \mu(E) \) for every energy assuming a pure material.

6. The sample lattice constants must be provided as a function of the distance from the sample surface, when needed.

7. A two dimensional world is assumed to minimize the computational power needed to run the program. A three dimensional version of the program is already developed but was not inves-
tigated thoroughly until this point because of the long time it takes to get any result from the program.

8. The detailed geometry of the experimental setup must be provided by the user.

### 3.7.2.3 How the program works

The program starts by assuming that the x-ray source emits x-rays with intensity $I(E_i)$ in all possible directions. A 0.002 keV incident beam resolution is a reasonable choice to preserve the Lorentzian shape of the characteristic lines. The source has a large number of smaller hot spots that are separated by a distance called $\Delta Y$ that typically is on the order of 10 $\mu m$. Each little hot spot will emit x-rays in all directions at equally spaced angular directions $\Delta \theta$. Figure 3.37 shows the assumed source.

Each emitted x-ray will be tested if it will pass the collimators of the incident beam. Once an x-ray passes the incident beam collimators, the point where it hits the sample will be calculated. Then while the x-ray is traveling inside the sample we assume that it can diffract at all points every $\Delta P$ along its path inside the sample as illustrated in figure 3.38. Each possible diffraction event will change the direction of the x-ray by an angle $\theta_{E,x}$ that is calculated using Bragg’s law. $\theta_{E,x}$ will be calculated using the user provided strain profile $d_{hkl}(x)$ where $x$ is the distance between the point of the diffraction event and the sample surface (not to be confused with $x_s$ which defines the probing volume location with respect to the sample surface). After that the diffracted x-ray will be tested whether it will pass the diffracted beam collimators. If it does, the total distance it traveled inside the sample will be calculated. The
intensity of that diffracted x-ray will then be calculated using a calculated linear attenuation coefficient \( \mu(E) \) that depends on the sample material and the energy of the incident x-ray or using a user provided \( \mu \).

The same process will then be repeated for all possible incident x-rays \((\theta_i, Y_i, E_i)\) and all possible diffracted x-rays and all possible penetrations and incident energies from the source. Any diffracted intensity will be recorded as a function of the sample position and the diffracted energy and the sample coordinates and the diffraction point inside the probing volume. Finally, the energy dispersive data is convoluted with the detector function. We can see now that the simulation is essentially a large integration over all possible emitted x-ray locations on the source hot spot and all possible directions within the collimation for all possible diffraction points inside the sample in all possible directions for all incident energies. The simulation parameters \( \Delta Y, \Delta \theta \) and \( \Delta P \) are essentially integration variables and must be as small as possible for best results. However, very small values will lead to a long computation time. To simulate energy dispersive diffraction experiments with fixed \( \theta < 5^\circ \) and beam collimation around 100 \( \mu m \), reasonable values for \( \Delta Y \) range from 5 \( \mu m \) to 20 \( \mu m \) and \( \Delta \theta \) ranges from \( 50 \times 10^{-6} \) radians to \( 500 \times 10^{-6} \) radians. \( \Delta P \) typically ranges from 20 \( \mu m \) to 200 \( \mu m \). Using such values mean that the computation time is less than ten seconds per point in a depth profile or \( \theta-2\theta \) scan. Angle-dispersive \( \theta-2\theta \) XRD experiments are typically modeled in increments of 0.0005\(^\circ\) while depth scans are typically modeled in increments of 1 \( \mu m \) to 25 \( \mu m \).
3.7.3 Experimental and simulation results

Aluminum was one of the most frequently used materials during this research. This choice is logical especially during the initial stages of this research. Aluminum has a relatively low attenuation compared to Titanium and Nickel which makes it easy to align. It is also an isotropic material which made the experimental results easier to understand especially during the initial stages of this research. Figure 3.39 shows the expected intensity depth profile for aluminum when $\theta = 2.562^\circ$. The incident beam, for this simulated data as well as all of the simulated data in this thesis, is similar to the actual white incident beam shown in figure 3.4. The collimation of the incident and diffracted beams is assumed to be 135$\mu m$ and the distance between each set of collimators is 1 $m$. The same thing applies to the diffracted beam collimators. These details apply to all of the simulated data in this thesis. The actual aluminum experimental data for $\theta = 2.562^\circ$ is plotted on top of the simulated data in the same figure 3.39. Figure 3.42 shows the simulated energy dispersive data for the same intensity depth profile shown in figure 3.39. This simulated profile in figure 3.42 is in agreement with the experimental energy dispersive depth profile shown in figure 3.41.

![Comparison between the ideally expected and experimental intensity depth profiles of a strain free Al sample using [111] XRD peak. The solid line is an actual experimental intensity depth profile for a strain-free Al sample using [111] peak. The EDXRD intensity depth profile for the experimental data is shown in figure 3.41 while the simulated EDXRD intensity depth profile is shown in figure 3.42. The x-axis is the position of the center of the probing volume with respect to the sample surface.](image-url)
Figure 3.40  Al [111] EDXRD peak location for a strain-free sample. The solid line represents the expected data using the simulator. The dotted curve is the actual experimental data. The x-axis is the position of the center of the probing volume with respect to the sample surface.

3.7.3.1 Depth profile resolution test

So far the system and its simulator seem to be in a good agreement. The experimental HOPG crystal data shown in figures 3.24(a) and 3.24(b) and 3.16 as well as the experimental unstrained aluminum data shown in figures 3.39 and 3.41 behaved as expected. However, in order to demonstrate the measurement resolution, one more crucial test was done. In this test the sample is constituted of three layers of Nickel. Each layer is 15 \( \mu m \) thick and is separated from the next layer by a 200 \( \mu m \) thick tape layer. The sample structure is illustrated in figure 3.43. The experimental intensity depth profile of this sample is shown in figure 3.44(b). This data is in agreement with the result predicted by the system simulator shown in figure 3.44(a). The dotted curves in figures 3.44(b) and 3.44(a) are the experimental and simulated data, respectively, after removing the effect of the attenuation. The data in figure 3.44(b) not only shows the resolution of the system, it also proves that diffraction peaks can be obtained from metals from the surface as well as from few hundreds of micrometers underneath the surface of the metal by just moving the sample across the beam.
Figure 3.41 Unstrained Al experimental EDXRD intensity depth profile. This data integrated intensity depth profile is in figure 3.39. \( \theta = 2.562^\circ \).

Figure 3.42 Simulated Al EDXRD [111] intensity depth profile. The total intensity depth profile is in figure 3.39.
3.7.3.2 Strained samples simulation results and analysis

We showed previously in figure 3.32 how the strain affects the intensity when the diffraction angle is fixed such that it gives the maximum intensity from the characteristic line for non-strained sample. For example if we probe an aluminum sample using [111] diffraction peak when \( \theta = 2.562^\circ \), we expect the intensity versus strain relation to be as shown in figure 3.35. A simple theoretical strain model to start with is a step strain function like the one shown in figure 3.45(a). One important difference between the intensity depth profiles of the positive and the negative strain values is that the negative strain values give higher intensity than the non-strained material while the positive strain close to the surface will give lower intensity compared to the non strained material as shown in figure 3.45(b). Figure 3.47 shows an actual experimental intensity depth profile for a shot peened aluminum sample. The change in the intensity compared to the ideal strain-free data suggests that the strain in the peened region has a positive value. Later in section 3.8 we will present more verifications for this finding using \( \theta-2\theta \) scans traditional technique and using the energy dispersive measurements. We explain why the intensity is different for positive and negative strain signs even when they have the same value in section 3.8.1. Another important thing is the difference between the energy dispersive intensity depth profiles of both strain values. See figure 3.46. We will show later in section 3.8 how the total intensity and the energy dispersive intensity depth profiles will be used to calculate the strain.

Figure 3.43 An illustration for a depth scan in a specially prepared sample. The sample consists of three nickel layers separated by 200 \( \mu \)m tape layers. The intensity depth profile as expected by the simulator is in figure 3.44(a). Figure 3.44(b) shows the scan result.
Figure 3.44 (a) Simulated and (b) experimental intensity depth profiles for [111] XRD from three 15 µm thick Ni layers separated by 200 µm thick tape layers. The dotted curves are the corresponding intensity depth profiles after correcting them for attenuation. $2\theta = 5.889^\circ$. $E = W_{K\alpha}$. 

(a) Simulated result.

(b) Experimental result.
Now that we emphasized the difference between the intensity depth profiles of strain-free materials and materials with either positive or negative strain values, we would like to discuss the differences in the energy dispersive data between the strained and the strain-free materials. In a strain free material depth scan such as the one shown in figure 3.41, a vertical slice in the data will give the energy dispersive data at a specific probing volume location. In a strain free material we expect the position of the diffraction peak in energy space to be the same at all depths when the whole probing volume is inside the sample. However, the story is different when only part of the probing volume is inside the sample. Initially when the tip of the probing volume starts getting into the sample, the average diffraction angle will be slightly higher than the average diffraction angle when the whole volume is inside the sample due to the incident beam divergence. This means that the energy dispersive diffraction peak location will be at a lower energy compared to the strain free material when the whole volume is inside the sample. By fitting each vertical slice in the data shown in figure 3.41 at each location with a gaussian peak, we get the diffraction peak location in the energy space versus depth. The result for such a process is shown in figure 3.40 for both the simulated energy dispersive intensity depth profile and the experimental one shown in figure 3.41 and figure 3.42. The agreement between the energy versus depth for the experimental and the simulated data is another demonstration for the validity of the simulator and our understanding of the exact details of the data that was presented in this section and the data that will be presented later in sections 3.8.1.3 and 3.8.2 and 3.8.3.2.
(a) Theoretical ±0.003Å strain function 250µm deep for Al $d_{111}(x)$.

(b) Intensity depth profiles for the strain profiles in figure 3.45(a). The x-axis is the position of the center of the probing volume with respect to the sample surface.

Figure 3.45 Intensity depth profiles expected for theoretical $d_{111}(x)$ strain profiles. The corresponding energy-dispersive intensity depth profiles are shown in figure 3.46.
Figure 3.46  Expected energy-dispersive intensity depth profiles, from our model, for the strain profiles in figure 3.45. The integrated intensity depth profiles are shown in figure 3.45(b)
Figure 3.47 Experimental depth profile from a 4A shot-peened aluminum sample using [111] diffraction peak and incident white beam as shown in figure 3.4. $\theta = 2.562^\circ$. The dotted line in figure 3.47(a) is the ideal depth profile for a strain-free sample. The x-axis is the position of the center of the probing volume with respect to the sample surface.
3.7.4 Energy and intensity distribution inside the probing volume

Although the strain measurements discussed briefly on pages 43 and 54 and 59 and later in section 3.8 calculate the average strain inside the probing volume using the total peak intensity or the average peak energy, understanding the energy and intensity distribution inside the probing volume is of great importance as it helps understand the intensity depth profiles in many cases such as when the probe is partially inside the sample or when there is a significant strain gradient inside the probing volume. Studying the energy and intensity distribution inside the probing volume offer a new area where the validity of the simulation program can be checked and open the door for improving the strain calculation methods that we already discussed briefly and will discuss later in more details. These distributions, as shown in section 3.6.1, help determine the actual measurement resolution. The easiest and most obvious case where we can start, is to investigate those distributions in an unstrained sample. Later we will discuss how introducing a strain will alter those distributions.

We already discussed why the unstrained sample intensity depth profile shown in figure 3.39 have this shape. This is mainly due to the shape of the probing volume as shown in figure 3.27. What we did not mentioned earlier is that the distribution of intensities that contribute to the diffraction signal is not spatially uniform inside the probing volume. Figure 3.48 shows such a distribution at various probe locations. When the tip of the probe starts hitting the sample, see figure 3.48(a), the diffracted x-rays will have the highest possible diffraction angle for a given collimation and beam divergence. At this point the diffracted energy will be the lowest possible energy allowed by the collimation. In an Al [111] XRD, and when \( \theta = 2.562^\circ \), this diffracted energy is part of the bremsstrahlung radiation. The characteristic line will not contribute to the intensity at that point. the low diffracted signal intensity is due to the smaller portion of the sample being probed as well as the intensity of the incident energy allowed by the collimation. Probing deeper inside the sample will result in a larger effective probing volume and wider range of possible diffraction angles. This wider range means that a larger portion of the incident characteristic line can satisfy Bragg’s diffraction condition for the given allowed divergence. This will result in a higher diffracted intensity. However, since the tip of the probe is now deeper inside the sample, the intensity contributing to the diffraction signal from that part of the sample becomes less while the total intensity will be higher. The point where the diffracted signal is maximized depends on
Figure 3.48 Unstrained Aluminum [111] XRD calculated total intensity spatial distribution inside the probing volume for various sample locations. The intensity color scale is shown in figure 3.51(e). $\theta = 2.562^\circ$. 
the x-ray linear attenuation coefficient of the sample material. For example, in the case of aluminum
the maximum intensity that can be achieved is when the probing volume is about 20 µm short of being
fully inside the sample, see figure 3.48(e). The intensity gained by probing deeper, which essentially
means larger effective probing volume, will not compensate for the intensity lost due to the attenuation
resulting from the longer x-ray path inside the sample, see figure 3.48(f). On the other hand, in a tita-
nium sample, using the same collimation and [101] peak, the maximum intensity is achieved when the
probe is 70 µm short of being fully inside the sample.

The energy distributions at selected points inside the probing volume in a strain-free sample are

![Energy distributions at selected points inside the probing volume](image)

Figure 3.49 strain-free Al sample energy distribution at points along a line perpendicular to the sample surface inside the probing volume.

shown in figures 3.49 and 3.50. Figure 3.49 shows the energy distributions along a line extending from
the deepest point inside the sample to the closest point to the surface. The shown coordinates in this
figure are \((x, y)\) locations with respect to the center of the probing volume in micrometers. The deepest
point along this line inside the sample has the largest possible diffraction angle which, as expected, will
result in lower diffracted energies. However, those lower energies will have low intensities because
they will mainly be coming from the bremsstrahlung rather than from \(K_{\alpha1}\). Moving closer to the center
of the probe will increase the intensity as well as the energy of the diffracted x-rays from those points.
Figure 3.50 shows that all points with the same vertical distance from the surface will approximately
diffract the same energy. This is due to the fact that, for all of those points along a horizontal line inside the probe, the diffraction angle will not significantly differ from one point to the other.

Introducing a strain in the sample will change the intensity distribution inside the probing volume. A

![Graph showing energy distribution](image)

**Figure 3.50** strain-free Al sample energy distribution at points along a line parallel to the sample surface inside the probing volume.

positive $\Delta d$, when the system angle is set such that [111] peak is tuned to be at $K_{\alpha 1}$ for the unstrained sample, means that a lower range of energies will make it to the detector compared to the unstrained sample. $K_{\alpha 1}$ will not mainly diffract from the center of the probing volume. It will diffract from the portion that is closer to the sample surface. On the other hand, negative $\Delta d$ means $K_{\alpha 1}$ will diffract from deeper region inside the probing volume. This is illustrated in figure 3.51 in the case of Al [111] and $\theta = 2.562^\circ$. These intensity distributions for various $\Delta d$ values, see figure 3.51, explain the intensity difference close to the surface in the intensity depth profile between the positive and negative $\Delta d$ values. In fact, the strained sample intensity from the surface region, compared to the unstrained one, is a reliable way of telling the strain sign. The energy distributions at selected points inside the probing volume when $\Delta d = +0.003\text{Å}$, see figure 3.51(a), are shown in figures 3.52 and 3.53. The corresponding energy distributions when $\Delta d = -0.003\text{Å}$, see figure 3.51(b), are shown in figures 3.54 and 3.55. More energy distributions when $\Delta d = \pm 0.005\text{Å}$ are shown in figures 3.56, 3.57, 3.58 and 3.59.
Figure 3.51 Al [111] calculated total intensity spatial distribution inside the probing volume for various strain values. $\theta = 2.562^\circ$. 
Figure 3.52 Al sample with $\Delta d = +0.003$ energy distribution at points along a line perpendicular to the sample surface inside the probing volume.

Figure 3.53 Al sample with $\Delta d = +0.003$ energy distribution at points along a line parallel to the sample surface inside the probing volume.
Figure 3.54 Al sample with $\Delta d = -0.003$ energy distribution at points along a line perpendicular to the sample surface inside the probing volume.

Figure 3.55 Al sample with $\Delta d = -0.003$ energy distribution at points along a line parallel to the sample surface inside the probing volume.
Figure 3.56  Al sample with $\Delta d = +0.005$ energy distribution at points along a line perpendicular to the sample surface inside the probing volume.

Figure 3.57  Al sample with $\Delta d = +0.005$ energy distribution at points along a line parallel to the sample surface inside the probing volume.
Figure 3.58 Al sample with $\Delta d = -0.005$ energy distribution at points along a line perpendicular to the sample surface inside the probing volume.

Figure 3.59 Al sample with $\Delta d = -0.005$ energy distribution at points along a line parallel to the sample surface inside the probing volume.
3.8 Strain calculation methods

Using our experimental setup, the strain can be measured using three different techniques. In this section, the detailed procedure for each method will be presented. A comparison between the three different methods will be presented later in section 3.8.4 and we will show that, within the most probable ranges of uncertainties, the three different methods result in the same strain profile for the same sample.

3.8.1 Method 1: using the total intensity depth profiles

This is a new technique that we are proposing. It is also the fastest one as it does not require the higher statistics needed for the other two methods because, unlike the energy or theta dispersive methods, no curve-fitting is needed. The main reason why we will present the other two methods in sections 3.8.2 and 3.8.3 is to demonstrate the consistency and validity of the various measurements obtained using this unique diffraction setup. This method, like the rest of the methods that will be discussed, depends heavily on the simulation program. The key feature enabling this method is the requirement of a characteristic line from a tube source, the tungsten $K_{α1}$ in our case, in the incident beam.

The goal here is to construct a relation between a normalized intensity value and the strain. The idea comes from the fact that for a given incident energy with a very narrow, well known, energy distribution, namely the $K_{α1}$ energy, and for a diffraction angle fixed at the angle $θ_{K_{α1},d_0}$ that meets Bragg’s diffraction condition for the unstrained lattice planes spacing $d_0$ at the energy $E_{K_{α1}}$, then any small change in the lattice parameter $Δd$ will result in moving the diffracted energy $E_{K_{α1}}$ off the center of the diffracted beam collimation in $θ$ space. This will result in a drop in the intensity of the diffracted beam. Further increase in $Δd$ will result in further drop in the intensity of the diffracted beam by moving the diffracted beam further off the center of the collimation. $Δd$ can be measured until the point where the whole incident very narrow energy distribution cannot make it to the detector because it cannot meet Bragg’s diffraction condition for the sample $Δd$ at the fixed angle $θ_{K_{α1},d_0}$ within the divergence of the beam collimation. A relation between the intensity and $Δd$ can then be constructed for a given probing volume location inside the sample.
The maximum possible diffracted intensity, when the whole probing volume is inside the sample, is achieved when the sample is strain free. Close to the sample surface, where the probing volume is partially inside the sample, there are a number of factors that influence the diffraction intensity, namely, the volume and shape of the diffraction zone, the symmetry of the collimation and the depth of the diffraction volume in the sample. Although these factors are important, the volume of the diffraction zone is the most important one when probing regions very close to the sample surface. This complexity in the relation between the intensity and $\Delta d$ and the probing volume location suggests using the setup modeling program. In this method, a knowledge of the sample x-ray linear attenuation coefficient is necessary. It is also very important that the sample surface is located as accurately as possible.

The simulation program takes the exact dimensions of the experimental setup and the divergence of the incident and diffracted beam and the sample location into account. It start by assuming a uniform source radiating uniformly in all directions. Those x-rays that make it through the incident beam collimation form the incident beam that will hit the sample surface. This incident beam is a divergent white beam. The sample is assumed to be oriented such that the angle between the sample surface and the line that goes through the center of the incident beam collimators is the same as the angle between the sample surface and the line that goes through the center of the diffracted beam collimators. This angle is the system nominal diffraction angle. This nominal diffraction angle should be chosen such that the unstrained lattice parameter meets Bragg’s diffraction condition for the center of $K_{a1}$ line at that angle. The diffracted beam, like the incident beam, has a divergence that is defined by the openings of the collimators and the location of each collimator. The only relevant x-rays are the ones that diffract within the probing volume and make it through the diffracted beam collimators to the detector. Those x-rays will have different energies and will diffract at different diffraction angles such that each one of them meets Bragg’s diffraction condition for the lattice parameter at the point where it was diffracted. The sample surface location not only controls each individual simulated x-ray path length inside the sample, it also determines the effective probing volume. The effective probing volume is the portion of the probing volume defined by the collimation that actually has a sample material inside it. When the probing volume is partially inside the sample, the detected intensity will be low because the system is probing less material, i.e. the effective probing volume is very small. The location of the energy-
dispersive XRD peak will also be affected by the sample surface location because the possible range of diffraction angles depends on the dimensions of the effective probing volume. When the tip of the probing volume is barely hitting the sample surface, the allowed range of diffraction angles will be very small. On the other hand, when the probing volume is fully inside the sample, the possible range of diffraction angles will be defined by the collimators geometry. More information about the simulation program can be found in section 3.7.2.

The first step in the process of converting intensity-depth profile into strain-depth profile is to generate a set of simulated intensity depth profiles using the setup modeling program for a large number of constant strain depth profiles in the sample under investigation. For example the planes spacing for aluminum [111] is 2.3379 Å. A ±0.01 Å strain range in steps of 0.0002 Å, for example, is sufficient. A subset of such intensity depth profiles for aluminum is shown in figure 3.60. Note that a positive $\Delta d$ close to the surface will result in a lower intensity compared to the unstrained sample, while a negative $\Delta d$ value will result in a higher intensity in the region close to the surface. This is due to the different energy distribution inside the probing volume in these two cases. More details about this issue can be found in section 3.7.4.

The next step is to calculate the ratio between each one of those intensity depth profiles and the ideal strain-free intensity depth profile. The result of such a process for an aluminum sample is shown in figure 3.61. By combining the intensity ratios depth profiles, a two dimensional plot that relates the intensity ratio versus depth and strain can be constructed. The result of such a process is shown in figure 3.62. In other words: an intensity ratio depth profile for a given $\Delta d$ value such as the ones shown in figure 3.61 represent a horizontal slice from figure 3.62 at that $\Delta d$ value.

Once a plot like the one shown in figure 3.62 is constructed for a given sample and system geometry, we can take a vertical slice from that plot at any given depth as shown in figure 3.63. The possible strain values can then be obtained for a given depth and intensity ratio values. See figure 3.64. The correct strain value can then be chosen either with the help of the energy dispersive data or by choosing the physically acceptable value. More details about the calculation procedure is in the next section.
Figure 3.60 Calculated intensity depth profiles of various positive and negative strain values for Al [111] diffraction peak and incident $K_{\alpha 1}$. The $x$-axis is the position of the center of the probing volume with respect to the sample surface.
Figure 3.61 The calculated ratios between Al intensity depth profiles shown in figure 3.60 and a strain-free Al intensity depth profile.
Figure 3.62 Two dimensional plot that relates the strain and the intensity ratios at various depths. This plot is constructed using data set similar to the one shown in figure 3.61. The x-axis is the position of the center of the probing volume with respect to the sample surface.
Figure 3.63 Vertical slices when taken from figure 3.62 represent the relation between the intensity ratio with respect to the ideal strain-free profile and the strain at a given depth. This plot can be considered as a lookup table that gives the strain for a given intensity ratio at a specific depth.
Figure 3.64 A vertical slice from figure 3.62 at $x = -20 \, \mu m$. The intensity versus strain relation at any depth will give two possible strain values for a given intensity-ratio value.

