



British Society For Strain
Measurement Meeting, NPL, 27th
September 2023
Measuring Residual Stress Using a
Laboratory X-Ray Source, the $\sin^2\psi$
Method

Judith Shackleton, C. Eng.

Worked for Rolls-Royce Civil Large Engines Now
Retired!

judithshackleton@compuserve.com

The $\sin^2\psi$ Method

What are We Measuring?

- We measure the ELASTIC Strain. We can determine
 - Magnitude of the stress,
 - Its direction,
 - Its nature
 - Compressive or tensile
- We use the planes of the crystal lattice as an atomic scale "strain gauge".

The $\sin^2\psi$ Method; How Does it Work?

We measure *STRAIN* (ε) not *STRESS* (σ)

- We *CALCULATE STRESS* from the *STRAIN* & the *ELASTIC CONSTANTS*
- We use the planes $d_{\{hkl\}}$, of the crystal lattice as a strain gauge
 - We can measure the change in d -spacing, Δd
 - *Strain* = $\varepsilon = \Delta d/d$
- What is a crystalline material?
- What are d -spacings?

Why do We Need to Know about Basic Crystallography if we are using the $\sin^2\psi$ Method to Measure Residual Stress?

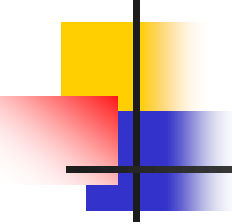
- When we use the $\sin^2\psi$ method we actually measure strain & convert to stress using the **elastic constants**; we don't measure the stress directly
- We determine the **strain** by measuring the changes in the **spacing between the planes in a crystalline material**
- Consequently, we need understand,
 - What is **crystalline material**?
 - What's the shape & size of its structure?
 - The shape of the crystal is defined by the crystal system & the Bravais lattice
 - How to measure the distances between the planes
 - We need to describe precisely how we have made our measurements so that others can understand the results

What is a Crystalline Material?

- Most materials have some type of crystal structure & we can (usually) detect this by X-ray diffraction
- A crystal is a regular repeated array of identical lattice points which can be atoms, ions or molecules.
 - The lattice is effectively infinite in three dimensions
 - Each lattice point is **IDENTICAL** to all of the others
 - A crystal lattice is sometimes called a "space lattice" because it "fills space". There are **no systematic holes** in the lattice (only "mistakes")
- The structures of all common metals (& a lot of uncommon ones!) have been known for many years & are readily available on the internet & in the literature
 - **Fortunately, most metals have very simple structures which are very easy to measure**

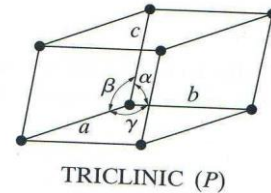
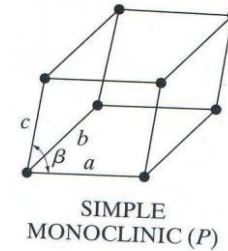
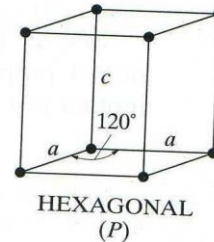
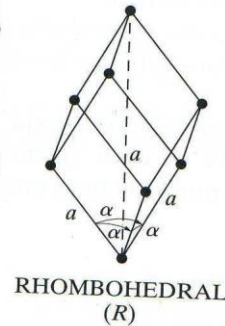
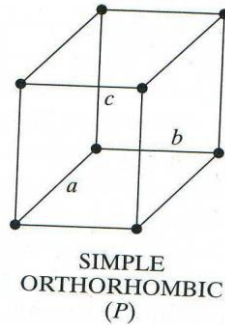
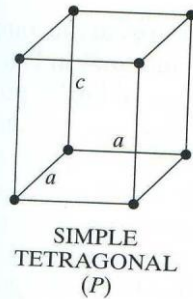
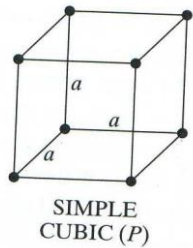


What is a Crystalline Material?



- A crystal is a regular repeated array of identical lattice points which can be atoms, ions or molecules.
 - Considered on the atomic scale, the lattice is effectively infinite
- The smallest unit of structure, which reflects the overall shape of the crystal is called a **UNIT CELL**
- There are just seven basic shapes (cubic, hexagonal, etc.) of unit cell; these are the seven **CRYSTAL SYSTEMS**
- Some unit cells have extra atoms and this gives the fourteen **BRAVAIS LATTICES** (symmetries)

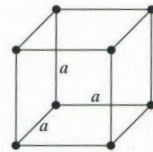
What do the Seven Crystal Systems Look Like?



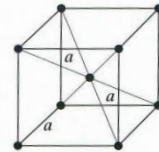
Simpler,
Higher
Symmetry
Unit Cells

More Complex, Lower
Symmetry Unit Cells

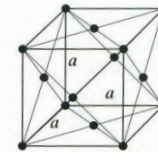
The Unit Cells of All of the 14 Bravais Lattices



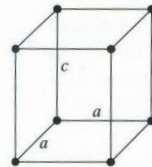
SIMPLE CUBIC (*P*)



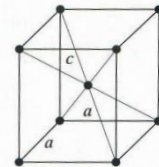
BODY-CENTERED CUBIC (*I*)



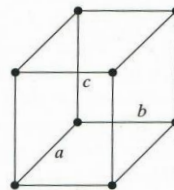
FACE-CENTERED CUBIC (*F*)



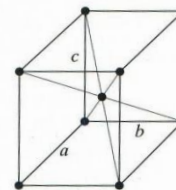
SIMPLE TETRAGONAL (*P*)



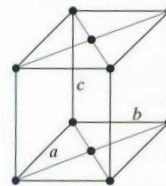
BODY-CENTERED TETRAGONAL (*I*)



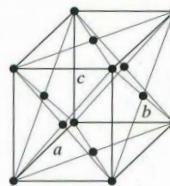
SIMPLE ORTHORHOMBIC (*P*)



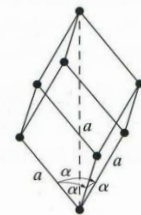
BODY-CENTERED ORTHORHOMBIC (*I*)



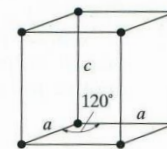
BASE-CENTERED ORTHORHOMBIC (*C*)



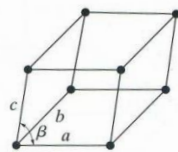
FACE-CENTERED ORTHORHOMBIC (*F*)



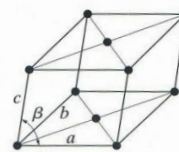
RHOMBOHEDRAL (*R*)



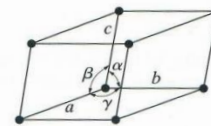
HEXAGONAL (*P*)



SIMPLE MONOCLINIC (*P*)



BASE-CENTERED MONOCLINIC (*C*)



TRICLINIC (*P*)

Sometimes when we add extra atoms to the unit cell it is equivalent to another lattice. Hence there are only **14 Bravais lattices**

Structures of Common Metals;⁹ Cubic Ones



■ Cubic Metals

- These metals are face centred cubic,
 - Aluminium
 - All nickel alloys (Li720, Inconel 718, RR1000, Waspaloy)
 - All austenitic steels
 - Copper
 - Lead
- These metals are body centred cubic
 - Ferrite
 - Martensite (actually slightly tetragonal, but usually cannot tell as a_0 & c_0 are very similar)
 - Chromium
 - β Titanium

Structures of Common Metals; Hexagonal Ones?