Figure 3.65 Shot peening a sample surface.
3.8.1.1 Example

The best way to explain this method is to show an actual example. We will show here an example for calculating the strain profile for 6A shot-peened aluminum sample. It should be emphasized here that the system modeling program must use the exact same parameters (collimation, dimensions, sample location ... etc.) as the actual experimental setup in order to be able to get the correct results.

Figure 3.66 shows the integrated intensity depth profile of [111] peak from such a sample. The corresponding energy dispersive intensity depth profile is shown in figure 3.67(b). The first step is to normalize the integrated intensity depth profile. This process consists of two steps: the first one is to locate the sample surface. The position coordinate in figure 3.66 must be shifted such that the first diffraction data point, when the tip of the probing volume starts hitting the sample surface, matches that of the ideally unstrained sample. This process must be performed with the best possible accuracy as we will show later that this is a significant uncertainty source in the calculated strain-depth profile.

Using a 135 \( \mu m \) collimation and a very small angle of diffraction (\( \theta = 2.562^\circ \) in this case), the first data point that contains actual diffraction data is when the center of the probing volume is at \( x = -70 \mu m \) with respect to the sample surface. A quick way to shift the data is to assume that the first data point, with a significant intensity above the background or noise level, has the coordinate \( x = -70 \mu m \) and the rest of the data points should be shifted accordingly. The uncertainty in locating the sample surface in this case equals the depth scan step size. Typically the scan step size ranges from 5 \( \mu m \) to 25 \( \mu m \).

This method is sufficient when the sample under investigation gives a reasonably good diffraction signal as it will be easy to tell if a data point is above the background radiation level. When the sample diffraction signal is not that strong, locating the sample surface may become difficult. The intensity when a small percentage of the probing volume is inside the sample may not be high enough to differentiate the actual diffraction signal from the background. In such a case, a small HOPG crystal that is positioned on the sample surface will help locate the surface more accurately. HOPG crystals are very efficient in diffracting x-rays and the diffraction signal from such a crystal should be very strong even when counting for a short period of time (less than one second). The HOPG crystal will give a diffraction signal with different energy from the sample diffraction signal energy, so, with the help of the energy dispersive detector, it should not interfere with the sample diffraction signal. The point we
stop getting any HOPG signal in the depth profile is when the location of the center of the probing volume is \( x = +70 \, \mu m \). At this point the probing volume is fully inside the sample and the rest of the data should be shifted accordingly.

The next step is to normalize the intensity-depth profile of the ideally unstrained sample. This profile must be normalized such that the ideal normalized intensity matches that of the experimental intensity-depth profile deep inside the sample where the sample is supposed to be strain free. The result of the normalization procedure for a 6A shot-peened aluminum sample is shown in figure 3.68.

Once the ideal and the experimental intensity depth profiles are normalized, the ratio of the experimental depth profile to the ideal unstrained profile should be calculated. The result of such a process is shown in figure 3.69. At this point we can use the lookup table generated for the sample under investigation and the experimental setup geometry. The process of generating such a table was described in pages 82 through 84. Each point in the intensity ratio depth profile in figure 3.69 can then be used to look up a strain value from figure 3.62. This can be done by taking a vertical slice from figure 3.62 at the location of each point in figure 3.69. Examples of such slices at different locations can be found in figure 3.63.

Each intensity ratio value gives two possible strain values at each depth as shown in figure 3.64. The two results for the 6A shot-peened aluminum are shown in figure 3.70. One way to determine which \( \Delta d \) value is the correct one is to investigate the energy dispersive data. Using curve fitting on vertical slices from figure 3.67(b), we can obtain the diffraction peak location depth profile. Figure 3.73 shows the result of such a process for 6A shot-peened aluminum. It is obvious from figure 3.73, using Bragg’s law, that the correct \( \Delta d \) value in this example is the positive \( \Delta d \) value. The choice becomes difficult or even irrelevant for points beyond 350 \( \mu m \) as each possible \( \Delta d \) value is within the uncertainty range of the other value. We assumed from the first place, when the data was normalized, that the sample is strain free in that region. In section 3.8.3 we will show another way of verifying \( \Delta d \) sign using \( \theta -2 \theta \) angle-dispersive XRD. Another point that we should consider in picking the right \( \Delta d \) value is that the strain depth profile should be continuous. Obviously erratic jumps from the possible positive \( \Delta d \) to the negative one is not allowed. Next, we need to convert the strain depth profile from the probing volume position coordinate system (see figure 3.71) to the sample system of coordinates
where \( x = 0 \) at the sample surface (see figure 3.72).

It should be emphasized here that in the shot-peening process, the sample surface bombardment will result in plastic deformation to the surface. The deformation will tend to thin out the bombarded surface layer and stretch it sideways with respect to the bombardment direction. However, this will not happen unless the sample is very thin. In a thick sample, this process will result in generating an internal force that will push the sample surface as shown in figure 3.65. This will result in a larger \( \Delta d_{hkl} \) value for the crystal structure layers that are parallel to the surface and smaller \( \Delta d_{hkl} \) value for the lattice planes that are perpendicular to the sample surface. This explanation is consistent with our observations throughout this thesis.

![Figure 3.66 Intensity depth profiles for 6A shot-peened [111] aluminum at \( \theta = 2.562^\circ \) and \( \theta = 2.552^\circ \).](image)

\( E = W_{K_{\alpha 1}}. \)
Figure 3.67 Energy dispersive intensity depth profiles for 6A shot-peened [111] aluminum at $\theta = 2.562^\circ$ and $\theta = 2.552^\circ$.
Figure 3.68  Normalized intensity depth profile of a 6A shot-peened Al sample.

Figure 3.69  Normalized experimental intensity to ideal intensity ratio depth profile of a 6A shot-peened Al sample. The x-axis is the position of the center of the probing volume with respect to the sample surface.
Figure 3.70 Tracing the normalized intensity in figure 3.69 on top of the lookup table in figure 3.62 will result in two possible strain-depth profiles.

Figure 3.71 Strain depth profile of 6A shot-peened Al. The values on the x-axis represent the location of the center of the probing volume with respect to the sample surface.
Figure 3.72 Strain depth profile of 6A shot-peened Al after translating the probing volume location to an *averaged depth* inside the sample with estimated uncertainties. The averaged depth is the location of the *effective* probing volume. See page 50 for more details.

Figure 3.73 Position of aluminum [111] energy-dispersive diffraction peak versus depth for a 6A shot-peened aluminum sample.
3.8.1.2 Error analysis

There are several sources of measurement uncertainty for this technique. Many of them are shared among the three methods. They are listed here starting from the most significant to the least significant one:

- The beam collimators misalignment. A $\pm 10 \ \mu m$ uncertainty in positioning the collimators across the beam will result in about $\pm 0.002 \ \AA$ in $\Delta d_{hkl}$ measurement within the first 100 $\mu m$ from the surface and about $\pm 0.001 \ \AA$ for deeper measurements. This amount of uncertainty may not be acceptable in many situations. However, a more careful alignment with a $\pm 2 \ \mu m$ uncertainty should be possible with the current setup as the positional accuracy of all the collimator holders and the sample linear positioners is less than or equal 2 $\mu m$ and the collimator surface roughness is significantly less than 1 $\mu m$. Developing such a more accurate alignment procedure should be part of any future work.

- Pin-pointing the sample surface location with respect to the probing volume. The accuracy of locating the sample surface depends mostly on the depth profile step size, even when the HOPG crystal technique which we discussed earlier on page 90 is used. Identifying the first point where the probing volume starts hitting the sample in the intensity depth profile is an easy task, when the diffraction signal is well above the background, as shown in figure 3.74. The HOPG crystal technique is more helpful only when the count rate is low. The depth profile step size typically ranges from 5 $\mu m$ to 25 $\mu m$. Other factors that may affect the accuracy of locating the sample surface include the count rate and the background level. In most of the samples we studied so far, the scan step size is the dominant factor. A $\pm 10 \ \mu m$ uncertainty in locating the sample surface will typically result in a $\Delta d_{hkl}$ uncertainty of $\pm 0.001 \ \AA$ or less in the measurements that fall within the first 100 $\mu m$ from the surface. This uncertainty falls to about $\pm 0.0001 \ \AA$ or less for deeper measurements.

- Intensity-depth-profile normalization with respect to an ideal strain-free intensity depth profile. This is not a very significant source of measurement uncertainty. The uncertainty in normalizing the data is typically less than 5% of the normalization factor value. This will result in a
Figure 3.74 6A shot peened Al-6061 [111] intensity depth profile on a logarithmic scale. The uncertainty in locating the sample surface is the scan step size.

$\Delta d_{hkl}$ uncertainty of $\pm 0.0002\text{Å}$ or less assuming that the sample surface and the uncertainty in determining it are already determined. Estimating the sample surface location differently will result in a slightly different intensity normalization factor, however, the uncertainty in this factor as well as the sample surface location will roughly stay the same. This source of measurement uncertainty can be significantly reduced if a simple statistical method such as a least squares minimization process is used. However, with the previously mentioned sources of uncertainty, improving this one will not improve the overall measurement accuracy.

- Natural fluctuation in the diffraction signal intensity. Obviously this source of measurement uncertainty depends on the sample material and the counting time at each step during the scan process. In all of the intensity depth profiles presented in this thesis, this source of measurement uncertainty accounts for $\pm 0.0001\text{Å}$ or less in $\Delta d_{hkl}$ measurements across the whole depth profile.

- The natural width of the tungsten $K_{\alpha1}$ line is $43.2 \text{ eV} \pm 5 \text{ eV}$. This source of measurement uncertainty accounts for less than $\pm 0.0001\text{Å}$ in $\Delta d_{hkl}$ measurements across the whole depth profile.
The estimated ranges of uncertainty in $\Delta d_{hkl}$ shown in this thesis are the most likely measurements uncertainty ranges. This most likely measurement uncertainty is estimated by plotting a distribution of all of possible combinations of the uncertainty sources listed above. The most likely $\Delta d_{hkl}$ value will be the center of that distribution. The most likely uncertainty range is the range that covers 70% of this distribution around the central value. An example of the uncertainties distribution when $x = 170 \, \mu m$ in 6A aluminum shot-peened sample is shown in figure 3.75.

### 3.8.1.3 More results

Figure 3.76(a) shows the intensity depth profiles of an aluminum sample for various shot-peening levels. Calculating the strain with the aid of the strain-intensity data in figure 3.62 yields the strain depth profiles shown in figure 3.76(b). More aluminum intensity depth profiles and their corresponding strain profiles calculated using this intensity method can be found in appendix C.1.

It should be emphasized here that the sample grain size should be smaller that the beam collimation. A large grain size may cause some fluctuation in the diffracted beam intensity in the regions inside the sample where Bragg’s law is not met or where the number of grains that meet the diffraction condition changes significantly from one point to another inside the sample. To make sure that any intensity drop is not due to the grain size/distribution effect, the diffraction angle can be changed to a slightly different angle such that it meets Bragg’s diffraction condition for the strained plane spacing $(d) = d_0 + \Delta d$ and the incident energy $E_0$. Then an intensity depth profile should be collected at that angle. The new profile should gain intensity where the intensity was low at the angle $\theta_{E_0,d_0}$ depth profile. That is, when the tungsten characteristic line $K_{a1}$ is used, the intensity depth profile at $\theta_{K_{a1},d_0}$ will have maximum intensity when $d = d_0$. In the region where $d = d_0 + \Delta d$, the maximum intensity is achieved when $\theta = \theta_{K_{a1},d_0+\Delta d}$. Examples of such depth profiles for 8A 10A and 12A shot peening levels are shown in appendix C.1.
Figure 3.75  Distribution of all the possible uncertainties combinations when \( x = 170 \, \mu m \) in a 6A shot peened aluminum [111] strain depth profile. \( \Delta d \) most likely uncertainty bars represent 70% of all the possible \( \Delta d \) uncertainty combinations. \( E = K_{\alpha 1} \) and \( \theta = 2.562^\circ \).
Figure 3.76 Aluminum intensity and strain depth profiles for various shot-peening levels. The strain is calculated using the intensity-change method.
3.8.2 Method 2: using the energy dispersive intensity depth profiles

This method is a well established method. It is generally less accurate than the previous intensity method because of its dependence on the statistics of the intensity of the energy dispersive data rather than the integrated intensity. Although this method is well established, we are presenting here a very rigorous and detailed treatment for the various instrument functions which will result in more accurate strain measurements. This method is more time consuming and less accurate when compared to the intensity method but we are studying it to demonstrate the validity of first method in section 3.8.1 as well as the consistency between the various methods.

In an energy dispersive fixed angle x-ray diffraction experiment, assuming an ideal white beam with no divergence, the relation between the energy and the planes spacing is \( d_{hkl} E = \text{constant} \). However, this relation gets complicated when the incident beam is a characteristic line, which naturally has a Lorentzian shape, and when the divergence of the beam is of the same order as the width of the characteristic line. This relation gets even more complicated when the probing volume is partially inside the sample. This is another place where the diffraction setup simulator can be very helpful.

To use this method, the diffraction angle \( \theta \) is fixed such that it satisfies Bragg’s diffraction condition for the incident characteristic line, the tungsten \( K_{\alpha 1} \) in this case, for the unstrained lattice parameter. In the case of an aluminum sample and incident \( W_{K_{\alpha 1}} = 59.318 \text{ keV} \), the diffraction angle should be \( \theta = 2.562^\circ \). The angle-dispersive diffraction peak curve should look like the simulated dotted curve in figure 3.77. Any slight change in the lattice parameter will result in a different EDXRD peak location at that fixed angle. Due to the shape of the incident beam and the geometry of the collimation, the diffraction peak is not simply another peak in a different location that satisfies the simple Bragg law. Instead, the diffraction peak looks like two overlapping peaks with one strong peak, the result of diffracting the characteristic line, in a position that is very close to the location of the characteristic line, the other peak will be a broader less intense diffraction peak from the bremsstrahlung. This broader less intense peak is the result of diffracting the bremsstrahlung radiation. The dashed and the solid curves in figure 3.77 are examples of two different diffraction peaks for two different strain values. In an actual experimental data, the diffraction peak does not look like two overlapping peaks because of the detector limited resolution. The HPGe detector used here has a 0.4 keV resolution. By convoluting the
diffraction peaks shown in figure 3.77 with this instrument function we get the peaks shown in figure 3.78. The next step is to establish a relation between this measured peak location and the strain. This can be accomplished by creating a large number of curves such as the ones in figure 3.77. A strain range of ±0.01 Å in steps of 0.0002 Å around the unstrained lattice parameter value, \( d_{111} = 2.3378 \) Å, is a reasonable choice. Such set of curves are combined in a two dimensional plot in figure 3.80. The peaks in figure 3.77 represent vertical slices from figure 3.80 at the corresponding strain values. The result of applying the detector instrument function to every vertical slice from figure 3.80 is shown in figure 3.81. Again, each vertical slice from figure 3.81 represents the diffraction peak for the corresponding strain on the x-axis. The centers of these peaks are represented by the dotted line in figure 3.81. This line represents the relation between the energy-dispersive diffraction peak location and the strain at a fixed angle. This dotted curve is the same as the solid curve in figure 3.79. All of the simulated data shown in figures 3.77 and 3.78 and 3.79 and 3.80 and 3.81 are for aluminum [111] and \( \theta = 2.562^\circ \) when the whole probing volume is inside the sample. The same process should be repeated for all possible probing volume locations to get the energy-strain relation at that probing volume location. A subset of such curves is shown in figure 3.82. Combining these energy-strain curves in one two-dimensional plot will result in figure 3.83 which is basically a strain look-up table for aluminum when [111] peak is used and the incident energy is \( W_{K\alpha} = 59.318 \) keV and \( \theta = 2.562^\circ \).

Once a table like the one in figure 3.83 is obtained, an energy dispersive intensity depth profile at the same angle and energy that were used to generated the lookup table need to be collected in the lab. In addition to the 6A shot peening level data shown in figure 3.67(b), more data for more shot peening levels (specifically for 8A, 10A and 12A) can be found in appendix C.2. A vertical slice from the energy-dispersive intensity depth profile at a given location represents the diffraction peak at that location. By fitting the vertical slices with gaussian functions, the EDXRD peak location versus depth is constructed. The result of such a process for non peened aluminum sample is shown in figure 3.40. The ideally expected energy peak location versus depth is the dashed curve in that same figure 3.40. The experimental depth profile is obviously in a good agreement with the theoretical one.

Figure 3.84 shows Al [111] energy-dispersive peak location versus depth for 4A 8A and 12A shot-peened samples. This figure illustrates the difference in these profiles for various peening levels. The
higher shot-peening levels, as expected, have the highest deviation from the unstrained sample while lower shot-peening levels show a smaller deviation from the unstrained profile. More data for more shot peening levels can be found in appendix C.2.

The next step is to use the energy-peak-location depth profile along with the two dimensional plot in figure 3.83 to find the strain depth profile. Such strain-depth profiles are shown in figure 3.85 (see appendix C.2 for more peening levels). As expected, higher shot-peening levels resulted in deeper strain profiles.

Figure 3.77 The ideally expected shape for Al [111] energy dispersive diffraction peak for strain-free and strained samples. $\theta = 2.562^\circ$. 
Figure 3.78 Al [111] energy dispersive diffraction peak for various strain values after applying the detector function 0.4 keV.

Figure 3.79 The relation between the location of Al [111] energy dispersive diffraction peak and the strain as calculated from figure 3.78. The dotted line represents the ideally expected value from Bragg’s law.
Figure 3.80  The intensity of Al energy dispersive [111] XRD peak versus $d_{111}$.

The incident beam is the tungsten $K_{\alpha 1}$ with some bremsstrahlung radiation similar to the white beam shown in figure 3.4. $\theta = 2.562^\circ$. 

$\Delta d=0.004$ Å  
$\Delta d=0.010$ Å  
$\Delta d=0.000$ Å  
$\Delta d=0.004$ Å
Figure 3.81 The intensity of Al energy dispersive [111] XRD peak versus $d_{111}$ after applying the detector resolution. The incident beam is the tungsten $K_{\alpha 1}$ with some bremsstrahlung radiation similar to the white beam shown in figure 3.4. $\theta = 2.562^\circ$. 
Figure 3.82  The expected relation between the energy-dispersive location of aluminum [111] diffraction peak and [111] lattice planes spacing for various probing volume locations when $\theta = 2.562^\circ$. 
Figure 3.83 A two dimensional plot constructed using a set of plots similar to what is shown in figure 3.82. This plot can be used as a lookup table to find the strain for a given energy dispersive peak location at a given probing volume location for aluminum sample when $\theta = 2.562^\circ$. The x-axis is the position of the center of the probing volume with respect to the sample surface.
3.8.2.1 Error analysis

The sources of measurements uncertainties for this method, from the most significant one to the least significant one, are:

- The uncertainty in locating the energy dispersive diffraction peak. This uncertainty has two causes: The uncertainty in the MCA-detector system calibration and the peak fitting process. More information about this calibration process can be found in appendix A. Typically the error in the energy dispersive detector calibration is less than ±0.01 keV. Obviously, locating the center of the diffraction peak depends heavily on the count rate. This source of error typically results in a ±0.001Å in Δdhkl measurements close to the sample surface and less than ±0.0005Å afterward. Deeper in the sample, when the count rate starts going down significantly, this source of error may become very significant to the point where a measurement may become useless. Typically, usable depth profiles can be as deep as 500 µm to 800 µm in aluminum and 250 µm in titanium using this method.

- Pin-pointing the sample surface location with respect to the probing volume. A ±10 µm uncertainty in locating the sample surface, for example, will result in typically ±0.001Å error in Δdhkl measurements close to the sample surface. Deeper in the sample this error drops to about ±0.0002Å.

- The collimators misalignment. A ±10 µm uncertainty in positioning the collimators is typical. In this method, this source of error will result in about ±0.0005Å in Δdhkl measurements across the whole depth profile.

- The ±5 eV uncertainty in the natural width of the Kα1 line will typically result in ±0.0002Å in Δdhkl measurements across the whole depth profile.
Figure 3.84 Energy-dispersive [111] diffraction peak location at different depth for different shot-peening levels in an aluminum sample.
Figure 3.85 Change in lattice planes spacing versus depth calculated using the energy dispersive method for various shot-peening levels.
3.8.3 Method 3: using traditional angle-dispersive diffraction technique

This method is the most time consuming method. It will be used only to demonstrate the consistency between the various measurement techniques. In this case, the energy dispersive detector is used to discriminate against all energies but the tungsten narrow $K_{\alpha 1}$ line. The probing volume is moved to a specific location inside the sample, then the detector and the sample are rotated while keeping the probing volume in its position. The process is then repeated for various locations inside the sample. As with the previous two methods, taking the geometry of the setup into account is of utmost importance especially when the probing volume is partially inside the sample. Figure 3.86 shows the relation between the lattice planes spacing for an aluminum sample and the diffraction angle at various locations. The same data in this figure can be presented in a different and more informative way as shown 3.87.

Once the angle dispersive diffraction data is collected, figure 3.87 can be used as a lookup table to find the lattice parameter for that angular peak position and probing volume position.

![Figure 3.86 The expected relation between the angular location of Al [111] diffraction peak and [111] lattice planes spacing for various probing volume locations when $E = 59.318$ keV.](image)
Figure 3.87 The calculated angular location of Al [111] diffraction peak for a given planes spacing and probing volume location. This data is the same as the one shown in figure 3.86 but shown in a different way.
3.8.3.1 Error analysis

Starting with the most significant source of uncertainty, the sources of measurement uncertainty for this method are:

- Defining the sample surface location. This source of error is significant only close to the sample surface when the probing volume is not fully inside the sample. Otherwise it is almost negligible in this method. Close to the sample surface, this error can be as high as $\pm 0.002 \text{Å}$ in the first 10 $\mu m$ to 50 $\mu m$ from the surface and will typically drop to 0.001Å to 0.0005Å for measurements between 50 $\mu m$ to 100 $\mu m$ from the surface. Figure 3.86 and 3.87 provide a good guide for estimating this error in an aluminum sample. An estimate for the range of possible values of the probing volume location will directly give the possible range of $\Delta d_{hkl}$ value.

- The collimators misalignment. A $\pm 10 \mu m$ uncertainty in positioning them will result in $\pm 0.0005 \text{Å}$ in $\Delta d_{hkl}$ across the depth profile when the probing volume is fully inside the sample.

- Defining the detector angular zero position. Typically the detector location is defined within $\pm 0.001^\circ$. This uncertainty will result in $\pm 0.001 \text{Å}$ uncertainty in $\Delta d_{hkl}$ across the depth profile.

- Statistical error in locating the center of the angle-dispersive diffraction peak. This uncertainty depends heavily on the intensity of the diffraction peak. Typically the uncertainty in the diffraction peak fitting process is less than $0.0005^\circ$. This will result in less than $\pm 0.0005 \text{Å}$ error in $\Delta d_{hkl}$ depth profile.

3.8.3.2 Results

For a strain-free sample, and when the probing volume is fully inside the sample, we expect the diffraction peak location to stay the same at all depths. This is illustrated in figure 3.88(a) for a strain-free Al-6061 sample and incident energy $W_{K\alpha 1} = 59.318 \text{ keV}$. The angular position of [111] diffraction peak is then found using curve fitting from which the lattice parameter can be found using figure 3.87. The result is shown in figure 3.89. A similar set of $\theta$-$2\theta$ for 4Å shot-peened Al-6061 sample is shown in figure 3.88(b) with the angle-dispersive peak location found using curve fitting in figure 3.89. More data for more shot peening levels can be found in appendix C.3.
Figure 3.88  (a) Non-peened and (b) 4A shot-peened Al-6061 [111] $\theta$-dispersive XRD peaks at different depths.
Figure 3.89 4A peened and non-peened Al-6061 [111] θ-dispersive XRD peaks at different depths.
3.8.4 Comparison and suggested improvements

Figures 3.90 and 3.91 show 4A and 10A aluminum $\Delta d(x)_{111}$ depth profiles, respectively, for various shot-peening levels measured using the three different methods discussed in sections 3.8.1, 3.8.2 and 3.8.3. More data comparing the three different methods for more peening levels can be found in appendix C.4. The three different methods seem to agree very well within the given ranges of measurement uncertainty. More about how to improve the measurements and reduce the uncertainties can be found in chapter 4. Note that some of the sources of measurement uncertainty discussed in sections 3.8.1.2 and 3.8.2.1 and 3.8.3.1 are systematic errors that affect all of the collected data equally. A system mis-alignment or detector calibration error are examples of such systematic errors. These sources of error will usually change all of the measured energy or intensity profiles consistently. A possible example of such systematic error can be seen in figure 3.91. Note in that figure that $\Delta d(x)_{111}$ depth profile measured using the normalized intensity method and the profile measured using the angle-dispersive method have the same shape but one of them is shifted vertically by approximately the same amount from the other one. Such a disagreement might be due to a detector calibration error for example. These systematic sources of error are easier to eliminate compared to the completely random ones such as the statistical fluctuations in the intensity.
Figure 3.90 change in lattice planes spacing for a 4A shot-peened aluminum measured using the three different methods.
Figure 3.91 change in lattice planes spacing for a 10Å shot-peened aluminum measured using the three different methods.
3.9 Residual stress thermal relaxation

Now that we have finished discussing how to, nondestructively, measure strain depth profiles using the three different techniques in section 3.8, we are showing here a preliminary study of the residual stress thermal relaxation as an example of such a measurement. Thermal relaxation of residual stresses is important for many reasons. For example it:

- Causes critical distortions in some parts of jet engines which, at least, affects the engine performance (50; 51).
- Affects the apparent interfacial fracture toughness of polymer/metal interfaces (52).
- Impacts the reliability of multilayered Micro-Electro Mechanical Systems (MEMS) (53).
- Reduces the reliability of welds and initiates cracks (54).
- Affects the quality of the interconnects on wafers (55).

There are already some studies, using destructive techniques, on thermal relaxation of residual stresses in some alloys such as Ti-6Al-4V (51) and IN100 (50). We demonstrate in this section the possibility of performing such measurements in a matter of minutes with enough sensitivity, nondestructively, in a relatively low-cost lab-based setup. The measurement techniques we presented in section 3.8 is fast enough to enable, for example, in situ measurements of thermal residual stress relaxation. Such a study can be part of a future research project. In situ measurement of the thermal relaxation of the residual stress profile is not possible using the currently available destructive techniques for many obvious reasons such as the long time it takes to make the measurement.

Another advantage for the nondestructive technique we presented earlier is the ability to perform repeated measurements on the same sample. For example, in this section, we will show how the strain depth profile is affected by exposing the same sample repeatedly to the same heat cycle.

Intensity depth profiles of 6A shot peened Al-6061 samples [111] diffraction peak exposed to 300°C for various periods of time are shown in figure 3.92. The samples were put in a 300°C preheated oven for the indicated period of time and then they were allowed to cool down before collecting the data. The strain depth profiles calculated using the normalized intensity method are shown in figure 3.93 and
using the energy dispersive method in figure 3.96. It is hard to draw any conclusion about the first 50 μm under the surface. However, the shot peened region is about 300 μm deep. We found, as evident in figure 3.93, that the strain relaxes at a slower rate close to the sample surface. The relaxation rate is higher in the regions that are farther from the surface. A plot of Δd against the heating time for selected depths for the data shown in figure 3.93 is shown in figure 3.94. The decay of the ratio \( \frac{Δd(t)}{Δd(t=0)} \) with time is shown in figure 3.95. More data for 9A and 12A shot peened Al 6061 samples can be found in appendix C.5.

We also collected some preliminary data to study the effect of exposing the same sample to the same heat cycle several times. As an example of such a process, we collected the intensity depth profiles from an aluminum 6061 sample after exposing it multiple times to a temperature of 300°C for 30 minutes. The sample was allowed to cool down after each heat cycle before collecting the intensity depth profile. The result of such a process is shown in figure 3.97. The strain depth profiles as calculated using the normalized intensity method are shown in figure 3.98. A similar result using the energy dispersive method is shown in figure 3.99. As we can see in figure 3.98, the relaxation rate is higher in the regions that are farther from the sample surface. More results for 9A and 12A shot peened aluminum 6061 can be found in appendix C.5.
Figure 3.92 6A shot-peened Al-6061 [111] intensity depth profiles after various exposure times to 300°C. The x-axis is the position of the center of the probing volume with respect to the sample surface.
Figure 3.93 $\Delta d_{[111]}$ depth profiles of the heat treated 6A shot-peened Al-6061 samples in figure 3.92 as measured using the intensity change method.
Figure 3.94  6A shot-peened Al-6061 $\Delta d_{[111]}$ thermal relaxation vs. exposure time for selected depths. Temperature = 300°C.

Figure 3.95  6A shot-peened Al-6061 $\frac{\Delta d_{[111]}(t)}{\Delta d_{[111]}(t=0)}$ thermal relaxation vs. exposure time for selected depths. Temperature = 300°C.
Figure 3.96 $\Delta d_{[111]}$ depth profiles of the heat treated 6A shot-peened Al-6061 samples in figure 3.92 as measured using the energy dispersive method.
Figure 3.97 6A shot-peened Al [111] intensity depth profiles after multiple of exposures to 300°C for 30 minutes. The x-axis is the position of the center of the probing volume with respect to the sample surface.
Figure 3.98 $\Delta d_{[111]}$ depth profiles of the heat treated 6A shot-peened aluminum samples in figure 3.97 as measured using the intensity change method.
Figure 3.99 $\Delta d_{[111]}$ depth profiles of the heat treated 6A shot-peened aluminum samples in figure 3.97 as measured using the energy method.
CHAPTER 4. Summary and Future Work

4.1 Contributions

As stated in chapter 1 and chapter 2, residual stresses can be measured using a variety of techniques. Each technique has its own advantages and disadvantages (see table 2.1). Most of these techniques are destructive if a depth profile is obtained. The only nondestructive techniques are either electromagnetic techniques or diffraction techniques that require a synchrotron or a neutron source. Electromagnetic techniques suffer from many drawbacks. Magnetic methods that utilize the magnetoelastic effect have about 1 mm spatial resolution. This spatial resolution is not acceptable many applications such as residual stresses in shot-peened surfaces. These magnetic methods suffer also from being limited to ferromagnetic materials and the measurement is very sensitive to other material properties such as the hardness, the texture and the grain size. Eddy current based techniques have a good spatial resolution, but they do not even work in some materials because of the sensitivity to the cold work or the heat treatment of the sample. Synchrotron and neutron based measurements can, nondestructively, construct a measurement depth profile. However, such facilities exist in a handful of places around the world and the beam time is very limited.

In this thesis, we presented the results of building an experimental setup for measuring strain depth profiles in materials. This experimental setup can, nondestructively, measure strain depth profiles using three different techniques: a new XRD normalized-intensity technique, angle-dispersive XRD, and energy-dispersive XRD. The experimental results presented are supported with the results of a modeling program. This modeling program is very helpful in many ways: It helped predict and explain the intensity and strain depth profiles in many cases that were otherwise ambiguous. It helped convert intensity, angle, and energy measurements into strain values. This modeling program is also extremely helpful in estimating the uncertainties in the strain depth profiles.
This new high energy XRD strain measurement technique is unique in many aspects:

- Nondestructive strain depth profiles, more than 1 mm in aluminum and about 300 \( \mu m \) in titanium, can be constructed. This high penetration is achieved by using 320kVp x-ray tube source.

- Lab based: The fact that this is a lab based technique means that it is cost effective and has significantly better accessibility. Measurements can be done on a large number of samples, cost effectively, around the clock.

- Strain measurements can be done using a normalized intensity value by utilizing the width of the incident characteristic line and the divergence of the incident and diffracted beam. Traditional, diffraction based, strain measurement methods rely on locating a diffraction peak in \( \theta \) space by moving the detector around the sample. Being able to get a measurement without moving the detector around the sample means a significantly faster measurement. A measurement can be made by reading a diffraction peak total intensity at a single point in space rather than making the same intensity measurement at different locations around the sample to construct an angle-dispersive diffraction peak. Measurements on aluminum and titanium, for example, take only few minutes per point along the depth profile. Also since no curve fitting is needed in this measurement to locate a diffraction peak, it is more accurate than the traditional techniques that are more prone to the statistical fluctuations and require locating a diffraction using statistical curve fitting techniques.

- A depth profile spatial resolution as low as 50 \( \mu m \) is possible. The measurement spatial resolution in materials like aluminum and titanium is mainly limited by the sample grain size.

- Unlike the electromagnetic methods, the system described in this thesis can perform measurements on a wide range of samples. It is not limited by the cold work or the magnetic properties of the material. A measurement can be done on any material as long as it has a lattice structure and the geometry of the sample does not block the diffracted beam.

Being able to construct nondestructive, high resolution strain depth profiles in a few minutes in materials like aluminum and titanium has a significant impact on many fields. For example, jets engines
turbine blades and gears can be investigated for the harmful and undesirable tensile strains in critical regions in order to predict possible parts failures before they happen. The fact that this is a nondestructive technique means that the investigated parts do not have to be discarded if it turns out that those parts still meet the required safety standards. The fact that those measurements can be done within a reasonable time is a big advantage in cases where routine measurements or measurements on large number of samples are desired.

Finally, we presented a preliminary study of the thermal relaxation of residual stress in shot-peened Al-6061 alloy. We found that the residual stress relaxes at a faster rate deeper inside the sample. The relaxation rate is slower in the surface region. A more detailed study of this behavior and its causes will be part of a future work.

### 4.2 Suggested improvements and future work

There are many improvements and upgrades that can be done to enhance the system as well as the data analysis software:

- As already stated, the three different measurement methods depend heavily on the experimental setup modeling program. This ability to model the measurement process is an emerging capability that is being recognized. It is important to be able to understand the complex elements affecting a measurement. There is still a lot of room for improving this modeling program. The most important improvement that is currently being worked on is adding a third spatial dimension. The program currently assumes two dimensional system. The third dimension that is vertical to the source-sample-detector plane is completely ignored mainly because the time it takes to complete a simulation run with the third dimension included is prohibitively very long. The code will benefit from further optimization although for a 3D modeling, even after optimizing the code, techniques like parallel computing or GPGPU might be needed\(^1\). Although the agreement between the experimental and the theoretical data is very good in its current state, the agreement is expected to be even better especially close to the sample surface when the probing

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\(^1\)General purpose computing on graphics processing units (GPGPU) is the technique of using a graphics processing unit (GPU), which typically handles computation only for computer graphics, to perform computation in applications traditionally handled by the CPU. This allows for running thousands of simultaneous threads on a single machine.
volume is partially inside the sample and when there is a steep strain gradient inside the probing volume.

- The simulation program currently gives the average $\Delta d_{hkl}$ inside the probing volume at a given depth for a specific energy or normalized intensity value. In some cases, the strain gradient inside the probing volume can be high enough to make this assumption impractical or inaccurate. One of the planned improvements for the simulator is to divide the probing volume into smaller volumes and let the program work with these smaller volumes. The program then returns the average $\Delta d_{hkl}$ for each one of those smaller volumes. The plan is to make the measurement resolution limited by how fine the probing volume is stepped inside the sample in a depth profile measurement rather than how wide or narrow the collimation is.

- One of the most significant sources of error in the experimental data is the uncertainty in the system alignment. In all of the data presented in this thesis, an error of $\pm 10 \mu m$ in positioning the collimators is assumed. However, the collimators positioners have a $\pm 1.5 \mu m$ positional repeatability. There is some room for improving the system alignment process. Pushing this alignment error from this $\pm 10 \mu m$ to a $\pm 2 \mu m$ error, for example, will shrink the error bars by roughly half of their current values. This can be done by first improving the concentricity of the rotary stages. Currently the rotary stages are concentric within about $\pm 100 \mu m$. It is not actually clear if this concentricity can be improved further or not. We then need to try aligning the system collimators using low energy ($< 20 keV$) x-rays. This will hopefully reduce the effect of having a collimator rotated slightly around its center in the source-sample-detector plane which will reduce the effect of the x-rays leaking through the collimators edges. However, it is not clear whether the x-ray tube hottest spot position on the target changes with the tube voltage/current or even if that approach will be more beneficial than the current alignment process. These potential alignment improvements do not require any modifications to the current setup.

- Defining the sample surface is another significant source of uncertainty. Currently this is estimated to be about $\pm 20 \mu m$. This error can also be improved significantly by, for example, depositing a material with a very low x-ray attenuation and a known diffraction signal on the
sample surface. The energy dispersive detector becomes very helpful in this case because the sample diffraction signal will still be unaltered by the deposited material signal because of the different diffracted energies from the sample and the deposited material.

- So far, the strain depth profiles can be measured using the three techniques in section 3.8. A fourth strain depth profile calculation method is still under investigation. This fourth method uses curve fitting to find the best strain depth profile that will give an experimental intensity depth profile with the aid of the setup modeling program. This approach needs either a functional or numerical model for the strain depth profile of shot peened material. There are already some simple functional and more complicated numerical forms that describe strain depth profiles (56–69). The problem with the functional forms is that they do not seem to be accurate enough to explain the intensity depth profiles we are getting from shot peened aluminum. The numerical models, though more accurate, have their own share of problems. When these numerical models that describe shot peened material strain depth profile are incorporated in the experimental setup modeling program, the modeling runs maybe become very slow depending on the complexity of the model.

- The system can currently make two dimensional strain maps. With a simple upgrade, the system should be able make three dimensional strain maps. This upgrade requires adding a third linear stage to the sample holder and requires updating the data acquisition and control software to be able to communicate with that third axis.

- Working with titanium samples: Most of the data presented in this thesis is Al-6061 diffraction data; however, some preliminary data from titanium Ti-6Al-4V alloy was collected. Nondestructive strain measurements from this titanium alloy are of a great importance because of its wide spread usage in jet engines. These preliminarily data demonstrate the ability to collect intensity depth profiles, from which strain depth profiles can be constructed, from titanium alloys. An experimental intensity depth profile collected from a titanium sample with various surface treatments is shown in figure 4.1. The solid curve in figure 4.1 is the intensity depth profile collected from a low-strain titanium sample. Shot-peened titanium surfaces typically have strain profiles
that extend to about 200 µm below the surface. This is demonstrated in figure 4.1. The intensity depth profiles from the shot-peened and non-peened surfaces have different intensities until the point where the two surfaces have the same strain. Laser peened surfaces typically have deeper strain profiles that can be as deep as 1 mm which explains why the intensity depth profile from the laser peened surface does not match the one from the low strain sample within the shown scan range. Figure 4.1 demonstrates that intensity, and consequently strain, measurements can be obtained from titanium samples from regions as deep as 300 µm under the surface. We showed in section 3.8 how to convert aluminum intensity depth profiles to strain depth profiles. This process is fundamentally the same for all materials. However, converting such intensity depth profiles from titanium samples to strain depth profiles will be part of a future work.

![Intensity Depth Profiles](image)

**Figure 4.1** Ti-6Al-4V [101] XRD peak total intensity depth profiles when the peak is tuned to be at $K_{\alpha1}$ line location. The x-axis is the position of the center of the probing volume with respect to the sample surface.

- Study of in-situ thermal relaxation of residual stresses: We are not aware of any literature in this area. In-situ measurement of the thermal relaxation of the residual stress profile is not possible using the currently available destructive techniques for many obvious reasons such as the long time it takes to make the measurement. Such a study may, for example, reveal new information about how the strain depth profiles evolve during the temperature ramp-up or cool-down.
• Plastic deformations (cold-work) effect on the thermal relaxation of residual stresses: Increased shot-peening intensity increases the plastic deformations in the shot-peened material, especially close to the sample surface, which results in broadening the diffraction peak. That is why the diffraction peaks in figure 3.88(b) are very broad close to the sample surface compared to the diffraction peaks deeper inside the same sample or compared to the diffraction peaks from the non-peened sample in figure 3.88(a). Figure 4.2 shows the diffraction peak FWHM depth profile of a 12A shot-peened Al-6061. Note the increased diffraction peak FWHM close to the sample surface in the shot peened region. The increased cold work in the surface region seems to slow down the residual stress thermal relaxation (see figures 3.92, 3.93, 3.94, and 3.95 in section 3.9). The exact effect of this increased cold-work on the residual stress thermal relaxation has yet to be investigated.

![Diagram](image)

**Figure 4.2** 12A shot-peened Al-6061 [111] diffraction peak FWHM depth profile.
APPENDIX A. Detector-MCA Calibration

To calibrate the detector we used $^{241}\text{Am}$ radioactive isotope. This isotope naturally radiates the spectrum shown in figure A.1. The peaks 1 through 9 shown in figure A.1 have the energies and intensities shown in table A.1 (70). The centers of the peaks 1 through 9 in figure A.1 should be determined using curve fitting. The result is shown in table A.2 and plotted in figure A.2. Note the linearity of the calibration over a wide range of energies. The last column in table A.2 shows the difference between the actual tabulated value for $^{241}\text{Am}$ peaks listed in table A.1 as obtained from reference (70) and the calculated value after fitting the nine peaks with a linear function. Note that the error is generally around 0.01 keV or less which meets the necessary required accuracy for measuring the energy.

<table>
<thead>
<tr>
<th>Peak #</th>
<th>Energy (keV)</th>
<th>I (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>26.34482</td>
<td>2.402</td>
</tr>
<tr>
<td>2</td>
<td>33.19643</td>
<td>0.1263</td>
</tr>
<tr>
<td>3</td>
<td>43.4231</td>
<td>0.0738</td>
</tr>
<tr>
<td>4</td>
<td>55.562</td>
<td>0.018118</td>
</tr>
<tr>
<td>5</td>
<td>59.54122</td>
<td>35.94</td>
</tr>
<tr>
<td>6</td>
<td>69.763</td>
<td>0.00294</td>
</tr>
<tr>
<td>7</td>
<td>98.972</td>
<td>0.02034</td>
</tr>
<tr>
<td>8</td>
<td>102.982</td>
<td>0.01954</td>
</tr>
<tr>
<td>9</td>
<td>125.302</td>
<td>0.004089</td>
</tr>
</tbody>
</table>

Table A.1 List of selected gamma rays energies emitted from $^{241}\text{Am}$ (70).
Figure A.1. $^{241}$Am spectrum.
<table>
<thead>
<tr>
<th>Peak #</th>
<th>Peak location</th>
<th>Energy (keV)</th>
<th>Calculated energy using curve fit</th>
<th>Error (keV)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2330.70</td>
<td>26.34482</td>
<td>26.344</td>
<td>-0.001</td>
</tr>
<tr>
<td>2</td>
<td>2937.77</td>
<td>33.19643</td>
<td>33.197</td>
<td>0.000</td>
</tr>
<tr>
<td>3</td>
<td>3843.14</td>
<td>43.4231</td>
<td>43.417</td>
<td>-0.006</td>
</tr>
<tr>
<td>4</td>
<td>4917.78</td>
<td>55.562</td>
<td>55.548</td>
<td>-0.014</td>
</tr>
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<td>59.54122</td>
<td>59.551</td>
<td>0.009</td>
</tr>
<tr>
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<td>69.763</td>
<td>69.780</td>
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</tr>
<tr>
<td>7</td>
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<td>98.972</td>
<td>98.963</td>
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</tr>
<tr>
<td>8</td>
<td>9118.64</td>
<td>102.982</td>
<td>102.968</td>
<td>-0.014</td>
</tr>
<tr>
<td>9</td>
<td>11097.10</td>
<td>125.302</td>
<td>125.302</td>
<td>0.000</td>
</tr>
</tbody>
</table>

Table A.2  Comparison between the tabulated gamma rays energies from $^{241}\text{Am}$ and the energies calculated assuming linear fit.

\[ E = 0.01128833 \times C# + 0.03399420 \]
\[ R^2 = 0.9999985 \]

Figure A.2  MCA calibration: fitting nine peaks from $^{241}\text{Am}$ source with a straight line.
APPENDIX B. System Alignment Process

This section is not intended to give the exact details of the alignment procedure. The goal here is to provide enough information about the alignment procedure in order to help understand the various sources of error and give some insight on how to improve this process, and subsequently the accuracy of the measurement. For more detailed alignment process please refer to the system operation manual.

The alignment process goal is:

- Make sure that the detectors 210RT rotary table and the sample 206RT rotary table have the same center of rotation. We relied on the rotary stages manufacturer in making sure that the stacked rotary stages are concentric. However, the concentricity of these stages can be improved even more. The details of this step will not be discussed here and can be found in the system operation manual.

- The incident beam collimators should define a line between the hottest spot on the x-ray source and the center of rotation of the detector and the sample rotary stages.

- The diffracted beam collimators should define a line that connects the rotary stages center of rotation and the detectors opening.

- The hottest spot on the source, the detector and the collimators should be leveled before proceeding with the alignment process.

- Define the zero position of the detector angular location $2\theta = 0^\circ$. The four collimators should form a straight line that passes from the x-ray source through the four collimators and the rotary stages center of rotation to the detector.

The alignment process, assuming that all components are on the same vertical level, can be summarized in the following steps:
1. The x-ray tube has two spot settings: 3.6\text{mm} and 1.9\text{mm}, see figure B.1. Although the small spot setting has a lower maximum power rating, its intensity distribution is significantly smaller than the large spot setting. Since we will be working with collimations that are significantly less than both spot settings, the setting with the tighter intensity distribution will serve better. The tube should be operating in the \textit{small spot} mode at all times.

![Figure B.1 Pinhole images of the x-ray tube large and small spots. The two images use the same spatial and intensity scales and the same exposure.](image_url)

(a) Large spot: 3.6 mm setting.  
(b) Small spot: 1.9 mm setting.

2. Move the detector $2\theta$ arm to its zero position. This can be done with the aid of a crosshair and the x-ray source and an imaging detector. An image should be taken for a crosshair placed at different locations on the detector $2\theta$ arm. The $2\theta$ arm position should be fine tuned until the point where sliding the crosshair on the $2\theta$ arm does not shift the location of its image. Sample images for the crosshair at various locations are shown in figure B.2.

3. Define the rotary stages center of rotation: A specially prepared pin with a fine hole that passes through the center of the pin should be installed on top of a goniometer or a two dimensional x-y miniature linear stage. The pin-goniometer assembly should then be installed on top of the
rotary stages. Using a telescope, or a two dimensional imaging detector and the x-ray source, the pin position should be adjusted using the goniometer, or the x-y miniature linear stages, such that moving the two rotary stages from 0° to 360° results in rotating the pin around its center without precession. The fine hole underneath the tip of the pin will help aligning the collimators later. Figure B.3 shows an image of the pin-hole. The pin tip, and subsequently the hole underneath it, should now be on top of the center of rotation.

4. All x-rays that do not pass through the hole, and subsequently the center of rotation, must be blocked using a proper shielding. Most of the counts that reach the detector at this point should be coming through the center of rotation, see figure B.4.
5. The pin should next be rotated such that the maximum intensity through the fine hole is achieved as shown in figure B.5. The hole-pin and the hottest spot on the tube now form a line that will next be used as a guide in the rest of the alignment process. The detector at this point is wide open and should be positioned roughly at $\theta = 0^\circ$. Accurate positioning of the detector is not necessary at this point as the detector is wide open. A two dimensional imagining detector can be used instead of the HPGe detector if available.

6. Position the first collimator, the one closest to the source, in the beam. Scan this collimator in steps of 2 $\mu m$ to 10 $\mu m$ in the beam and then put the collimator back in the position of the maximum intensity. Curve fitting can be used to locate the position of the maximum intensity. See figure B.6.

7. Position the second collimator in the beam and use curve fitting to find the position of the maximum intensity. See figure B.7. Move this collimator back to the position of maximum intensity. The incident beam should now be well defined. An image of the direct incident beam with only the first and the second collimators installed is shown in figure B.8.

8. Repeat the previous step for the third, see figure B.9, and the forth collimators, see figure B.10. The system should now be properly aligned assuming all of the previous steps were performed...
Figure B.5 Rotating the fine hole, after defining the center of rotation, to locate the position of maximum intensity. At the position of the maximum intensity the hole should be looking directly at the hottest spot on the x-ray tube.

Figure B.6 Scanning the first set of collimators through the beam to locate the position of the maximum intensity.
Figure B.7 Scanning the second set of collimators through the beam to locate the position of the maximum intensity after positioning the first collimator in its position. The incident beam should now be well defined.

Figure B.8 Two dimensional image of the incident beam after installing the first and the second collimators. The collimators are 135 µm open and 1 mm apart.
properly. For more detailed alignment procedure refer to the system alignment document in the system operation manual.

Figure B.9  Scanning the third set of collimators through the beam to locate the position of the maximum intensity.

9. Perform a one dimensional scan with the detector $2\theta$ arm around the direct straight through beam. The resulting intensity distribution around $\theta = 0^\circ$ should be symmetric and uniform if the alignment was done properly. More tests using an HOPG crystal can be done to verify the alignment even further. A sample $2\theta$ arm scan around $\theta = 0^\circ$ is shown in figure B.11.
Figure B.10  Scanning the forth set of collimators through the beam to locate the position of the maximum intensity.

Figure B.11  $2\theta$ detector arm scan around $2\theta = 0^\circ$ after completing the alignment procedure.
APPENDIX C. More Strain Measurements

C.1 More strain depth profiles measured using the normalized intensity method

Intensity depth profiles, as well as strain depth profiles, for 6A shot peening level of an aluminum sample was shown in section 3.8.1.1. In this section we list similar sets of data for more shot peening levels. Intensity depth profiles for non-peened 4A 6A 8A 9A and 12A are shown in figure C.1. The corresponding strain depth profiles are shown in figure C.2. To illustrate the fact that the changes in the intensity profiles shown in figure C.1 are due to changes in the lattice parameter, intensity depth profiles for those shot peening levels at a different angle from the one used in figure C.1 are shown in figures C.3 and C.6 and C.7 for 8A and 10A and 12A shot peening levels respectively. Note the gain in the intensity, at the points where the intensity was low, as a result of just changing the diffraction angle. The corresponding energy dispersive intensity depth profiles are shown in figures C.4 and C.5 and C.8.
Figure C.1: Intensity depth profiles for an aluminum sample with different shot-peening levels. The x-axis is the position of the center of the probing volume with respect to the sample surface.
Figure C.2 Change in lattice planes spacing versus depth calculated using the intensity change method for various shot-peening levels.
Figure C.3 Intensity depth profiles for 8A shot-peened [111] aluminum at $\theta = 2.562^\circ$ and $\theta = 2.552^\circ$. 
Figure C.4 Energy dispersive intensity depth profiles for 8A shot-peened [111] aluminum at $\theta = 2.562^\circ$ and $\theta = 2.552^\circ$. 
Figure C.5  Energy dispersive intensity depth profiles for 10A shot-peened [111] aluminum at $\theta = 2.562^\circ$ and $\theta = 2.552^\circ$. 
Figure C.6 Intensity depth profiles for 10A shot-peened [111] aluminum at $\theta = 2.562^\circ$ and $\theta = 2.552^\circ$.

Figure C.7 Intensity depth profiles for 12A shot-peened [111] aluminum at $\theta = 2.562^\circ$ and $\theta = 2.552^\circ$. 
Figure C.8 Energy dispersive intensity depth profiles for 12A shot-peened [111] aluminum at $\theta = 2.562^\circ$ and $\theta = 2.552^\circ$. 
C.2 More strain depth profiles measured using the energy dispersive method

In section 3.8.2 we presented some strain depth profile measurements using the energy dispersive technique. More data will be shown in this section. Figures C.9 C.10 C.11 C.12 C.13 C.14 and C.15 show aluminum [111] EDXRD peak location depth profile for non-peened, 4A, 6A, 8A, 9A, 10A, and 12A shot peening levels respectively. The corresponding strain depth profiles are shown in figures C.16 and C.17.

Figure C.9 Position of aluminum [111] energy-dispersive diffraction peak versus depth for a non-peened aluminum sample.
Figure C.10  Position of aluminum [111] energy-dispersive diffraction peak versus depth for a 4A shot-peened aluminum sample.

Figure C.11  Position of aluminum [111] energy-dispersive diffraction peak versus depth for a 6A shot-peened aluminum sample.
Figure C.12 Position of aluminum [111] energy-dispersive diffraction peak versus depth for a 8A shot-peened aluminum sample.

Figure C.13 Position of aluminum [111] energy-dispersive diffraction peak versus depth for a 9A shot-peened aluminum sample.
Figure C.14  Position of aluminum [111] energy-dispersive diffraction peak versus depth for a 10A shot-peened aluminum sample.

Figure C.15  Position of aluminum [111] energy-dispersive diffraction peak versus depth for a 12A shot-peened aluminum sample.
Figure C.16 Change in lattice planes spacing versus depth calculated using the energy dispersive method for various shot-peening levels.
Figure C.17 Change in lattice planes spacing versus depth calculated using the energy dispersive method for various shot-peening levels.
C.3  More strain depth profiles measured using the angle dispersive method

The angle dispersive strain depth profile measurement technique was discussed in section 3.8.3. Strain depth profiles of 4A shot peened aluminum measured using this technique was presented in that section. In this section we will present more measurements for more shot peening levels. Figures C.18, C.19, C.20, and C.21 show sets of $\theta$-$2\theta$ scans at different depths for 6A, 8A, 10A, and 12A shot peening levels respectively. Strain depth profiles calculated using the data shown in those figures are shown in figure C.22.
(a) XRD peaks at different depths.

(b) Peaks locations at different depths.

Figure C.18  6A peened Al [111] θ-dispersive XRD peaks at different depths.
(a) XRD peaks at different depths.

(b) Peaks locations at different depths.

Figure C.19  8A peened Al [111] $\theta$-dispersive XRD peaks at different depths.
(a) XRD peaks at different depths.

(b) Peaks locations at different depths.

Figure C.20  10A peened Al [111] θ-dispersive XRD peaks at different depths.
Figure C.21 12A peened Al [111] $\theta$-dispersive XRD peaks at different depths.
Figure C.22 Change in lattice planes spacing versus depth calculated using the angle-dispersive diffraction method for various shot-peening levels.
C.4 More data comparing the three different methods

Figures C.23, C.24, C.25, C.26, C.27, and C.28 show comparison between strain depth profiles, obtained using the three different methods discussed in sections 3.8.1 and 3.8.2 and 3.8.3, for 4A 6A 8A 9A 10A and 12A shot peened aluminum samples. Note that the measurements agree within the margins of error calculated as discussed in sections 3.8.1.2 and 3.8.2.1 and 3.8.3.1. Suggested improvements to reduce the uncertainties in the measurements are discussed in section 3.8.4 and section 4.2.
Figure C.23 change in lattice planes spacing for a 4A shot-peened aluminum measured using the three different methods.
Figure C.24 change in lattice planes spacing for a 6A shot-peened aluminum measured using the three different methods.
Figure C.25 change in lattice planes spacing for a 8A shot-peened aluminum measured using the three different methods.
Figure C.26 change in lattice planes spacing for a 9A shot-peened aluminum measured using the intensity change and the energy dispersive methods.
Figure C.27 change in lattice planes spacing for a 10Å shot-peened aluminum measured using the three different methods.
Figure C.28  change in lattice planes spacing for a 12Å shot-peened aluminum measured using the three different methods.
C.5 More data from heat treated samples

In section 3.9 we discussed some residual stress thermal relaxation preliminary data of 6A shot peened aluminum. In this appendix we present similar data for 9A and 12A shot peened samples.

Figure C.29 shows the intensity depth profiles of 9A shot peened aluminum exposed to $300^\circ C$ for various periods of time. The strain depth profiles as measured using the normalized intensity method is shown in figure C.30. The strain profiles as measured using the energy dispersive method are shown in figure C.31. A similar set of data for 12A shot peened aluminum is shown in figures C.32, C.33, and C.34.

We discussed the residual stress thermal relaxation resulting from exposing the same sample to the same heat cycle multiple times. The results for 6A shot peened aluminum data is shown in figures 3.97, 3.98, and 3.99. Similar result for 12A shot peened aluminum sample exposed to multiple cycles of 30 minutes at $300^\circ C$ is shown in figures C.35, C.36, and C.37.
Figure C.29 9A shot-peened Al [111] intensity depth profiles after various exposure times to 300°C. The x-axis is the position of the center of the probing volume with respect to the sample surface.
Figure C.30  $\Delta d_{[111]}$ depth profiles of the heat treated 9A shot-peened aluminum samples in figure C.29 as measured using the intensity change method.
Figure C.31 $\Delta d_{[111]}$ depth profiles of the heat treated 9A shot-peened aluminum samples in figure C.29 as measured using the energy dispersive method.
Figure C.32 12A shot peened Al [111] intensity depth profiles after various exposure times to 300°C. The x-axis is the position of the center of the probing volume with respect to the sample surface.
Figure C.33 \( \Delta d_{[11]} \) depth profiles of the heat treated 12A shot-peened aluminum samples in figure C.32 as measured using the intensity change method.
Figure C.34 $\Delta d_{[111]}$ depth profiles of the heat treated 12A shot-peened aluminum samples in figure C.32 as measured using the energy dispersive method.
Figure C.35  12A shot-peened Al [111] intensity depth profiles after multiple of exposures to 300°C for 30 minutes. The x-axis is the position of the center of the probing volume with respect to the sample surface.
Figure C.36 $\Delta d_{[11]}$ depth profiles of the heat treated 12A shot-peened aluminum samples in figure C.35 as measured using the intensity change method.
Figure C.37  $\Delta d_{[111]}$ depth profiles of the heat treated 12A shot-peened aluminum samples in figure C.35 as measured using the energy method.
APPENDIX D. Data Acquisition and Control Software

This appendix lists part of the data acquisition and control program source code. More details about the functionality of the program can be found in section 3.3. The program is written in Visual C++ 9 .Net. The following programs must be installed on the client machine to run the program:

- Ortec Connection A11-B32 or Maestro MCA emulation software.
- Compumotor Motion Planner or compusrv ActiveX control and motion server.
- Microsoft .Net 3.0 or later.

All try-catch statements are omitted here and should be added around any part of the code that may throw an exception. All of graphical interface and data plotting and display code is not shown here. Please refer to the actual code for a fully functional code.

D.1 Common motion control and MCA functions and variables

```c
String ^ Motor1Min; // Motor 1 minimum software limit
String ^ Motor2Min; // Motor 2 minimum software limit
String ^ Motor3Min; // Motor 3 minimum software limit
String ^ Motor4Min; // Motor 4 minimum software limit
String ^ Motor5Min; // Motor 5 minimum software limit
String ^ Motor6Min; // Motor 6 minimum software limit
String ^ Motor7Min; // Motor 7 minimum software limit
String ^ Motor8Min; // Motor 8 minimum software limit
String ^ Motor1Max; // Motor 1 maximum software limit
String ^ Motor2Max; // Motor 2 maximum software limit
```
String Motor3Max; // Motor 3 maximum software limit
String Motor4Max; // Motor 4 maximum software limit
String Motor5Max; // Motor 5 maximum software limit
String Motor6Max; // Motor 6 maximum software limit
String Motor7Max; // Motor 7 maximum software limit
String Motor8Max; // Motor 8 maximum software limit
String Motor1Name; // Motor 1 display name
String Motor1Factor; // Motor 1 steps per unit
String Motor1V; // Motor 1 maximum speed
String Motor1A; // Motor 1 acceleration
String Motor1Backlash; // Motor 1 minimum backlash value
String Motor1Enabled; // Enable (1) Disable (0) motor 1
int Motor1Axis; // Motor 1 axis number
String Motor1Position; // Motor 1 current position
String Motor1Unit; // Motor 1 unit (mm, cm, degree... etc)
String Motor2Name; // Motor 2 display name
String Motor2Factor; // Motor 2 steps per unit
String Motor2V; // Motor 2 maximum speed
String Motor2A; // Motor 2 acceleration
String Motor2Backlash; // Motor 2 minimum backlash value
String Motor2Enabled; // Enable (1) Disable (0) motor 2
int Motor2Axis; // Motor 2 axis number
String Motor2Position; // Motor 2 current position
String Motor2Unit; // Motor 2 unit (mm, cm, degree... etc)
String Motor3Name; // Motor 3 display name
String Motor3Factor; // Motor 3 steps per unit
String Motor3V; // Motor 3 maximum speed
String Motor3A; // Motor 3 acceleration
String Motor3Backlash; // Motor 3 minimum backlash value
String Motor3Enabled; // Enable (1) Disable (0) motor 3
int Motor3Axis; // Motor 3 axis number
String Motor3Position; // Motor 3 current position
```java
String Motor3Unit; // Motor 3 unit (mm, cm, degree... etc)
String Motor4Name; // Motor 4 display name
String Motor4Factor; // Motor 4 steps per unit
String Motor4V; // Motor 4 maximum speed
String Motor4A; // Motor 4 acceleration
String Motor4Backlash; // Motor 4 minimum backlash value
String Motor4Enabled; // Enable(1) Disable(0) motor 4
int Motor4Axis; // Motor 4 axis number
String Motor4Position; // Motor 4 current position
String Motor4Unit; // Motor 4 unit (mm, cm, degree... etc)
String Motor5Name; // Motor 5 display name
String Motor5Factor; // Motor 5 steps per unit
String Motor5V; // Motor 5 maximum speed
String Motor5A; // Motor 5 acceleration
String Motor5Backlash; // Motor 5 minimum backlash value
String Motor5Enabled; // Enable(1) Disable(0) motor 5
int Motor5Axis; // Motor 5 axis number
String Motor5Position; // Motor 5 current position
String Motor5Unit; // Motor 5 unit (mm, cm, degree... etc)
String Motor6Name; // Motor 6 display name
String Motor6Factor; // Motor 6 steps per unit
String Motor6V; // Motor 6 maximum speed
String Motor6A; // Motor 6 acceleration
String Motor6Backlash; // Motor 6 minimum backlash value
String Motor6Enabled; // Enable(1) Disable(0) motor 6
int Motor6Axis; // Motor 6 axis number
String Motor6Position; // Motor 6 current position
String Motor6Unit; // Motor 6 unit (mm, cm, degree... etc)
String Motor7Name; // Motor 7 display name
String Motor7Factor; // Motor 7 steps per unit
String Motor7V; // Motor 7 maximum speed
String Motor7A; // Motor 7 acceleration
```
String Motor7Backlash; // Motor 7 minimum backlash value
String Motor7Enabled; // Enable (1) Disable (0) motor 7
int Motor7Axis; // Motor 7 axis number
String Motor7Position; // Motor 7 current position
String Motor7Unit; // Motor 7 unit (mm, cm, degree... etc)
String Motor8Name; // Motor 8 display name
String Motor8Factor; // Motor 8 steps per unit
String Motor8V; // Motor 8 maximum speed
String Motor8A; // Motor 8 acceleration
String Motor8Backlash; // Motor 8 minimum backlash value
String Motor8Enabled; // Enable (1) Disable (0) motor 8
int Motor8Axis; // Motor 8 axis number
String Motor8Position; // Motor 8 current position
String Motor8Unit; // Motor 8 unit (mm, cm, degree... etc)
Double Start1D; // One-dimensional scan starting location
Double End1D; // One-dimensional scan end location
Double Step1D; // One-dimensional scan step
Double Start2DX; // Two-dimensional scan x starting location
Double End2DX; // Two-dimensional scan x end location
Double Step2DX; // Two-dimensional scan x step
Double Start2DY; // Two-dimensional scan y starting location
Double End2DY; // Two-dimensional scan y end location
Double Step2DY; // Two-dimensional scan y step
String K6Address; // 6K motion controller IP address on the network
String MyAddress; // This computer IP address on the network
String K6MAC; // MAC address of the 6K controller
Double Intercept; // MCA linear calibration parameter
Double Slope; // MCA linear calibration parameter
Int16 Gain; // Number of active channels in the MCA
Int16 MCARefreshTime; // Display/refresh MCA live/true times every (ms)
Int16 WaitTime; // Time to wait for motion to complete after move order
Double StartT2T; // Angular xrd scan starting angle
Double EndT2T; // Angular xrd scan end angle
Double StepT2T; // Angular xrd scan step angle
int Scan1DMotor; // One-dimensional scan motor axis
int Scan2DXMotor; // Two-dimensional scan x motor axis
int Scan2DYMotor; // Two-dimensional scan y motor axis
int numberOfXSteps; // Number of steps in x direction in 2D scan
int numberOfYSteps; // Number of steps in y direction in 2D scan
array< float > X2DData; //
array< int > Y2DData;
array< float > X1DData1; // Energies (E) read from the MCA in 1D scan
array< float > X1DData2; // All locations (X) in a 1D scan range
array< int > Y1DData1; // Intensity (E) read from the MCA in 1D scan
array< int > Y1DData2; // Integrated intensity of (X) in a 1D scan
array< int > YT2TData1; // Intensity of (E) read from MCA in t−2t scan
array< int > YT2TData2; // Integrated intensity in a t−2t scan at (T)
array< float > XT2TData1; // Energies (E) read from the MCA in t−2t scan
array< float > XT2TData2; // All angular locations (T) in t−2t scan
array< float > XData; // Energies in energy-dispersive data collection
array< int > YData; // Intensities in ED data collection
int Scan2DAxis1; // 2D scan x axis
int Scan2DAxis2; // 2D scan y axis
Boolean MoveToNext; // Indicates whether to move to next step or
// not in a scan when the scan timer ticks
int StepCounter; // Current step count in a scan
int numberOfSteps1D; // Number of steps in a 1D scan
int numberOfStepsT2T; // Number of steps in a t−2t scan
array< int,2 > All1DData; // All energy dispersive data collected in a
// 1D scan. Will be saved in a GRD file.
array< int,2 > AllT2TData; // All energy dispersive data collected in a
// t−2t scan. Will be saved in a GRD file.
array< int,2 > All2DDataSum; // 2D scan all data > grd
array< int,3 > All2DDataEne; // 2D scan all energy dispersive data
int StepCounter1D; // current step number in a 1D scan
int StepCounterT2T; // current step number in a t−2t scan
Boolean MoveToNext1D; // whether to move to next step or not in 1D scan
    // when the scan timer ticks
Boolean MoveToNext1T; // whether to move to next step or not in time scan
    // when the scan timer ticks
Boolean MoveToNextT2T; // whether to move to next step or not in t−2t scan
    // when the scan timer ticks
double YdataTotal; // Temp variable to hold total intensity in a scan
int MinChannel; // ignore MCA channels below this channel
int MaxChannel; // ignore MCA channels above this channel
int TimeScanStepsNumber; // number of steps in time scan
float TimeScanStepLength; // time step length in time scan
array<String^>^ MotorsNames; // List of all motors names
array<String^>^ MotorsUnits; // List of all motors displacement unit
array<float^>^ MotorsFactors; // List of motor steps count in each unit
array<float^>^ MotorsBacklash; // List of all motors minimum backlash
array<float^>^ MotorsRes; // List of steps number in one revolution
    // for all motors
int TimeStepCounter; // current step number in a time scan
String^ TempK6Response; // temp variable to hold current 6K response
Int16 Motor1LastMotionSign; // Motor 1 direction of last move
Int16 Motor2LastMotionSign; // Motor 2 direction of last move
Int16 Motor3LastMotionSign; // Motor 3 direction of last move
Int16 Motor4LastMotionSign; // Motor 4 direction of last move
Int16 Motor5LastMotionSign; // Motor 5 direction of last move
Int16 Motor6LastMotionSign; // Motor 6 direction of last move
Int16 Motor7LastMotionSign; // Motor 7 direction of last move
Int16 Motor8LastMotionSign; // Motor 8 direction of last move
String^ Motor1Soft; // Enable–disable motor 1 soft limits
String^ Motor2Soft; // Enable–disable motor 2 soft limits
String^ Motor3Soft; // Enable–disable motor 3 soft limits
String* Motor4Soft; //Enable/disable motor 4 soft limits
String* Motor5Soft; //Enable/disable motor 5 soft limits
String* Motor6Soft; //Enable/disable motor 6 soft limits
String* Motor7Soft; //Enable/disable motor 7 soft limits
String* Motor8Soft; //Enable/disable motor 8 soft limits
String* Motor1Hard; //Enable/disable motor 1 hard limits
String* Motor2Hard; //Enable/disable motor 2 hard limits
String* Motor3Hard; //Enable/disable motor 3 hard limits
String* Motor4Hard; //Enable/disable motor 4 hard limits
String* Motor5Hard; //Enable/disable motor 5 hard limits
String* Motor6Hard; //Enable/disable motor 6 hard limits
String* Motor7Hard; //Enable/disable motor 7 hard limits
String* Motor8Hard; //Enable/disable motor 8 hard limits
String* Motor1Dir; //Direction of positive motion for motor 1
String* Motor2Dir; //Direction of positive motion for motor 2
String* Motor3Dir; //Direction of positive motion for motor 3
String* Motor4Dir; //Direction of positive motion for motor 4
String* Motor5Dir; //Direction of positive motion for motor 5
String* Motor6Dir; //Direction of positive motion for motor 6
String* Motor7Dir; //Direction of positive motion for motor 7
String* Motor8Dir; //Direction of positive motion for motor 8
String* Motor1Pulse; //Motor 1 pulse duration (see 6K manual)
String* Motor2Pulse; //Motor 2 pulse duration (see 6K manual)
String* Motor3Pulse; //Motor 3 pulse duration (see 6K manual)
String* Motor4Pulse; //Motor 4 pulse duration (see 6K manual)
String* Motor5Pulse; //Motor 5 pulse duration (see 6K manual)
String* Motor6Pulse; //Motor 6 pulse duration (see 6K manual)
String* Motor7Pulse; //Motor 7 pulse duration (see 6K manual)
String* Motor8Pulse; //Motor 8 pulse duration (see 6K manual)
String* Motor1PLIMLVL; //Motor 1 positive limit level (see 6K manual)
String* Motor2PLIMLVL; //Motor 2 positive limit level (see 6K manual)
String* Motor3PLIMLVL; //Motor 3 positive limit level (see 6K manual)
```cpp
public: array<int> * ReadDataFromMCA(AxUMCBILib::AxUCONN2 CONNECTION) //Read current MCA buffer{
    array<int> * MCAData;
    bool IsMCAOpen = false;
    IsMCAOpen = CONNECTION->IsOpen;
    if (!IsMCAOpen)
    {
        CONNECTION->Open(); //open MCA if not opened
    }
```
array<int>^ MCAData;
MCAData = safe_cast<array<int>^>(CONNECTION->GetData(
    MinChannel, MaxChannel - MinChannel - 1));
return MCAData;
}

private: void ReadK6Response()
//******************************************************************************************
//Read current response in 6K buffer
//******************************************************************************************
{
    String^ emp = String::Empty;
    TempK6Response = String::Empty;
    String^ delim = ">\t\v\f\n";
    array<wchar_t>^ delimar = delim->ToCharArray();
    TempK6Response = Com6SrvrNet->ReadResponse();
    TempK6Response = TempK6Response->Trim(delimar);
    if (((TempK6Response->CompareTo(emp) == 0) ||
         (TempK6Response->CompareTo(">") == 0) ||
         (TempK6Response->Length <= 1))
    {
        TempK6Response = String::Empty;
        return;
    }
    if (MotorsStatusBox->Text->Length > 1000)
    {
        MotorsStatusBox->Text = String::Empty;
    }
    MotorsStatusBox->Text = String::Concat(">",
        TempK6Response, "\n", MotorsStatusBox->Text);
}

private: void InitializeMCA(AxUMCBILib::AxUCONN^ CONNECTION,
    int MyGain, int MyMinChannel, int MyMaxChannel, int MyMCAOffset)
I n i t i a l i z e M C A  b e f o r e s t a r t i n g a s c a n
{
    if (CONNECTION->IsOpen)
    {
        CONNECTION->Comm( "STOP" );
    }
    if (CONNECTION->IsOpen)
    {
        CONNECTION->Close();
    }
    CONNECTION->Open();
    catch (Exception *)
    {
        CONNECTION->Comm( String::Concat("SET_GAIN_CONVERSION", Convert::ToString(MyGain)));
        CONNECTION->Comm("SET_GATE_ANTICOINCIDENT");
        CONNECTION->Comm( String::Concat("SET_LLD", Convert::ToString(MyMinChannel)));
        CONNECTION->Comm( String::Concat("SET_ULD", Convert::ToString(MyMaxChannel)));
        if (! (CONNECTION->Type)->Contains("927"))
        {
            CONNECTION->Close();
            CONNECTION->Comm( String::Concat("SET_OFFSET_FINE", Convert::ToString(MyMCAOffset)));
        }
    }
    CONNECTION->Close();
}

v o i d  C o m 6 S r v r  N e t S t o p N o w ( v o i d )
// F o r c e i m m e d i a t e s t o p o f a l l c u r r e n t l y m o v i n g a x e s
{
    K6Command = "$S:"
}
int Com6SrvrNetGoTo(int Axis, float Distance, bool ReturnImmediately)

// instruct Axis to go to a position
{
    K6Command = String::Empty;
    K6Command = "DRIVE";
    K6Command = String::Concat(K6Command, Motor1Enabled,
            Motor2Enabled, Motor3Enabled, Motor4Enabled, Motor5Enabled,
            Motor6Enabled, Motor7Enabled, Motor8Enabled, ":" );
    Com6SrvrNet->WriteCmd(K6Command);
    ReadK6Response();
    MotorsPositionTimer->Enabled = true;
    UpdateMotorsPositionsTimer->Enabled = true;
    K6Command = "MA11111111: ";
    Com6SrvrNet->WriteCmd(K6Command);
    ReadK6Response();
    if (Axis == 1)
    {
        // if Axis 1
        K6Command = String::Concat("D",
                Convert::ToString(Distance),"","","","":" ");
        Com6SrvrNet->WriteCmd(K6Command);
        ReadK6Response();
        K6Command = "GO10000000: ";
        Com6SrvrNet->WriteCmd(K6Command);
        ReadK6Response();
    }
    if (Axis == 2)
    {
    }
// if Axis 2
K6Command = String::Concat("D,",
        Convert::ToString(Distance),",",",":");
Com6SrvrNet->WriteCmd(K6Command);
ReadK6Response();
K6Command = "GO01000000:";
Com6SrvrNet->WriteCmd(K6Command);
ReadK6Response();
}
if (Axis == 3)
{

    // if Axis 3
K6Command = String::Concat("D,,",
        Convert::ToString(Distance),",",",":");
Com6SrvrNet->WriteCmd(K6Command);
ReadK6Response();
K6Command = "GO00100000:";
Com6SrvrNet->WriteCmd(K6Command);
ReadK6Response();
}
if (Axis == 4)
{

    // if Axis 4
K6Command = String::Concat("D,,",
        Convert::ToString(Distance),",",":");
Com6SrvrNet->WriteCmd(K6Command);
ReadK6Response();
K6Command = "GO00010000:";
Com6SrvrNet->WriteCmd(K6Command);
ReadK6Response();
}
if (Axis == 5)
if Axis 5
K6Command = String::Concat("D,,," ,
Convert::ToString(Distance),",," );
Com6SrvrNet->WriteCmd(K6Command);
ReadK6Response();
K6Command = "GO00001000:" ;
Com6SrvrNet->WriteCmd(K6Command);
ReadK6Response();
}
if (Axis == 6)
{
// if Axis 6
K6Command = String::Concat("D,,," ,
Convert::ToString(Distance),",,:" );
Com6SrvrNet->WriteCmd(K6Command);
ReadK6Response();
K6Command = "GO00000100:" ;
Com6SrvrNet->WriteCmd(K6Command);
ReadK6Response();
}
if (Axis == 7)
{
// if Axis 7
K6Command = String::Concat("D,,," ,
Convert::ToString(Distance),"::" );
Com6SrvrNet->WriteCmd(K6Command);
ReadK6Response();
K6Command = "GO00000010:" ;
Com6SrvrNet->WriteCmd(K6Command);
ReadK6Response();
}
if (Axis == 8)
{
    // if Axis 8
    K6Command = String::Concat("D,,", Convert::ToString(Distance), ",");
    Com6SrvcNet->WriteCmd(K6Command);
    ReadK6Response();
    K6Command = "GO00000001:");
    Com6SrvcNet->WriteCmd(K6Command);
    ReadK6Response();
}
// change motion mode
K6Command = "MA1111111:");
Com6SrvcNet->WriteCmd(K6Command);
ReadK6Response();
if (ReturnImmediately)
{
    // disable all drives
    K6Command = "DRIVE0000000:");
    Com6SrvcNet->WriteCmd(K6Command); // Disable All Drives
    ReadK6Response();
    return 1;
}
WaitForMotionToComplete(Axis);
K6Command = "DRIVE0000000:");
Com6SrvcNet->WriteCmd(K6Command); // Disable All Drives
ReadK6Response();
return 0;
}

int MotorGo(int Axis, float Distance, float BackLash, bool ReturnImmediately)
// instruct Axis to move additional distance
{
    if (Distance > 0)
    {
        Com6SrvrNetGo(Axis, Distance, ReturnImmediately);
    }
    if (Distance < 0)
    {
        Com6SrvrNetGo(Axis, Distance - 2 * BackLash, ReturnImmediately);
        Com6SrvrNetGo(Axis, 2 * BackLash, ReturnImmediately);
    }
    return 0;
}

int MotorGoTo(int Axis, float Target, float BackLash, bool ReturnImmediately)
// instruct Axis to move to Target and consider backlash
{
    float PositionNow = Com6SrvrNet->MotorPos(Axis)
        / *MotorsFactors[Axis-1];
    if ((Target - PositionNow) > 0)
    {
        Com6SrvrNetGoTo(Axis, Target, ReturnImmediately);
        return 0;
    } else if ((Target - PositionNow) < 0)
    {
        Com6SrvrNetGoTo(Axis, Target - 2 * BackLash, ReturnImmediately);
        Com6SrvrNetGoTo(Axis, Target, ReturnImmediately);
    }
    return 0;
}
```c
void Initialize6K(AxCom6srvrControl::AxCom6srvr6kNet* Com6Srvr)
// Initialize 6K controller when the program starts
{
    K6Command = "@DRIVE0:";
    Com6Srvr->WriteCmd(K6Command); // Disable All Drives
    ReadK6Response();
    K6Command = "CMDDIR";
    K6Command = String::Concat(K6Command,
                                Motor1Dir, Motor2Dir, Motor3Dir, Motor4Dir,
                                Motor5Dir, Motor6Dir, Motor7Dir, Motor8Dir, ":" );
    Com6Srvr->WriteCmd(K6Command);
    ReadK6Response();
    K6Command = "@AXSDEF0:";
    Com6Srvr->WriteCmd(K6Command); // Set All Motors to Stepper Motors
    ReadK6Response();
    K6Command = "@DRFLVL1:";
    Com6Srvr->WriteCmd(K6Command);
        // Set Drive Fault Level to 1 on all Drives
    ReadK6Response();
    K6Command = "@DRFEN1:";
    Com6Srvr->WriteCmd(K6Command); // Enable Checking the Drive Fault Input
    ReadK6Response();
    K6Command = String::Empty;
    K6Command = "DRES";
    K6Command = String::Concat(K6Command,
                                Convert::ToString(MotorsRes[0]), "," ,
                                Convert::ToString(MotorsRes[1]), "," ,
                                Convert::ToString(MotorsRes[2]), "," ,
                                Convert::ToString(MotorsRes[3]), "," ,
                                Convert::ToString(MotorsRes[4]), "," ,
                                Convert::ToString(MotorsRes[5]), "," ,
```
Convert::ToString(MotorsRes[6]), ",";
Convert::ToString(MotorsRes[7]), ":";
Com6Srvr->WriteCmd(K6Command);  // Set drives resolution
ReadK6Response();
K6Command = String::Empty;
K6Command = "SCALE1:";
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = String::Empty;
K6Command = "SCLA";
K6Command = String::Concat(K6Command,
Convert::ToString(MotorsRes[0]), ",";
Convert::ToString(MotorsRes[1]), ",";
Convert::ToString(MotorsRes[2]), ",";
Convert::ToString(MotorsRes[3]), ",";
Convert::ToString(MotorsRes[4]), ",";
Convert::ToString(MotorsRes[5]), ",";
Convert::ToString(MotorsRes[6]), ",";
Convert::ToString(MotorsRes[7]), ":");
Com6Srvr->WriteCmd(K6Command);  // scale acceleration
ReadK6Response();
K6Command = String::Empty;
K6Command = "SCLV";
K6Command = String::Concat(K6Command,
Convert::ToString(MotorsRes[0]), ",";
Convert::ToString(MotorsRes[1]), ",";
Convert::ToString(MotorsRes[2]), ",";
Convert::ToString(MotorsRes[3]), ",";
Convert::ToString(MotorsRes[4]), ",";
Convert::ToString(MotorsRes[5]), ",";
Convert::ToString(MotorsRes[6]), ",";
Convert::ToString(MotorsRes[7]), ":");
Com6Srvr->WriteCmd(K6Command); // scale velocity
ReadK6Response();
K6Command = String::Empty;
K6Command = "SCLD";
K6Command = String::Concat(K6Command,
    Convert::ToString(MotorsFactors[0]),",",,
    Convert::ToString(MotorsFactors[1]),",",,
    Convert::ToString(MotorsFactors[2]),",",,
    Convert::ToString(MotorsFactors[3]),",",,
    Convert::ToString(MotorsFactors[4]),",",,
    Convert::ToString(MotorsFactors[5]),",",,
    Convert::ToString(MotorsFactors[6]),",",,
    Convert::ToString(MotorsFactors[7]),":")
Com6Srvr->WriteCmd(K6Command); // scale distance
ReadK6Response();
K6Command = String::Empty;
K6Command = "@MC0:";
Com6Srvr->WriteCmd(K6Command); // Preset Mode
ReadK6Response();
K6Command = "@MA1:";
Com6Srvr->WriteCmd(K6Command); // Absolute Position Mode
ReadK6Response();
K6Command = "LH"; // Hard Limits
K6Command = String::Concat(K6Command,
    Motor1Hard,"","Motor2Hard","","Motor3Hard","",".
    Motor4Hard","","Motor5Hard","","Motor6Hard","",
    Motor7Hard,"","Motor8Hard":"")
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = "@LHAD100:"; // set deceleration to limit
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = "@LHADA50: ";
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = "LIMLVL";
K6Command = String::Concat(K6Command,
Motor1PLIMLVL,Motor1NLIMLVL,Motor1HLIMLVL,
Motor2PLIMLVL,Motor2NLIMLVL,Motor2HLIMLVL,
Motor3PLIMLVL,Motor3NLIMLVL,Motor3HLIMLVL,
Motor4PLIMLVL,Motor4NLIMLVL,Motor4HLIMLVL,
Motor5PLIMLVL,Motor5NLIMLVL,Motor5HLIMLVL,
Motor6PLIMLVL,Motor6NLIMLVL,Motor6HLIMLVL,
Motor7PLIMLVL,Motor7NLIMLVL,Motor7HLIMLVL,
Motor8PLIMLVL,Motor8NLIMLVL,Motor8HLIMLVL," : ");
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = String::Empty;
K6Command = "LSNEG";
K6Command = String::Concat(K6Command,
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = String::Empty;
K6Command = "LSPOS";
K6Command = String::Concat(K6Command,
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = String::Empty;
K6Command = "LS";
K6Command = String::Concat(K6Command,
Motor29Soft," , ",Motor30Soft" );
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = "@HOMA5: ";
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = "PULSE";
K6Command = String::Concat(K6Command,
    Motor1Pulse,"","Motor2Pulse","","Motor3Pulse","","Motor4Pulse","",
    "Motor5Pulse","","Motor6Pulse","","Motor7Pulse","","Motor8Pulse":"" );
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = "@HOMA5A: ";
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = "@HOMAD5: ";
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = "@HOMADA5: ";
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = "@HOMBAC1: ";
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = "@HOMDF0: ";
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = "@HOMECD0: ";
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = "@HOMV5: ";
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = "@HOMVF1:";
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
Com6Srvr->FsEnabled = false;
Com6Srvr->FsUpdateRate = 200;
Com6Srvr->FsEnabled = true;
K6Command = String::Empty;
K6Command = "DRIVE";
K6Command = String::Concat(K6Command,
                   Motor1Enabled, Motor2Enabled, Motor3Enabled, Motor4Enabled,
                   Motor5Enabled, Motor6Enabled, Motor7Enabled, Motor8Enabled, ":");
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = String::Empty;
K6Command = "V";
K6Command = String::Concat(K6Command,
                   Motor1V, "", Motor2V, "", Motor3V, "", Motor4V, "", Motor5V, "", Motor6V, "", Motor7V, "", Motor8V, ":");
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = String::Empty;
K6Command = "PSET";
K6Command = String::Concat(K6Command,
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = String::Empty;
K6Command = "A";
K6Command = String::Concat(K6Command,
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = String::Empty; // Set Motors Acceleration
String Motor1AA = Convert::ToString(
    Math::Round(Convert::ToSingle(Motor1A) / 2.0, 2));
String Motor2AA = Convert::ToString(
    Math::Round(Convert::ToSingle(Motor2A) / 2.0, 2));
String Motor3AA = Convert::ToString(
    Math::Round(Convert::ToSingle(Motor3A) / 2.0, 2));
String Motor4AA = Convert::ToString(
    Math::Round(Convert::ToSingle(Motor4A) / 2.0, 2));
String Motor5AA = Convert::ToString(
    Math::Round(Convert::ToSingle(Motor5A) / 2.0, 2));
String Motor6AA = Convert::ToString(
    Math::Round(Convert::ToSingle(Motor6A) / 2.0, 2));
String Motor7AA = Convert::ToString(
    Math::Round(Convert::ToSingle(Motor7A) / 2.0, 2));
String Motor8AA = Convert::ToString(
    Math::Round(Convert::ToSingle(Motor8A) / 2.0, 2));
K6Command = String::Empty;
K6Command = "AA";
K6Command = String::Concat(K6Command,
    Motor1AA, Motor2AA, Motor3AA, Motor4AA, Motor5AA, Motor6AA, Motor7AA, Motor8AA);
Com6Srvr->WriteCmd(K6Command);
ReadK6Response();
K6Command = String::Empty;
K6Command = "AD";
K6Command = String::Concat(K6Command,
```cpp
int Com6SrvrNetGo(int Axis, float Distance, bool ReturnImmediately)
{
    K6Command = String::Empty;
    K6Command = "DRIVE";
    K6Command = String::Concat(K6Command,
        Motor1Enabled, Motor2Enabled, Motor3Enabled, Motor4Enabled,
        Motor5Enabled, Motor6Enabled, Motor7Enabled, Motor8Enabled, ":" );
    Com6SrvrNet->WriteCmd(K6Command);
    ReadK6Response();
    MotorsPositionTimer->Enabled = true;
    UpdateMotorsPositionsTimer->Enabled = true;
    K6Command = "MA00000000:";
    Com6SrvrNet->WriteCmd(K6Command);
    ReadK6Response();
    if (Axis == 1)
    {
        K6Command = String::Concat("D", Convert::ToString(Distance)," ", ",", ", ", ",":");
    }
```
Com6SrvrNet->WriteCmd(K6Command);
ReadK6Response();
K6Command = "GO10000000:"
Com6SrvrNet->WriteCmd(K6Command);
ReadK6Response();
}

if (Axis == 2)
{
   K6Command = String::Concat("D, ", Convert::ToString(Distance), ", , , , :");
   Com6SrvrNet->WriteCmd(K6Command);
   ReadK6Response();
   K6Command = "GO01000000:");
   Com6SrvrNet->WriteCmd(K6Command);
   ReadK6Response();
}

if (Axis == 3)
{
   K6Command = String::Concat("D, , ", Convert::ToString(Distance), ", , , , :");
   Com6SrvrNet->WriteCmd(K6Command);
   ReadK6Response();
   K6Command = "GO00100000:");
   Com6SrvrNet->WriteCmd(K6Command);
   ReadK6Response();
}

if (Axis == 4)
{
   K6Command = String::Concat("D, , , ", Convert::ToString(Distance), ", , , , :");
   Com6SrvrNet->WriteCmd(K6Command);
   ReadK6Response();
K6Command = " GO00010000: ";
Com6SrvrNet->WriteCmd(K6Command);
ReadK6Response();
}
if (Axis == 5)
{
    K6Command = String::Concat("D,,", Convert::ToString(Distance),",,");
    Com6SrvrNet->WriteCmd(K6Command);
    ReadK6Response();
    K6Command = " GO00001000: ";
    Com6SrvrNet->WriteCmd(K6Command);
    ReadK6Response();
}
if (Axis == 6)
{
    K6Command = String::Concat("D,,", Convert::ToString(Distance),",",":");
    Com6SrvrNet->WriteCmd(K6Command);
    ReadK6Response();
    K6Command = " GO00000100: ";
    Com6SrvrNet->WriteCmd(K6Command);
    ReadK6Response();
}
if (Axis == 7)
{
    K6Command = String::Concat("D,,", Convert::ToString(Distance),",":");
    Com6SrvrNet->WriteCmd(K6Command);
    ReadK6Response();
    K6Command = " GO00000010: ";
    Com6SrvrNet->WriteCmd(K6Command);
ReadK6Response();

if (Axis == 8)
{
    K6Command = String::Concat("D,\ldots," , Convert::ToString(Distance), ",");
    Com6SrvrNet->WriteCmd(K6Command);
    ReadK6Response();
    K6Command = "GO00000001:"
    Com6SrvrNet->WriteCmd(K6Command);
    ReadK6Response();
}

K6Command = "MA1111111:"
Com6SrvrNet->WriteCmd(K6Command);
ReadK6Response();
if (ReturnImmediately)
{
    // MotorsPositionTimer->Enabled = false;
    // UpdateMotorsPositionsTimer->Enabled = false;
    K6Command = "DRIVE00000000:");
    Com6SrvrNet->WriteCmd(K6Command); // Disable All Drives
    ReadK6Response();
    return 1;
}
WaitForMotionToComplete(Axis);
K6Command = "DRIVE00000000:"
Com6SrvrNet->WriteCmd(K6Command); // Disable All Drives
ReadK6Response();
return 0;
D.2 Energy-dispersive data collection

```csharp
private: void StartEDCollection_Click (System::Object^, System::EventArgs^)
{
    if (EnergyDispersiveTimer->Enabled == true) return;
    InitializeMCA (MCAEnergy->CONNECTION, Gain, MinChannel, MaxChannel, 0);
    MCAEnergy->InitializeMCA ();
    if (MCAEnergy->CONNECTION->Address == String::Empty) return;
    XData = gcnew array<float>(MaxChannel - MinChannel + 1);
    YData = gcnew array<int>(MaxChannel - MinChannel + 1);
    Array::Clear (XData, 0, XData->Length);
    Array::Clear (YData, 0, YData->Length);
    MCAEnergy->CONNECTION->Open ();
    int IndexCounter = 0;
    for (IndexCounter = XData->GetLowerBound (0);
        IndexCounter <= XData->GetUpperBound (0); IndexCounter++)
    {
        XData->SetValue ((Intercept + (IndexCounter + MinChannel) * Slope), IndexCounter);
    }
    CreateAndDisplayPlotWindows ();
    PlotForm->Show ();
    PlotForm->XYPlot->SetXScale (1.0f * Convert::ToInt32 (Intercept +
        MinChannel * Slope - 1), 1.0f * Convert::ToInt32 (Intercept +
        MaxChannel * Slope + 1), 1.0f * Convert::ToSingle (GetStep (Intercept +
        (MaxChannel - MinChannel) * Slope / 10)));
    GC::Collect ();
    EnergyDispersiveTimer->Interval = MCARefreshTime;
    MCAEnergy->CONNECTION->Open ();
    MCAEnergy->CONNECTION->Comm ("START");
```
private: void EDTimer_Tick(System::Object *, System::EventArgs *)
{
    if (MCAEnergy->CONNECTION->IsActive != false)
    {
        (ReadDataFromMCA(MCAEnergy->CONNECTION))->CopyTo(YData, 0);
        MCAMotionLiveBox->Text = Convert::ToString(Math::Round((Convert::ToSingle((
            Convert::ToString(MCAEnergy->CONNECTION->Comm("SHOW_LIVE")))
            ->Remove(0, 2))
            ->Remove(10, 3)/50), 1));
        MCAMotionTrueBox->Text = Convert::ToString(Math::Round((
            Convert::ToSingle(((Convert::ToString(MCAEnergy->CONNECTION->Comm("SHOW_TRUE")))
            ->Remove(0, 2))
            ->Remove(10, 3)/50), 1));
        if (PlotForm4->IsDisposed == false)
            PlotForm4->XYPlot->LoadPlotFromArray(XData, YData, Color::Blue, "I vs E");
    }
    if (MCAEnergy->CONNECTION->IsActive == false)
    {
        EnergyDispersiveTimer->Enabled = false;
        (ReadDataFromMCA(MCAEnergy->CONNECTION))->CopyTo(YData, 0);
        if (PlotForm4->IsDisposed == false)
            PlotForm4->XYPlot->LoadPlotFromArray(XData, YData, Color::Blue, "I vs E");
    }
}
D.3 One-dimensional energy-dispersive scan

```csharp
private: void Start1DScanButton_Click(System::Object^, System::EventArgs^)
{
    if (Scan1DTimer->Enabled == true) return;
    MoveToNext1D = false;
    InitializeMCA(MCAID->CONNECTION, Gain, MinChannel, MaxChannel, 0);
    MCAID->InitializeMCA();
    if (MCAID->CONNECTION->Address == String::Empty) return;
    numberOfSteps1D = 1;
    Start1D = Convert::ToDouble(Start1DBox->Text);
    End1D = Convert::ToDouble(End1DBox->Text);
    Scan1DMotor = Select1DMotorCombo->SelectedIndex + 1;
    Step1D = Convert::ToDouble(Step1DBox->Text);
    Step1D = Convert::ToDouble(Math::Floor((Step1D *
    *MotorsFactors[Scan1DMotor - 1]) / (Step1D *
    *MotorsFactors[Scan1DMotor - 1])));
    Step1DBox->Text = Convert::ToString(Step1D);
    numberOfSteps1D = Convert::ToInt32(Math::Abs((End1D -
    Start1D) / Step1D));
    if (End1D <= Start1D) return;
    All1DData = gcnew array<int, 2>(Math::Abs(numberOfSteps1D) + 1,
    MaxChannel - MinChannel + 1);
    for (int i = 0; i < All1DData->GetLength(0); i++)
    {
        for (int j = 0; j < All1DData->GetLength(1); j++)
        {
            All1DData->SetValue(Convert::ToInt32(0.0f), i, j);
        }
    }
    X1DData1 = gcnew array<float>(MaxChannel - MinChannel + 1);
    Y1DData1 = gcnew array<int>(MaxChannel - MinChannel + 1);
```
X1DData2 = gcnew array<float.Wrap>(Math::Abs(numberOfSteps1D)+1);
Y1DData2 = gcnew array<int.Wrap>(Math::Abs(numberOfSteps1D)+1);
Array::Clear(X1DData1,0,X1DData1->Length);
Array::Clear(Y1DData1,0,Y1DData1->Length);
Array::Clear(X1DData2,0,X1DData2->Length);
Array::Clear(Y1DData2,0,Y1DData2->Length);
MCA1D->CONNECTION->Open();
MCA1D->CONNECTION->Comm("CLEAR");
StepCounter1D = 0;
Scan1DMotor = Select1DMotorCombo->SelectedIndex + 1;
MotorGoTo(Scan1DMotor,Convert::ToSingle(Start1D),
*MotorsBacklash[Scan1DMotor-1],false);
int IndexCounter = 0;
float ftemp;
for ( IndexCounter = 0 ; IndexCounter <= MaxChannel
-MinChannel ; IndexCounter++ )
{
    ftemp = (float)(Intercept+(IndexCounter+MinChannel)*Slope);
    X1DData1->SetValue(ftemp,IndexCounter);
}
PlotForm2->XYPlot->SetXScale((float)(Math::Min(Start1D,End1D)),(float)(Math::Max(Start1D,End1D))
,(float)(GetStep(Math::Abs(End1D-Start1D)/10.0f)));
PlotForm1->XYPlot->SetXScale((float)Convert::ToInt32(Intercept
+(MinChannel-1)*Slope),(float)Convert::ToInt32(Intercept
+(MaxChannel+1)*Slope),(float)(GetStep(Intercept
+(MaxChannel-MinChannel)*Slope/10)));
for (int i=0; i <= numberOfSteps1D; i++)
{
    float x = Convert::ToSingle(Start1D + i * Step1D);
    X1DData2->SetValue(x,i);
}
GC::Collect();
MoveToNext1D = false;
Scan1DProgressBar->Minimum = 0;
Scan1DProgressBar->Value = 0;
Scan1DProgressBar->Maximum = Math::Abs(numberOfSteps1D) + 1;
Scan1DTimer->Interval = MCARefreshTime;
MCA1D->CONNECTION->Open();
MCA1D->CONNECTION->Comm("START");
Scan1DTimer->Enabled = true;
}

private: void Scan1DTimer_Tick(System::Object^, System::EventArgs^)
{
    Double CurrentPosition;
    Scan1DProgressBar->Value = StepCounter1D;
    (ReadDataFromMCA(MCA1D->CONNECTION))->CopyTo(Y1DData1, 0);
    if (PlotForm1->IsDisposed == false)
        PlotForm1->XYPlot->LoadPlotFromArray(X1DData1, Y1DData1,
            Color::Blue, "I_vs_E");
    MCAMotionLiveBox->Text =
        Convert::ToString(Math::Round((Convert::ToSingle((
            Convert::ToString(MCA1D->CONNECTION->Comm("SHOW_LIVE")))
        )->Remove(0, 2) -- > Remove(10, 3)/50), 1));
    MCAMotionTrueBox->Text =
        Convert::ToString(Math::Round((Convert::ToSingle((
            Convert::ToString(MCA1D->CONNECTION->Comm("SHOW_TRUE")))
        )->Remove(0, 2) -- > Remove(10, 3)/50), 1));
    if (MoveToNext1D == true)
    {
        (ReadDataFromMCA(MCA1D->CONNECTION))->CopyTo(Y1DData1, 0);
        MoveToNext1D = false;
        MCA1D->CONNECTION->Comm("STOP");
    }
YdataTotal = 0;
int dtemp;
for (int index1 = 0; index1 < (MaxChannel - MinChannel + 1); index1++)
{
    dtemp = Convert::ToInt32(Y1DData1->GetValue(index1));
    All1DData->SetValue(dtemp, StepCounter1D - 1, index1);
    YdataTotal = YdataTotal + dtemp;
}
Y1DData2->SetValue(Convert::ToInt32(YdataTotal), StepCounter1D - 1);
CurrentPosition = Com6SrvrNet->MotorPos(Scan1DMotor)/
*MotorsFactors[Scan1DMotor - 1];
X1DData2->SetValue(Convert::ToSingle(CurrentPosition),
StepCounter1D - 1);
if (PlotForm1->IsDisposed == false)
    PlotForm1->XYPlot->LoadPlotFromArray(X1DData1, Y1DData1,
Color::Blue,"I vs E");
if (PlotForm2->IsDisposed == false)
    PlotForm2->XYPlot->LoadPlotFromArray(X1DData2,
Y1DData2, Color::Blue,"I vs X");
MCA1D->CONNECTION->Comm("CLEAR");
ReadK6Response();
MotorGo(Scan1DMotor, Math::Sign(End1D -
Start1D)*Convert::ToSingle(Step1D),
*MotorsBacklash[Scan1DMotor - 1].false);
MCA1D->CONNECTION->Comm("START");
}
if (StepCounter1D - 1 >= (numberOfSteps1D))
{
    MoveToNext1D = false;
    MCA1D->CONNECTION->Comm("STOP");
    MCA1D->CONNECTION->Close();
Scan1DTimer->Enabled = false;
Scan1DProgressBar->Value = numberOfSteps1D;
CreateInstancesOfWindows();
PlotForm1->Show();
PlotForm2->Show();
Plot3DForm1->Show();
PlotForm1->XYPlot->LoadPlotFromArray(X1DData1, Y1DData1, Color::Blue, "I vs E");
PlotForm2->XYPlot->LoadPlotFromArray(X1DData2, Y1DData2, Color::Blue, "I vs X");
Plot3DForm1->ThreeDPlotControl->LoadPlotFromArray(All1DData, Convert::ToSingle(Start1DBox->Text), Convert::ToSingle(Step1DBox->Text), Convert::ToSingle(Intercept + MinChannel * Slope), Convert::ToSingle(Slope));
}
if (MCA1D->CONNECTION->IsActive == false)
{
    MoveToNext1D = true;
    StepCounter1D++;
}

D.4 Two-dimensional energy-dispersive scan
private: void Start2DScanButton_Click(System::Object^, System::EventArgs^)
{
    if (Scan2DTimer->Enabled == true) return;
    InitializeMCA(MCA2D->CONNECTION, Gain, MinChannel, MaxChannel, 0);
    MoveToNext1D = false;
    if (MCA2D->CONNECTION->Address == String::Empty) return;
    MCA2D->InitializeMCA();
    Scan2DAxis1 = Select2DXMotorCombo->SelectedIndex + 1;
Scan2DAxis2 = Select2DYMotorCombo->SelectedIndex + 1;
if (Start2DXBox->Text == String::Empty) return;
if (End2DXBox->Text == String::Empty) return;
if (Step2DXBox->Text == String::Empty) return;
if (Start2DYBox->Text == String::Empty) return;
if (End2DYBox->Text == String::Empty) return;
if (Step2DYBox->Text == String::Empty) return;
numberOfXSteps = 1;
numberOfYSteps = 1;
Start2DX = Convert::ToDouble(Convert::ToSingle(Start2DXBox->Text));
End2DX = Convert::ToDouble(Convert::ToSingle(End2DXBox->Text));
Step2DX = Convert::ToDouble(Convert::ToSingle(Step2DXBox->Text));
numberOfXSteps = Convert::ToInt32(Math::Round((End2DX - Start2DX)/Step2DX,0)) + 1;
Start2DY = Convert::ToDouble(Convert::ToSingle(Start2DYBox->Text));
End2DY = Convert::ToDouble(Convert::ToSingle(End2DYBox->Text));
Step2DY = Convert::ToDouble(Convert::ToSingle(Step2DYBox->Text));
numberOfYSteps = Convert::ToInt32(Math::Round((End2DY - Start2DY)/Step2DY,0)) + 1;
Step2DX = Convert::ToDouble(Math::Floor(((Step2DX * MotorsFactors[Scan2DAxis1 - 1])/(+ MotorsFactors[Scan2DAxis1 - 1])));
Step2DXBox->Text = Convert::ToString(Step2DX);
Step2DY = Convert::ToDouble(Math::Floor(((Step2DY * MotorsFactors[Scan2DAxis2 - 1])/(+ MotorsFactors[Scan2DAxis2 - 1])));
Step2DYBox->Text = Convert::ToString(Step2DY);
if (End2DX <= Start2DX) return;
if (End2DY <= Start2DY) return;
X2DData = gcnew array<float>(MaxChannel - MinChannel + 1);
Y2DData = gcnew array<int>(MaxChannel - MinChannel + 1);
All2DDataSum = gcnew array<int,2>(numberOfXSteps,numberOfYSteps);
All2DDataEne = gcnew array<int,3>(numberOfXSteps, numberOfYSteps, MaxChannel - MinChannel + 1);
for (int i = 0; i < All2DDataSum->GetLength(0); i++)
{
    for (int j = 0; j < All2DDataSum->GetLength(1); j++)
    {
        All2DDataSum->SetValue(Convert::ToInt32(0.0f), i, j);
    }
}
Array::Clear(X2DData, 0, X2DData->Length);
Array::Clear(Y2DData, 0, Y2DData->Length);
InitializeMCA(MCA2D->CONNECTION, Gain, MinChannel, MaxChannel, 0);
MCA2D->CONNECTION->Open(); MCA2D->CONNECTION->Comm("CLEAR");
StepCounter = 0;
MotorGoTo(Scan2DAxis2, Convert::ToSingle(Start2DY),
    *MotorsBacklash[Scan2DAxis2 - 1], false);
int IndexCounter = 0;
for (IndexCounter = X2DData->GetLowerBound(0); IndexCounter <= X2DData
    ->GetUpperBound(0); IndexCounter++)
{
    X2DData->SetValue((Intercept + (IndexCounter
        + MinChannel) * Slope), IndexCounter);
}
Plot3DForm2->ThreeDPlotControl->SetXScale(
    Convert::ToSingle(Start2DX), Convert::ToSingle(End2DX),
    Convert::ToSingle(Step2DX));
Plot3DForm2->ThreeDPlotControl->SetYScale(
    Convert::ToSingle(Start2DY),
    Convert::ToSingle(End2DY), Convert::ToSingle(Step2DY));
PlotForm2D->XYPlot->SetXScale(1.0f*Convert::ToInt32(Intercept +
    MinChannel*Slope - 1), 1.0f*Convert::ToInt32(Intercept
    + MaxChannel*Slope + 1), 1.0f*Convert::ToSingle(GetStep(Intercept
    + (MaxChannel - MinChannel)*Slope/10)));
GC::Collect();
Scan2DProgressBar->Minimum = 0;
Scan2DProgressBar->Maximum = numberOfXSteps*numberOfYSteps;
Scan2DTimer->Interval = MCAResetInterval;
MoveToNext = false;
MCA2D->CONNECTION->Open();
Scan2DTimer->Enabled = true;
}

private: void Scan2DTimer_Tick(System::Object ^, System::EventArgs ^)
{
    MCAMotionLiveBox->Text =
        Convert::ToString(Math::Round((Convert::ToSingle((
            Convert::ToString(MCA2D->CONNECTION->Comm("SHOW_LIVE"))
        )
            ->Remove(0,2) -> Remove(10,3)/50,1));
    MCAMotionTrueBox->Text =
        Convert::ToString(Math::Round((Convert::ToSingle((
            Convert::ToString(MCA2D->CONNECTION->Comm("SHOW_TRUE"))
        )
            ->Remove(0,2) -> Remove(10,3)/50,1));
    Double CurrentXPosition, CurrentYPosition;
    Scan2DProgressBar->Value = StepCounter;
    (ReadDataFromMCA(MCA2D->CONNECTION))->CopyTo(Y2DData,0);
    if (PlotForm2D->IsDisposed == false)
    {
        PlotForm2D->XYPlot->RemovePlot("I vs. E");
        PlotForm2D->XYPlot->LoadPlotFromArray(
            X2DData, Y2DData, Color::Green,"I vs. E");
    }
    if (MoveToNext == true)
    {
        (ReadDataFromMCA(MCA2D->CONNECTION))->CopyTo(Y2DData,0);
        MoveToNext = false;
MCA2D->CONNECTION->Comm("STOP");

CurrentXPosition = Com6SrvrNet->MotorPos( Scan2DAxis1 )/ *MotorsFactors[Scan2DAxis1 - 1];
CurrentYPosition = Com6SrvrNet->MotorPos( Scan2DAxis2 )/ *MotorsFactors[Scan2DAxis2 - 1];
if (PlotForm2D->IsDisposed == false)
{
    PlotForm2D->XYPlot->RemovePlot("I vs E");
    PlotForm2D->XYPlot->LoadPlotFromArray
        (X2DData, Y2DData, Color::Green,"I vs E");
}
MCA2D->CONNECTION->Comm("CLEAR");
ReadK6Response();
int TotalI = 0; int Ie = 0;
int ix = MyMod(StepCounter,numberOfXSteps);
int iy = StepCounter/numberOfXSteps;
for (int P = Y2DData->GetLowerBound(0); P <= Y2DData->GetUpperBound(0); P++)
{
    Ie = Convert::ToInt32(Y2DData->GetValue(P));
    TotalI = TotalI + Ie;
    All2DDataEne->SetValue(Ie, ix, iy, P);
}
All2DDataSum->SetValue(TotalI, ix, iy);
StepCounter++;
if (((StepCounter * 1.0)/numberOfXSteps) ==
    Math::Round(((StepCounter * 1.0)/numberOfXSteps), 0))
{
    MotorGo(Scan2DAxis2, Convert::ToSingle(
        Step2DYBox->Text), *MotorsBacklash[Scan2DAxis2 - 1], false);
    MotorGoTo(Scan2DAxis1, Convert::ToSingle(
        Start2DXBox->Text), *MotorsBacklash[Scan2DAxis1 - 1], false);
if (((StepCounter * 1.0)/numberOfXSteps) != Math::Round(((StepCounter * 1.0)/numberOfXSteps), 0)) {
    MotorGo(Scan2DAxis1, Convert::ToSingle(Step2DXBox->Text), *MotorsBacklash[Scan2DAxis1 - 1], false);
} MCA2D->CONNECTION->Comm("START");
}

if (MCA2D->CONNECTION->IsActive == false) {
    MoveToNext = true;
}

if ((StepCounter + 1) >= numberOfXSteps*numberOfYSteps) {
    MoveToNext = false;
    MCA2D->CONNECTION->Comm("STOP");
    MCA2D->CONNECTION->Close();
    Scan2DTimer->Enabled = false;
    Scan2DProgressBar->Value = numberOfXSteps*numberOfYSteps;
    Plot3DForm2->ThreeDPlotControl->LoadPlotFromArray(All2DDataSum, Convert::ToSingle(Start2DX), Convert::ToSingle(Step2DX), Convert::ToSingle(Start2DY), Convert::ToSingle(Step2DY));
}

D.5 Angular energy-dispersive XRD scan

private: void StartT2TScan_Click(System::Object^, System::EventArgs^) {
    if (ScanT2TTimer->Enabled == true) return;
MoveToNextT2T = false;
InitializeMCA(MCAT2T->CONNECTION, Gain, MinChannel, MaxChannel, 0);
MCAT2T->InitializeMCA();
if (MCAT2T->CONNECTION->Address == String::Empty) return;
numberOfStepsT2T = 1;
StartT2T = Convert::ToDouble(StartT2TBox->Text);
EndT2T = Convert::ToDouble(EndT2TBox->Text);
StepT2T = Convert::ToDouble(StepT2TBox->Text);
StepT2T = Convert::ToDouble(
Math::Floor((StepT2T * *MotorsFactors[Motor2Axis - 1]))/*MotorsFactors[Motor2Axis - 1])));
StepT2TBox->Text = Convert::ToString(StepT2T);
numberOfStepsT2T = 1 + Convert::ToUint((EndT2T - StartT2T)/StepT2T);
if (EndT2T <= StartT2T) return;
AllT2TData = gcnew array<int, 2>(numberOfStepsT2T, MaxChannel - MinChannel + 1);
for (int i = 0; i < AllT2TData->GetLength(0); i++)
{
    for (int j = 0; j < AllT2TData->GetLength(1); j++)
    {
        AllT2TData->SetValue(Convert::ToInt32(0.0f), i, j);
    }
}
XT2TData1 = gcnew array<float>(MaxChannel - MinChannel + 1);
YT2TData1 = gcnew array<int>(MaxChannel - MinChannel + 1);
XT2TData2 = gcnew array<float>(numberOfStepsT2T);
YT2TData2 = gcnew array<int>(numberOfStepsT2T);
Array::Clear(XT2TData1, 0, XT2TData1->Length);
Array::Clear(YT2TData1, 0, YT2TData1->Length);
Array::Clear(XT2TData2, 0, XT2TData2->Length);
Array::Clear(YT2TData2, 0, YT2TData2->Length);
StepCounterT2T = 0;
if (Motor1Axis < 1) return; // Two Theta
if (Motor2Axis < 1) return; // Theta
if (Motor4Axis < 1) return; // Det Linear
if (Motor5Axis < 1) return; // Det Rotary
MotorGoTo(Motor1Axis, Convert::ToSingle(StartT2T * 2), *MotorsBacklash[Motor1Axis - 1], false);
MotorGoTo(Motor2Axis, Convert::ToSingle(StartT2T), *MotorsBacklash[Motor2Axis - 1], false);
int IndexCounter = 0;
for (IndexCounter = XT2TData1->GetLowerBound(0);
    IndexCounter <= XT2TData1->GetUpperBound(0); IndexCounter++)
{
    XT2TData1->SetValue((Intercept + (IndexCounter + MinChannel) * Slope), IndexCounter);
}
PlotFormT2T2->XYPlot->SetXScale(Convert::ToSingle(StartT2T),
    Convert::ToSingle(EndT2T), Convert::ToSingle(GetStep((EndT2T
    - StartT2T) / 10)));
PlotFormT2T1->XYPlot->SetXScale(1.0f*Convert::ToInt32(Intercept +
    MinChannel*Slope - 1), 1.0f*Convert::ToInt32(Intercept +
    MaxChannel*Slope + 1), 1.0f*Convert::ToSingle(GetStep(Intercept +
    (MaxChannel - MinChannel)*Slope / 10)));
for (int i = 0; i < numberOfStepsT2T; i++)
{
    float x = Convert::ToSingle(StartT2T + i * StepT2T);
    XT2TData2->SetValue(x, i);
}
GC::Collect();
ScanT2TProgressBar->Minimum = 0;
ScanT2TProgressBar->Maximum = numberOfStepsT2T;
ScanT2TTimer->Interval = MCAResfreshTime;
MoveToNextT2T = false;
private: void ScanT2TTimer_Tick(System::Object^, System::EventArgs^) {
    Single CurrentThetaPosition;
    ScanT2TProgressBar->Value = StepCounterT2T;
    (ReadDataFromMCA(MCAT2T->CONNECTION))->CopyTo(YT2TData1, 0);
    if (PlotFormT2T1->IsDisposed == false)
    {
        PlotFormT2T1->XYPlot->LoadPlotFromArray(XT2TData1, YT2TData1, Color::Blue, "I vs E");
    }
    MCAMotionLiveBox->Text = Convert::ToString(Math::Round((Convert::ToSingle(((Convert::ToSingle(MCAT2T->CONNECTION->Comm("SHOW_LIVE")))->Remove(0, 2))->Remove(10, 3))/50), 1));
    MCAMotionTrueBox->Text = Convert::ToString(Math::Round((Convert::ToSingle(((Convert::ToSingle(MCAT2T->CONNECTION->Comm("SHOW_TRUE")))->Remove(0, 2))->Remove(10, 3))/50), 1));
    if (MoveToNextT2T == true)
    {
        (ReadDataFromMCA(MCAT2T->CONNECTION))->CopyTo(YT2TData1, 0);
        MoveToNextT2T = false;
        MCAT2T->CONNECTION->Comm("STOP");
        YdataTotal = 0;
        int dtemp;
        for (int index1 = 0; index1 < (MaxChannel
- MinChannel + 1) ; index1++)
{
    dtemp = Convert::ToInt32(YT2TData1->GetValue(index1));
    AllT2TData->SetValue(dtemp, StepCounterT2T, index1);
    YdataTotal = YdataTotal + dtemp;
}
YT2TData2->SetValue(YdataTotal, StepCounterT2T);
CurrentThetaPosition = Com6SrvrNet->MotorPos(Motor2Axis)/(*MotorsFactors[Motor2Axis - 1]);
XT2TData2->SetValue((CurrentThetaPosition), StepCounterT2T);
StepCounterT2T++;
if (PlotFormT2T1->IsDisposed == false) PlotFormT2T1->XYPlot->LoadPlotFromArray(XT2TData1, YT2TData1, Color::Blue,"I vs E");
if (PlotFormT2T2->IsDisposed == false) PlotFormT2T2->XYPlot->LoadPlotFromArray(XT2TData2, YT2TData2, Color::Blue,"I vs Theta");
MCAT2T->CONNECTION->Comm("CLEAR");
Com6SrvrNetStop();
MotorGoTo(Motor1Axis, 2* Convert::ToSingle((StartT2T + StepCounterT2T*StepT2T)), *MotorsBacklash[Motor1Axis - 1], false);
MotorGoTo(Motor2Axis, Convert::ToSingle((StartT2T + StepCounterT2T*StepT2T)), *MotorsBacklash[Motor2Axis - 1], false);
MCAT2T->CONNECTION->Comm("START");
}
if (MCAT2T->CONNECTION->IsActive == false)
{
    MoveToNextT2T = true;
}
if (StepCounterT2T >= numberOfStepsT2T)
{
    MoveToNextT2T = false;
MCAT2T->CONNECTION->Comm("STOP");
CreateInstancesOfWindows();
PlotFormT2T2->Show();
MCAT2T->CONNECTION->Close();
ScanT2TTimer->Enabled = false;
ScanT2TProgressBar->Value = numberOfStepsT2T;
PlotFormT2T2->XYPlot->LoadPlotFromArray(XT2TData2, YT2TData2, Color::Blue, "I\_\_vs\_\_Theta");
Plot3DForm3->ThreeDPlotControl->LoadPlotFromArray(AllT2TData, Convert::ToSingle(StartT2TBox->Text), Convert::ToSingle(StepT2TBox->Text), Convert::ToSingle(Intercept + MinChannel * Slope), Convert::ToSingle(Slope));
}
}

D.6 Energy-dispersive time scan

private: void StartTimeScan_Click(System::Object^, System::EventArgs^)
{
    if (TimeScanTimer->Enabled == true) return;
    if (String::IsNullOrEmpty(TimeScanStepsBox->Text)) return;
    if ((Convert::ToInt32(TimeScanStepsBox->Text) <= 0)) return;
    InitializeMCA(MCATime->CONNECTION, Gain, MinChannel, MaxChannel, 0);
    if (MCATime->CONNECTION->Address == String::Empty) return;
    MCATime->InitializeMCA();
    MCATime->CONNECTION->Open();
    X1DData1 = gcnew array<float>(MaxChannel - MinChannel + 1);
    Y1DData1 = gcnew array<int>(MaxChannel - MinChannel + 1);
    X1DData2 = gcnew array<float>(Convert::ToInt32(TimeScanStepsBox->Text));
    Y1DData2 = gcnew array<int>(Convert::ToInt32(TimeScanStepsBox->Text);
All1DData = gcnew array<int,2>(Convert::ToInt32(TimeScanStepsBox->Text),MaxChannel - MinChannel + 1);
for(int i = 0; i < All1DData->GetLength(0); i++)
{
    for(int j = 0; j < All1DData->GetLength(1); j++)
    {
        All1DData->SetValue(Convert::ToInt32(0),i,j);
    }
}
Array::Clear(X1DData1,0,X1DData1->Length);
Array::Clear(Y1DData1,0,Y1DData1->Length);
Array::Clear(X1DData2,0,X1DData2->Length);
Array::Clear(Y1DData2,0,Y1DData2->Length);
int IndexCounter = 0;
for(IndexCounter = X1DData1->GetLowerBound(0);
    IndexCounter <= X1DData1->GetUpperBound(0);
    IndexCounter++)
{
    X1DData1->SetValue((Intercept + (IndexCounter + MinChannel) *
                        Slope),IndexCounter);
}
for(IndexCounter = X1DData2->GetLowerBound(0);
    IndexCounter <= X1DData2->GetUpperBound(0);
    IndexCounter++)
{
    X1DData2->SetValue((IndexCounter),IndexCounter);
}
CreateInstancesOfWindows();
TimeScanFormIvsE->Show();
TimeScanFormIvsT->Show();
TimeScanFormGRD->Show();
GC::Collect();
TimeStepCounter = 0;
MoveToNext1T = false;
```csharp
private: void TimeScanTimer_Tick(System::Object^, System::EventArgs^)
{
    Object^ TestO;
    (ReadDataFromMCA(MCATime->CONNECTION))->CopyTo(Y1DData1, 0);
    if (TimeScanFormIvsE->IsDisposed == false)
    {
        TimeScanFormIvsE->XYPlot->LoadPlotFromArray(X1DData1,
```
if (MoveToNext1T == true) {
    (ReadDataFromMCA (MCATime->CONNECTION)) -> CopyTo (Y1DData1, 0);
    MCATime->CONNECTION->Comm ("STOP");
    YdataTotal = 0; int dtemp;
    for (int index1 = 0; index1 < (MaxChannel - MinChannel + 1); index1++) {
        dtemp = Convert::ToInt32 (Y1DData1->GetValue (index1));
        TestO = (Convert::ToInt32 (dtemp));
        All1DData->SetValue (TestO, TimeStepCounter - 1, index1);
        YdataTotal = YdataTotal + dtemp;
    }
    TestO = (Convert::ToInt32 (YdataTotal));
    Y1DData2->SetValue (TestO, TimeStepCounter - 1);
    if (TimeScanFormIvsE->IsDisposed == false) {
        TimeScanFormIvsE->XYPlot->LoadPlotFromArray (X1DData1,
            Y1DData1, Color : : Blue, "I vs E");
    }
    if (TimeScanFormIvsT->IsDisposed == false) {
        TimeScanFormIvsT->XYPlot->LoadPlotFromArray (X1DData2,
            Y1DData2, Color : : Blue, "I vs T");
    }
    MCATime->CONNECTION->Comm ("CLEAR");
    MoveToNext1T = false;
    if (TimeStepCounter < Convert::ToInt32 (TimeScanStepsBox->Text)) {
        MCATime->CONNECTION->Comm ("START");
    }
if (MCATime->CONNECTION->isActive == false)
{
    MoveToNext1T = true;   TimeStepCounter++;
}
if (MCATime->CONNECTION->isActive == true)
{
    MCAMotionLiveBox->Text =
        Convert::ToString(Math::Round((Convert::ToSingle((
            Convert::ToString(MCATime->CONNECTION->Comm("SHOW_LIVE"))
        ->Remove(0,2)) ->Remove(10,3))/50),1));
    MCAMotionTrueBox->Text =
        Convert::ToString(Math::Round((Convert::ToSingle((
            Convert::ToString(MCATime->CONNECTION->Comm("SHOW_TRUE"))
        ->Remove(0,2)) ->Remove(10,3))/50),1));
}
if ((TimeStepCounter) > Convert::ToInt32(TimeScanStepsBox->Text))
{
    MoveToNext1T = false;   MCATime->CONNECTION->Comm("STOP");
    MCATime->CONNECTION->Close();   TimeScanTimer->Enabled = false;
    TimeScanProgressBar->Value = TimeStepCounter - 1;
    CreateInstancesOfWindows();
    TimeScanFormGRD->Show();   TimeScanFormIvsT->Show();
    TimeScanFormIvsT->XYPlot->LoadPlotFromArray(X1DData2, Y1DData2,
        Color::Blue,"I vs T");
    TimeScanFormGRD->ThreeDPlotControl->LoadPlotFromArray(
        All1DData,0,1,Convert::ToSingle(Intercept + MinChannel
        * Slope), Convert::ToSingle(Slope));
}
This appendix shows the source code for the simulation program. More details about how the program works can be found in section 3.7.2. Note that all of the following variables and functions are members of the same class ID2P. An instance of this class must be declared by calling one of the constructors ID2P before calling any member function. All try-catch statements are omitted here and should be added around any part of the code that may throw an exception. All of graphical interface and data plotting and display code is not shown here. Please refer to the actual code for a fully functional code.

```cpp
private:
    Object* ob;
    double* SampleTheta; // system diffraction angle
    double* mu; // sample mu
    double* DeltaY; // simulation parameter
    double* DeltaTheta1; // simulation parameter
    double* DeltaP; // simulation parameter
    double* C1; // col1 opening
    double* C2; // col2 opening
    double* C3; // col3 opening
    double* C4; // col4 opening
    double* S1; // col1 misalignment
    double* S2; // col2 misalignment
    double* S3; // col3 misalignment
    double* S4; // col4 misalignment
    double* X1; // source to col1 distance
```


```c
double^ X2; // col1 to col2 distance
double^ X3; // col2 to sample distance
double^ Y1; // sample to col3 distance
double^ Y2; // col3 to col4 distance
double^ Y3; // col4 to detector distance
double^ C1Dx; // col1 lower point x coordinate
double^ C1Dy; // col1 lower point y coordinate
double^ C1Ux; // col1 upper point x coordinate
double^ C1Uy; // col1 upper point y coordinate
double^ C2Dx; // col2 lower point x coordinate
double^ C2Dy; // col2 lower point y coordinate
double^ C2Ux; // col2 lower point x coordinate
double^ C2Uy; // col2 lower point y coordinate
double^ SinSample2Theta;
double^ CosSample2Theta;
double^ C3Mx; // col3 mid point x coordinate
double^ C3My; // col3 mid point y coordinate
double^ C4Mx; // col4 mid point x coordinate
double^ C4My; // col4 mid point y coordinate
double^ C3Dx; // col3 lower point x coordinate
double^ C3Dy; // col3 lower point y coordinate
double^ C3Ux; // col3 upper point x coordinate
double^ C3Uy; // col3 upper point y coordinate
double^ C4Dx; // col4 lower point x coordinate
double^ C4Dy; // col4 lower point y coordinate
double^ C4Ux; // col4 upper point x coordinate
double^ C4Uy; // col4 upper point y coordinate
array<double^>^ P1;
array<double^>^ P2;
array<double^>^ L1;
array<double^>^ L2;
array<double^>^ L3;
```
array<double*>^ L4;
double^ ThetaMin;
double^ ThetaMax;
array<double*>^ SampleEnterPoint;
array<double*>^ PenetRange;
array<double*>^ ThetaIJMinMax;

private: static double h = 6.6260693e−34; // KMS
private: static double SpeedOfLight = 299792458.0; // KMS
private: static double elec = 1.60217653e−19; // KMS
private: array<double*,2>^ IncidentBeam;
private: array<double*,2>^ PlanesSpcArray;
public: double (^Dfunction)(double , array<double*>^);
array<double*,2>^ GetIEofXforDXretval;

public: GRD^ GetIEDepthProfile(array<double*>^ XData)
// get energy dispersive intensity depth profile
{
    GRD* grd = gcnew GRD();
grd->x0 = *XData[0];
grd->deltax = *XData[1] - *XData[0];
    int xLen = XData->GetLength(0);
array<double*,2>^ YData = GetIEofX(*XData[0]);
grd->y0 = *YData[0,0];
grd->deltay = *YData[0,1] - *YData[0,0];
grd->data = gcnew
            array<double*,2>(XData->GetLength(0), YData->GetLength(1));
for (int i = 0; i < YData->GetLength(1); i++)
{
    grd->data->SetValue(*YData[1,i],0,i);
}
for (int j = 1; j < xLen; j++)
{
public: array<
double*,2>* GetIEofXforDx(double X, array<double*>* p)
// get energy dispersive intensity at
// depth x using a function with the parameters p
{
    double Iytz = 0.0; double cf = *DeltaY * *DeltaThetai * *DeltaP * 1.0e13;
    double^ DiffX = 0.0; double^ DiffY = 0.0;
    double^ SampleEnterX = 0.0;
    double^ SampleEnterY = 0.0;
    double^ PlanesSpc = 0.0; double dx = 0;
    for (int i = 0; i < D_ArrayMaxSize; i++)
    {
        PlanesSpcArray->SetValue((double)(i),0,i);
        dx = Dfunction((double)(i),p);
        if ((dx > DMAX) || (dx < DMIN)) return nullptr;
        PlanesSpcArray->SetValue(dx,1,i);
    }
    double^ TotalDistance = 0.0;
    // Total distance travelled inside the material
    double^ TanThetaIJPlus2ThetaE = 0.0; double^ mu = 0.0;
    double^ TanThetaIJ = 0.0;
    double^ SinThetaIJ = 0.0; double^ CosThetaIJ = 0.0;
    int zmin = 0; int zmax = 0; double^ Ii = 0.0;
double^ x = 0.0;

array< double^ >^ dMinMax
    = GetPlanesSpcMinMax( PlanesSpcArray );
double^ MinPlanesSpc = *dMinMax[ 0 ];
double^ MaxPlanesSpc = *dMinMax[ 1 ];
double^ SinSampleThetaMinusThetaIJ;
double^ SinSampleThetaPlusThetaIJ;
double^ EMin = h * SpeedOfLight /
    ( elec * 2.0 * *MaxPlanesSpc * ( 1.0e−7 )
        * Math::Sin(*ThetaMax ) ) ;
double^ EMax = h * SpeedOfLight /
    ( elec * 2.0 * *MinPlanesSpc * ( 1.0e−7 )
        * Math::Sin(*ThetaMin ) ) ;
int j = 0;
int whiteLen = IncidentBeam->GetLength( 1 );
for ( int ie = 0; ie < whiteLen; ie++ )
{
    GetIEofXforDXretval->SetValue( 0.0, 1, ie );
}
while (*EMin > *IncidentBeam[ 0, j ] )
{
    if ( j >= ( whiteLen − 1 ) ) break ;
    j++ ;
}
zmin = j ;

j = 0 ;
while (*EMax > *IncidentBeam[ 0, j ] )
{
    if ( j >= ( whiteLen − 1 ) ) break ;
    j++ ;
}
zmax = j ;
\[ \text{double}\, \theta_{Ei} = 0.0; \quad \text{double}\, \Theta_{Ei} = 0.0; \quad // \text{Theta}(E_i) \]

\[ \text{bool}\, \text{PassedExit} = \text{false}; \]

\[ \text{double}\, X_s = X * 1.0e-6; \quad \text{int}\, Z = 0; \]

\[ \text{for}( \text{double}\, \gamma_i = *\text{P1}[1]; \quad \gamma_i <= *\text{P2}[1]; \quad \gamma_i = *\gamma_i + *\text{DeltaY}) \]

\[ // \text{Pass on every point in the source} \]

\[ \{ \]

\[ \text{ThetaIJMinMax} = \text{GetThetaRange}(\gamma_i, C1Dx, C1Dy, C1Ux, C1Uy, C2Dx, C2Dy, C2Ux, C2Uy); \]

\[ \text{for}( \text{double}\, \theta_{IJ} = \text{ThetaIJMinMax}[0]; \]

\[ \theta_{IJ} <= \text{ThetaIJMinMax}[1]; \]

\[ \theta_{IJ} = \theta_{IJ} + *\text{DeltaThetaI} \]

\[ // \text{Pass on every possible direction for} \]

\[ // \text{the x-rays from the source} \]

\[ \{ \]

\[ \text{TanThetaIJ} = \text{Math::Tan}(\theta_{IJ}); \]

\[ \text{SinThetaIJ} = \text{Math::Sin}(\theta_{IJ}); \]

\[ \text{CosThetaIJ} = \text{Math::Cos}(\theta_{IJ}); \]

\[ \text{SinSampleThetaMinusThetaIJ} = \]

\[ \text{Math::Sin}(\text{SampleTheta} - \theta_{IJ}); \]

\[ \text{SinSampleThetaPlusThetaIJ} = \]

\[ \text{Math::Sin}(\text{SampleTheta} + \theta_{IJ}); \]

\[ \text{if}\, (\text{PassedEnterSlits2}(\gamma_i, X_1, X_2, \]

\[ \text{TanThetaIJ}, C1, C2, S1, S2)) \]

\[ \{ \]

\[ \text{SampleEnterPoint} = \text{GetSampleEnterPoint}(\]

\[ \gamma_i, \theta_{IJ}, \text{SampleTheta}, X_s, X_1, X_2, X_3); \]

\[ *\text{SampleEnterX} = *\text{SampleEnterPoint}[0]; \]

\[ *\text{SampleEnterY} = *\text{SampleEnterPoint}[1]; \]

\[ \text{PenetRange} = \text{GetPenetRange}(\gamma_i, \]

\[ \theta_{IJ}, \text{SampleTheta}, X_s, X_1, X_2, X_3, \]

\[ C3Dx, C3Dy, C3Ux, C3Uy, \]

\[ C4Dx, C4Dy, C4Ux, C4Uy); \]
for (int z = zmin; z <= zmax; z++)
{
    Ei = *IncidentBeam[0, z];
    Ii = *IncidentBeam[1, z];
    if (*Ii <= 0.0) goto endEnergy;
    Iytz = 0;
    for (double* Penet = *PenetRange[0];
         *Penet <= *PenetRange[1];
         *Penet = *Penet + *DeltaP)
    {
        *DiffX = *SampleEnterX + *Penet * *CosThetaIJ;
        *DiffY = *SampleEnterY + *Penet * *SinThetaIJ;
        if (*Penet <= 0) goto endPenet;
        x = *Penet * *SinSampleThetaMinusThetaIJ;
        *PlanesSpc = (double)(
            GetDValueFromArray(PlanesSpcArray ,
            *x * 1.0e6));
        ThetaEi = (Math::Asin((h * SpeedOfLight) /
            ((1e-7) * elec * *Ei)) / (2 * *PlanesSpc));
        TanThetaIJPlus2ThetaE = Math::Tan(
            *ThetaIJ + 2 * *ThetaEi);
        try
        {
            PassedExit = PassedExitSlitsVer2(
                DiffX, DiffY,
                TanThetaIJPlus2ThetaE ,
                C3Dx, C3Dy, C3Ux, C3Uy,
                C4Dx, C4Dy, C4Ux, C4Uy);
        }
        catch(Object^ ob)
        {
            Debug::WriteLine(ob->ToString());
        }
    }
}
if (PassedExit)
{
    TotalDistance = 100 * Penet * (1 + *SinSampleThetaMinusThetaIJ/
    Math::Sin(*ThetaIJ
    - *SampleTheta + 2 * *ThetaEi));
    Iytz = Iytz
    + cf * (double)((*Ii
    * Math::Exp(-1 *
    *mu * *TotalDistance)) * 1.0e-2);
} // if passed all slits
endPenet::;
} // Penet
*GetIEofXforDXretval[1,z] =
*GetIEofXforDXretval[1,z]
+ (double)(Iytz);
Z++; endEnergy::;
} // EnergyI
} // if passed 1 & 2
} // ThetaIJ
} // Yi
return GetIEofXforDXretval;
}

public: array<double*,2>^ GetIEofX(double X)
// get energy dispersive intensity profile at given depth
{
double Iytz = 0.0;
double cf = *DeltaY * *DeltaThetai * *DeltaP * 1.0e13;
double^ DiffX = 0.0; double^ DiffY = 0.0;
double SampleEnterX = 0.0;
double SampleEnterY = 0.0;
double PlanesSpc = 0.0;
double TotalDistance = 0.0;

// Total distance travelled inside the material
double TanThetaIJPlus2ThetaE = 0.0;
double mu = 0.0;
double TanThetaIJ = 0.0;
double SinThetaIJ = 0.0;
double CosThetaIJ = 0.0;
int zmin = 0; int zmax = 0;
double Ii = 0.0; double x = 0.0;
array<
double>
 dMinMax =
 GetPlanesSpcMinMax(PlanesSpcArray);
double MinPlanesSpc = *dMinMax[0];
double MaxPlanesSpc = *dMinMax[1];
double SinSampleThetaMinusThetaIJ;
double SinSampleThetaPlusThetaIJ;
double EMin = h * SpeedOfLight / (elec * 
 2.0 * *MaxPlanesSpc * (1.0e-7) * Math::Sin(*ThetaMax));
double EMax = h * SpeedOfLight / (elec * 
 2.0 * *MinPlanesSpc * (1.0e-7) * Math::Sin(*ThetaMin));
int j = 0;
int whiteLen = IncidentBeam->GetLength(1);
for (int ie = 0; ie < whiteLen; ie++)
{
    GetIEofXforDXretval->SetValue(0.0,1,ie);
}
while (*EMin > *IncidentBeam[0,j])
{
    if (j >= (whiteLen - 1)) break;
    j++;
\[
\text{while } (*E_{\text{Max}} > *\text{IncidentBeam}[0,j])
\]
\[
\text{if } (j > (\text{whiteLen} - 1)) \text{ break;}
\]
\[
j++;}
\]
\[
z_{\text{max}} = j; \quad \text{double}^* E_{\text{i}} = 0.0;
\]
\[
\text{double}^* \Theta_{\text{Ei}} = 0.0; \quad // \Theta(E_{\text{i}})
\]
\[
\text{bool PassedExit} = \text{false};
\]
\[
\text{double}^* X_{\text{s}} = X \times 1.0 \times 10^{-6}; \quad \text{int} Z = 0;
\]
\[
\text{for (double}^* Yi = *P1[1]; *Yi < = *P2[1];
\]
\[
\quad *Yi = *Yi + *\text{DeltaY})
\]
\[
\quad // \text{Pass on every point in the source}
\]
\[
\{\text{ThetaIJMinMax} = \text{GetThetaRange}(Yi, C1Dx, C1Dy, C1Ux, C1Uy, C2Dx, C2Dy, C2Ux, C2Uy);
\]
\[
\text{for (double}^* \Theta_{\text{IJ}} = *\text{ThetaIJMinMax}[0];
\]
\[
\quad *\Theta_{\text{IJ}} <= *\text{ThetaIJMinMax}[1];
\]
\[
\quad *\Theta_{\text{IJ}} = *\Theta_{\text{IJ}} + *\text{DeltaThetai})
\]
\[
\quad // \text{Pass on every possible direction}
\]
\[
\quad // \text{for the x-rays from the source}
\]
\[
\{\text{TanThetaIJ} = \text{Math::Tan(*ThetaIJ)};
\]
\[
\text{SinThetaIJ} = \text{Math::Sin(*ThetaIJ)};
\]
\[
\text{CosThetaIJ} = \text{Math::Cos(*ThetaIJ)};
\]
\[
\text{SinSampleThetaMinusThetaIJ} =
\]
\[
\text{Math::Sin(*SampleTheta} - *\Theta_{\text{IJ}});
\]
\[
\text{SinSampleThetaPlusThetaIJ} =
\]
\[
\text{Math::Sin(*SampleTheta} + *\Theta_{\text{IJ}});
\]
\[
\text{if } (\text{PassedEnterSlits2}(Yi,X1,X2,}
TanThetaIJ(C1,C2,S1,S2))
{
    SampleEnterPoint = GetSampleEnterPoint(Yi, ThetaIJ, SampleTheta, Xs, X1, X2, X3);
    *SampleEnterX = *SampleEnterPoint[0];
    *SampleEnterY = *SampleEnterPoint[1];
    PenetRange = GetPenetRange(Yi, ThetaIJ, SampleTheta, Xs, X1, X2, X3,
                              C3Dx, C3Dy, C3Ux, C3Uy,
                              C4Dx, C4Dy, C4Ux, C4Uy);
    for(int z = zmin; z <= zmax; z++)
    {
        Ei = *IncidentBeam[0, z];
        Ii = *IncidentBeam[1, z];
        if (*Ii <= 0.0) goto endEnergy;
        Iy tz = 0;
        for(double ^ Penet = *PenetRange[0];
            *Penet <= *PenetRange[1];
            *Penet = *Penet + *DeltaP)
        {
            *DiffX = *SampleEnterX + *Penet* *CosThetaIJ;
            *DiffY = *SampleEnterY + *Penet* *SinThetaIJ;
            if (*Penet <= 0) goto endPenet;
            x = *Penet * *SinSampleThetaMinusThetaIJ;
            PlanesSpc = (double)(
                GetDValueFromArray(PlanesSpcArray, *x * 1.0e6));
            ThetaEi = (Math::Asin(((h * SpeedOfLight) / ((1e−7) * elec * *Ei))
                               / (2 * *PlanesSpc)));
            TanThetaIJPlus2ThetaE = Math::Tan(*ThetaIJ
                                              + 2 * *ThetaEi);
        }
    }
}
PassedExit = PassedExitSlitsVer2(
    DiffX, DiffY,
    TanThetaIJPlus2ThetaE,
    C3Dx, C3Dy, C3Ux, C3Uy,
    C4Dx, C4Dy, C4Ux, C4Uy);

if (PassedExit)
{
    TotalDistance = 100 * Penet * (1
        + *SinSampleThetaMinusThetaIJ /
        Math::Sin(*ThetaIJ - *SampleTheta
        + 2 * *ThetaEi));
    Iytz = Iytz + cf * (double)((*Ii
        * Math::Exp(-1 *
        *mu * *TotalDistance)) * 1.0e-2);
}
} // if passed all slits

endPenet;

} // Penet

*GetIEofXforDXretval[1,z] =
    *GetIEofXforDXretval[1,z]
    + (double)(Iytz);

Z++; endEnergy:

} // EnergyI

} // if passed 1 & 2

} // ThetaIJ

} // Yi

return GetIEofXforDXretval;

}
double c1, double c2, double c3, double c4,
double s1, double s2, double s3, double s4,
double theta, double mu,
double deltaYi, double deltaTheta, double deltaPenet)

// construct a class that represents the experimental setup.
// Intensity depth profiles can be calculated using this class
// use the given experimental setup dimensions
{
  PlanesSpcArray = gcnew array<double^,2>(2, D_ArrayMaxSize);
  IncidentBeam = ReadIncidentBeamFile(IncidentBeamFile);
  PlanesSpcArray = GetdvsxArray(a,b,c,
                              alpha, beta, gamma, H,K,L,0.0, D_ArrayMaxSize, 1.0);
  SampleTheta = theta * Math::PI/180.0;
  DeltaY  = deltaYi * 1.0e-6;
  DeltaTheta = deltaTheta * 1.0e-6 * Math::PI/180.0;
  DeltaP  = deltaPenet * 1.0e-6;
  C1  = c1 * 1.0e-6;
  C2  = c2 * 1.0e-6;
  C3  = c3 * 1.0e-6;
  C4  = c4 * 1.0e-6;
  S1  = s1 * 1.0e-6;
  S2  = s2 * 1.0e-6;
  S3  = s3 * 1.0e-6;
  S4  = s4 * 1.0e-6;
  X1  = x1;
  *X1 = *X1 * 1.0e-3;
  X2  = x2;
  *X2 = *X2 * 1.0e-3;
  X3  = x3;
  *X3 = *X3 * 1.0e-3;
  Y1  = y1;
  *Y1 = *Y1 * 1.0e-3;
\[ Y_2 = y_2; \]
\[ *Y_2 = *Y_2 * 1.0e-3; \]
\[ Y_3 = y_3; \]
\[ *Y_3 = *Y_3 * 1.0e-3; \]
\[ C_{1Dx} = *X_1; \]
\[ C_{1Dy} = -1 * *C_1/2.0 + *S_1; \]
\[ C_{1Ux} = *X_1; \]
\[ C_{1Uy} = *C_1/2.0 + *S_1; \]
\[ C_{2Dx} = *X_2 + *X_1; \]
\[ C_{2Dy} = -1 * *C_2 * 0.5 + *S_2; \]
\[ C_{2Ux} = *X_2 + *X_1; \]
\[ C_{2Uy} = *C_2 * 0.5 + *S_2; \]
\[ \text{SinSample2Theta} = \text{Math::Sin}(2 * *\text{SampleTheta}); \]
\[ \text{CosSample2Theta} = \text{Math::Cos}(2 * *\text{SampleTheta}); \]
\[ C_{3Mx} = *X_1 + *X_2 + *X_3 + *Y_3 + *\text{CosSample2Theta}; \]
\[ C_{3My} = *Y_3 + *\text{SinSample2Theta}; \]
\[ C_{4Mx} = *X_1 + *X_2 + *X_3 + (*Y_3 + *Y_2)* *\text{CosSample2Theta}; \]
\[ C_{4My} = (*Y_3 + *Y_2)* *\text{SinSample2Theta}; \]
\[ C_{3Dx} = *C_{3Mx} + ( *C_3 * 0.5 - *S_3) * *\text{SinSample2Theta}; \]
\[ C_{3Dy} = *C_{3My} + (-1 * *C_3 * 0.5 + *S_3) * *\text{CosSample2Theta}; \]
\[ C_{3Ux} = *C_{3Mx} + (-1 * *C_3 * 0.5 - *S_3) * *\text{SinSample2Theta}; \]
\[ C_{3Uy} = *C_{3My} + ( *C_3 * 0.5 + *S_3) * *\text{CosSample2Theta}; \]
\[ C_{4Dx} = *C_{4Mx} + ( *C_4 * 0.5 - *S_4) * *\text{SinSample2Theta}; \]
\[ C_{4Dy} = *C_{4My} + (-1 * *C_4 * 0.5 + *S_4) * *\text{CosSample2Theta}; \]
\[ C_{4Ux} = *C_{4Mx} + (-1 * *C_4 * 0.5 + *S_4) * *\text{SinSample2Theta}; \]
\[ C_{4Uy} = *C_{4My} + ( *C_4 * 0.5 + *S_4) * *\text{CosSample2Theta}; \]
\[ P_1 = \text{GetIntersectionOf}(C_{2Ux}, C_{2Uy}, C_{1Dx}, C_{1Dy}, 0.0, 0.0, 0.0, 1.0); \]
\[ P_2 = \text{GetIntersectionOf}(C_{1Ux}, C_{1Uy}, C_{2Dx}, C_{2Dy}, 0.0, 0.0, 0.0, 1.0); \]
\[ L_1 = \text{GetEquationOf}(C_{1Dx}, C_{1Dy}, C_{2Ux}, C_{2Uy}); \]
\[ L_2 = \text{GetEquationOf}(C_{1Ux}, C_{1Uy}, C_{2Dx}, C_{2Dy}); \]
\[ L_3 = \text{GetEquationOf}(C_{3Ux}, C_{3Uy}, C_{4Dx}, C_{4Dy}); \]
\[ L_4 = \text{GetEquationOf}(C_{3Dx}, C_{3Dy}, C_{4Ux}, C_{4Uy}); \]
\[ \Theta_{\text{Min}} = 0.5 \times (\mathbf{Atan}(L3[1]) - \mathbf{Atan}(L1[1])); \]
\[ \Theta_{\text{Max}} = 0.5 \times (\mathbf{Atan}(L4[1]) - \mathbf{Atan}(L2[1])); \]
SampleEntryPoint = gcnew array<double>(2);
PenetRange = gcnew array<double>(2);
ThetaIJMinMax = gcnew array<double>(2);

\[
\text{whiteLen} = \text{IncidentBeam.GetLength}(1);
\]
GetIEofXforDXretval = gcnew array<double, 2>(2, whiteLen);

\[
\text{for} (\text{int} \, ie = 0; \, ie < \text{whiteLen}; \, ie++)
\{
    \text{GetIEofXforDXretval} \rightarrow \text{SetValue}(\text{IncidentBeam}[0, \, ie], 0, \, ie);
    \text{GetIEofXforDXretval} \rightarrow \text{SetValue}(0.0, 1, \, ie);
\}
\]

\textbf{public}: ID2P(String* IncidentBeamFile, 
    double dfunction(double, array<double>^), 
    double x1, double x2, double x3, 
    double y1, double y2, double y3, 
    double c1, double c2, double c3, double c4, 
    double s1, double s2, double s3, double s4, 
    double theta, double mu, 
    double deltaYi, double deltaTheta, double deltaPenet)

// construct idp class using the given experimental setup dimensions
{
    PlanesSpcArray = gcnew array<double, 2>(2, D_ArrayMaxSize);
    IncidentBeam = ReadIncidentBeamFile(IncidentBeamFile);
    Dfunction = dfunction;
    SampleTheta = theta * Math::PI/180.0;
    DeltaY = deltaYi*1.0e-6;
    DeltaTheta = deltaTheta* 1.0e-6 * Math::PI/180.0;
    DeltaP = deltaPenet*1.0e-6;
    C1 = c1 * 1.0e-6;
C2 = c2 * 1.0e-6;
C3 = c3 * 1.0e-6;
C4 = c4 * 1.0e-6;
S1 = s1 * 1.0e-6;
S2 = s2 * 1.0e-6;
S3 = s3 * 1.0e-6;
S4 = s4 * 1.0e-6;
X1 = x1;
*X1 = *X1 * 1.0e-3;
X2 = x2;
*X2 = *X2 * 1.0e-3;
X3 = x3;
*X3 = *X3 * 1.0e-3;
Y1 = y1;
*Y1 = *Y1 * 1.0e-3;
Y2 = y2;
*Y2 = *Y2 * 1.0e-3;
Y3 = y3;
*Y3 = *Y3 * 1.0e-3;
C1Dx = *X1;
C1Dy = -1 * *C1/2.0 + *S1;
C1Ux = *X1;
C1Uy = *C1/2.0 + *S1;
C2Dx = *X2 + *X1;
C2Dy = -1 * *C2 * 0.5 + *S2;
C2Ux = *X2 + *X1;
C2Uy = *C2 * 0.5 + *S2;
SinSample2Theta= Math::Sin(2 * *SampleTheta);
CosSample2Theta= Math::Cos(2 * *SampleTheta);
C3Mx = *X1 + *X2 + *X3 + *Y3 + *CosSample2Theta;
C3My = *Y3 + *SinSample2Theta;
C4Mx = *X1 + *X2 + *X3 + (*Y3 + *Y2) * *CosSample2Theta;
C4My = (\ast Y3 + \ast Y2) \ast \sin\text{Sample2Theta};
C3Dx = \ast C3Mx + (\ast C3 \ast 0.5 - \ast S3) \ast \sin\text{Sample2Theta};
C3Dy = \ast C3My + (-1 \ast \ast C3 \ast 0.5 + \ast S3) \ast \cos\text{Sample2Theta};
C3Ux = \ast C3Mx + (-1 \ast \ast C3 \ast 0.5 - \ast S3) \ast \sin\text{Sample2Theta};
C3Uy = \ast C3My + (\ast C3 \ast 0.5 + \ast S3) \ast \cos\text{Sample2Theta};
C4Dx = \ast C4Mx + (\ast C4 \ast 0.5 - \ast S4) \ast \sin\text{Sample2Theta};
C4Dy = \ast C4My + (-1 \ast \ast C4 \ast 0.5 + \ast S4) \ast \cos\text{Sample2Theta};
C4Ux = \ast C4Mx + (-1 \ast \ast C4 \ast 0.5 - \ast S4) \ast \sin\text{Sample2Theta};
C4Uy = \ast C4My + (\ast C4 \ast 0.5 + \ast S4) \ast \cos\text{Sample2Theta};
P1 = \text{GetIntersectionOf}(C2Ux, C2Uy, C1Dx, C1Dy, 0.0, 0.0, 0.0, 1.0);
P2 = \text{GetIntersectionOf}(C1Ux, C1Uy, C2Dx, C2Dy, 0.0, 0.0, 0.0, 1.0);
L1 = \text{GetEquationOf}(C1Dx, C1Dy, C2Ux, C2Uy);
L2 = \text{GetEquationOf}(C1Ux, C1Uy, C2Dx, C2Dy);
L3 = \text{GetEquationOf}(C3Ux, C3Uy, C4Dx, C4Dy);
L4 = \text{GetEquationOf}(C3Dx, C3Dy, C4Ux, C4Uy);
ThetaMin = 0.5 \ast (\text{Math::Atan}(\ast L3[1]) - \text{Math::Atan}(\ast L1[1]));
 ThetaMax = 0.5 \ast (\text{Math::Atan}(\ast L4[1]) - \text{Math::Atan}(\ast L2[1]));
SampleEnterPoint = gcnew array<double>(2);
PenetRange = gcnew array<double>(2);
ThetaIJMinMax = gcnew array<double>(2);

private: static double GetDistance(double x1, double y1,
                                    double x2, double y2)
// get distance between two points
{
    return Math::Sqrt(Math::Pow(*x1 - *x2, 2.0)
                       + Math::Pow(*y1 - *y2, 2.0));
}
// get intersection of two lines
{
    array<
double^>^ retval = gcnew array<
double^>(2);
    retval[0] = (*x1 - *x2)/(*m2 - *m1);
    retval[1] = *x2 + *m2 * retval[0];
    return retval;
}

private: static array<
double^>^ GetIntersectionOf(
double^ x1, double^ y1,
double^ x2, double^ y2, double^ x3, double^ y3, double^ x4,
double^ y4) // get intersection of two lines
{
    array<
double^>^ retval = gcnew array<
double^>(2);
    double^ x =
    (*x1 * ( *x3 * ( *y2 - *y4 ) - *x4 * ( *y2 - *y3 )))
    - *x2 * ( *x3 * ( *y1 - *y4 ) - *x4 * ( *y1 - *y3 )))
    /( *x1 * ( *y3 - *y4 ) - *x2 * ( *y3 - *y4 )
    - ( *x3 - *x4 ) * ( *y1 - *y2 ));
    double^ y = *y2 -(( ( *x2 * ( *y3 - *y4 )
    - *x3 * ( *y2 - *y4 ) + *x4 * ( *y2 - *y3 ))*( *y1 - *y2 ))
    / ( *x1 * ( *y3 - *y4 ) - *x2 * ( *y3 - *y4 ) - ( *x3 - *x4 )
    * ( *y1 - *y2 )));
    retval->SetValue(*x,0);
    retval->SetValue(*y,1);
}

private: static array<
double^>^ GetEquationOf(
double^ x1, double^ y1,
double^ x2, double^ y2)
// get equation of line using two points
{
    array<
double^>^ retval = gcnew array<
double^>(2);
    retval->SetValue(*y1 - *x1 * (*y1 - *y2)/(*x1 - *x2)),0);
}
private: static array<double*> GetPlanesSpcMinMax(array<double*,2>* arr)
{
    array<double*> MINnMAX = gcnew array<double*>(2);
    double dmin = 0; double dmax = 0;
    double D = (double)(arr[1,0]); dmin = D; dmax = D;
    for (int i = 0; i < arr->GetLength(1); i++)
    {
        D = (double)(arr[1,i]);
        if (D <= dmin) dmin = D;
        if (D >= dmax) dmax = D;
    }
    MINnMAX[0] = dmin; MINnMAX[1] = dmax;
    return MINnMAX;
}

private: static bool PassedEnterSlits2(double^ Yi, double^ X1, double^ X2,
                                         double^ TanThetaIJ, double^ C1, double^ C2, double^ S1, double^ S2)
    // check if x-ray passes the incident beam collimators
    {
        double^ TempY = *Yi + *X1 * *TanThetaIJ;
        if (((*TempY<=(*(C1 / 2.0)+ *S1)) && (*TempY>=((-1* *C1/2.0))+ *S1)))
        {
            *TempY = *Yi + (*X1 + *X2)* *TanThetaIJ;
            if (((*TempY<=(*C2 * 0.5 + *S2)) && (*TempY>=(-1* *C2*0.5 + *S2)))
                {
                    return true;
                }
        }
    }
private: static double GetDValueFromArray(array<double,2>* arr, double x)
// read lattice parameter value from array at given depth
{
    return *(arr[1,(int)x]);
}

private: static array<double*>* GetPenetRange(double* Yi, double* ThetaIJ, double* ThetaS, double* Xs, double* X1, double* X2, double* X3, double* x1, double* y1, double* x2, double* y2, double* x3, double* y3, double* x4, double* y4)
// get the penetration range of an x-ray inside the probing volume
{
    array<double*>* retval = gcnew array<double*>(2);
    array<double*>* p1 = gcnew array<double*>(2);
    // intersection of incident beam with sample surface
    array<double*>* p2 = gcnew array<double*>(2);
    // intersection of incident beam with L2
    array<double*>* p3 = gcnew array<double*>(2);
    // intersection of incident beam with L3
    array<double*>* L1 = gcnew array<double*>(2);
    // L1 is the line defining the sample surface
    array<double*>* L2 = gcnew array<double*>(2);
    // L3 and L2 are the two lines defining the exit slits divergence
    // L2 is formed by (x2,y2)−(x3,y3)
    array<double*>* L3 = gcnew array<double*>(2);
    // L3 is formed by (x1,y1)−(x4,y4)
    L2 = GetEquationOf(x2,y2,x3,y3);
L3 = GetEquationOf(x1, y1, x4, y4);

double X6 = *X1 + *X2 + *X3 - *Xs * Math::Sin(*ThetaS);

// (X6, Y6) is a point on the sample surface

double Y6 = *Xs * Math::Cos(*ThetaS);

L1->SetValue(*Y6 - *X6 * Math::Tan(*ThetaS), 0);
L1->SetValue(Math::Tan(*ThetaS), 1);

double LX0 = Yi;

double LM0 = Math::Tan(*ThetaIJ);

double LX1 = *L1[0];

double LM1 = *L1[1];

double LX2 = *L2[0];

double LM2 = *L2[1];

double LX3 = *L3[0];

double LM3 = *L3[1];

p1 = GetIntersectionOf(LX0, LM0, LX1, LM1);

p2 = GetIntersectionOf(LX0, LM0, LX2, LM2);

p3 = GetIntersectionOf(LX0, LM0, LX3, LM3);

double D1 = GetDistance(p1[0], p1[1], p2[0], p2[1]);

double D2 = GetDistance(p3[0], p3[1], p2[0], p2[1]);

double D3 = GetDistance(p3[0], p3[1], p1[0], p1[1]);

if (D3 >= D2)
{
    retval->SetValue(D1, 0);
    retval->SetValue(D3, 1);
}

if (D3 < D2)
{
    retval->SetValue(0.0, 0);
    retval->SetValue(D3, 1);
}

return retval;
private: static array<double> GetSampleEnterPoint(double Yi,
    double ThetaIJ, double ThetaS, double Xs,
    double X1, double X2, double X3)
{
    // get the point where an x-ray hits the sample
    double X6 = *X1 + *X2 + *X3 - *Xs * Math::Sin(*ThetaS);
    // (X6, Y6) is a point on the sample surface
    double Y6 = *Xs * Math::Cos(*ThetaS);
    double a1 = *Yi;    // y0 of line 1
    double b1 = Math::Tan(*ThetaIJ);    // slope of line 1
    double a2 = *Y6 - *X6 * Math::Tan(*ThetaS);    // y0 of line 2
    double b2 = Math::Tan(*ThetaS);    // slope of line 2
    return GetIntersectionOf(a1, b1, a2, b2);
    // the point where Yi ThetaIJ ray hit the sample at ThetaS
}

private: static double GetSlopeOf(double x1, double y1,
    double x2, double y2) // get slope of line
{
    return (*y2 - *y1) / (*x2 - *x1);
}

private: static bool PassedExitSlitsVer2(double DiffX, double DiffY,
    double TanThetaIJPlus2ThetaE, double X1, double Y1,
    double X2, double Y2, double X3, double Y3, double X4,
    double Y4)
// check if diffracted x-ray passes the diffracted beam collimators
{
    bool Passed3 = false; bool Passed4 = false;
    bool Passed5 = false; bool Passed6 = false;
    bool PassedAllSlits = false;
    if ((*DiffY + *TanThetaIJPlus2ThetaE *(*X1 - *DiffX)) >= *Y1)
Passeq3=true;
Passeq4=false;
if (Passeq3)
{
    if ( (*DiffY+*TanThetaIJPlus2ThetaE*(X2-*DiffX)) <= *Y2)
        Passeq4=true;
Passeq5=false;
if (Passeq4)
{
    if ( (*DiffY+*TanThetaIJPlus2ThetaE*(X3-*DiffX)) >= *Y3)
        Passeq5=true;
Passeq6=false;
if (Passeq5)
{
    Passeq6=false;
    if ( (*DiffY+*TanThetaIJPlus2ThetaE*(X4-*DiffX)) <= *Y4)
        Passeq6=true;
    if (Passeq6)
    {
        PasseqAllSlits=true;
    } // if passed 6
} // if passed 5
} // if passed 4
} // if passed 3
return PasseqAllSlits;
}

private: static array<double^,2>^ ReadIncidentBeamFile(String^ FileName)
// load incident beam from x-y text file
{
    if (FileName == nullptr) return nullptr;
    if (FileName == String::Empty) return nullptr;

IO::StreamReader^ sr = gcnew StreamReader(FileName);
int LinesCount = 0;
String^ AllInputData = sr->ReadToEnd(); sr->Close();
String^ delimStr1 = "\t"; String^ delimStr2 = "\n";
array<Char^> delimiter1 = delimStr1->ToCharArray();
array<Char^> delimiter2 = delimStr2->ToCharArray();
array<Char^> delimiter3 = delimStr2->ToCharArray();
AllInputData = AllInputData->Trim(delimiter3);
array<String^>^ splitLines = nullptr;
array<String^>^ CurrentLine;
splitLines = AllInputData->Split(delimiter2, 9999999);
LinesCount = splitLines->Length;
array<double^,2^>^ dat = gcnew array<double^,2^>(2,LinesCount);
for (int i = 0; i < LinesCount; i++)
{
    CurrentLine = splitLines[i]->Split(delimiter1,10);
    dat[0,i] = (double)(CurrentLine[0]); //x
    dat[1,i] = (double)(CurrentLine[1]); //a
}
return dat;

private: static array<double^>^ GetThetaRange(double^ yi,
    double^ cldx, double^ cldy, double^ clux, double^ cluy,
    double^ c2dx, double^ c2dy,double^ c2ux, double^ c2uy)
//get the possible angular range of x-rays
//emitted from the source within collimation
//this is the divergence of the incident beam
{
    array<double^>^ retval = gcnew array<double^>(2);  
    double^ T1 = Math::Atan(GetSlopeOf(0.0,yi,cldx,cldy));
    double^ T2 = Math::Atan(GetSlopeOf(0.0,yi,clux,cluy));
\( \text{double}^* T3 = \text{Math}:\text{Atan}(\text{GetSlopeOf}(0.0, y_i, c2dx, c2dy)); \)
\( \text{double}^* T4 = \text{Math}:\text{Atan}(\text{GetSlopeOf}(0.0, y_i, c2ux, c2uy)); \)
\( \text{retval}[0] = \text{Math}:\text{Max}(*T1,*T3); \)
\( \text{retval}[1] = \text{Math}:\text{Min}(*T2,*T4); \)
\( \text{return retval; } \)

private: static double GetPlanesSpc(int H, int K, int L, double a, double b, double c, double alpha, double beta, double gamma)

// get \( d_{\{hkl}\} \) for the given lattice parameters
{
alpha = (Math::PI/180.0) * alpha;
beta = (Math::PI/180.0) * beta;
gamma = (Math::PI/180.0) * gamma;
double D = 0.0;
D = ((1.0 + 2.0 * Math::Cos(alpha) * Math::Cos(beta)
 * Math::Cos(gamma)
 - Math::Cos(alpha) * Math::Cos(alpha) - Math::Cos(beta)
 * Math::Cos(beta)
 - Math::Cos(gamma) * Math::Cos(gamma)))
D = 1.0 / D;
double term1 = H * H * Math::Sin(alpha)
 * Math::Sin(alpha)/ ( a * a );
double term2 = K * K * Math::Sin(beta)
 * Math::Sin(beta)/ ( b * b );
double term3 = L * L * Math::Sin(gamma)
 * Math::Sin(gamma)/ ( c * c );
double term4 = (2.0 * H * K / ( a * b ))
 * (Math::Cos(alpha) * Math::Cos(beta) - Math::Cos(gamma));
double term5 = (2.0 * L * K / ( c * b ))
 * (Math::Cos(gamma) * Math::Cos(beta) - Math::Cos(alpha));
double term6 = (2.0 * H * L / ( a * c ))
\[ \mathbf{D} = D \times ( \text{term1} + \text{term2} + \text{term3} + \text{term4} + \text{term5} + \text{term6} ) ; \]
\[ \mathbf{D} = 1.0 / \mathbf{D} ; \]
\[ \mathbf{D} = (\mathbf{D} : \text{Sqrt}(\mathbf{D})) ; \]
\[ \text{return} \ \mathbf{D}; \]

private: static array<double*,2>* GetFunctionValuesArray(String* Function, double* xmin, double* xmax, double* xstep)
// build array for the lattice parameter vs
// depth using a function for the lattice parameter
{
    Fitting::ExpressionParser* parser = gcnew Fitting::ExpressionParser();
    Hashtable* h = gcnew Hashtable();
    int len = (int)((*xmax - *xmin) / *xstep);
    array<double*,2>* retval = gcnew array<double*,2>(2, len+1);
    int counter = 0;
    for (double x = *xmin; x <= *xmax; x = x + *xstep)
    {
        retval[0,counter] = x;
        h->Add( "x", Convert::ToString(x) );
        result = parser->Parse( Function, h );
        retval[1,counter] = result;
        h->Clear(); counter++;
    }
    return retval;
}

private: static array<double*,2>* GetdvsxArray(String* f1, String* f2, String* f3, String* f4, String* f5, String* f6, int H, int K, int L, double xmin, double xmax, double xstep)
// build array for the lattice parameter vs
// depth using a function for each lattice parameter
{
array<
double,2>
arr
= GetFunctionValuesArray(f1, xmin, xmax, xstep);
array<
double,2>
arrb
= GetFunctionValuesArray(f2, xmin, xmax, xstep);
array<
double,2>
arrc
= GetFunctionValuesArray(f3, xmin, xmax, xstep);
array<
double,2>
arralpha
= GetFunctionValuesArray(f4, xmin, xmax, xstep);
array<
double,2>
arrbeta
= GetFunctionValuesArray(f5, xmin, xmax, xstep);
array<
double,2>
arrgamma
= GetFunctionValuesArray(f6, xmin, xmax, xstep);
int len = arr->GetLength(1);
array<
double,2>
retval = gcnew array<
double,2>(2, len);
double D = 0; double x;
double a = 0; double c = 0; double b = 0;
double alpha = 0; double beta = 0; double gamma = 0;
for (int i = 0; i < len; i++)
{
    a = *arr[1, i]; b = *arrb[1, i]; c = *arrc[1, i];
    alpha = *arralpha[1, i]; beta = *arrbeta[1, i];
    gamma = *arrgamma[1, i]; x = *arr[0, i];
    D = GetPlanesSpc(H,K,L,a,b,c,alpha,beta,gamma);
    retval->SetValue(D,1,i); retval->SetValue(x,0,i);
}
return retval;
}
BIBLIOGRAPHY


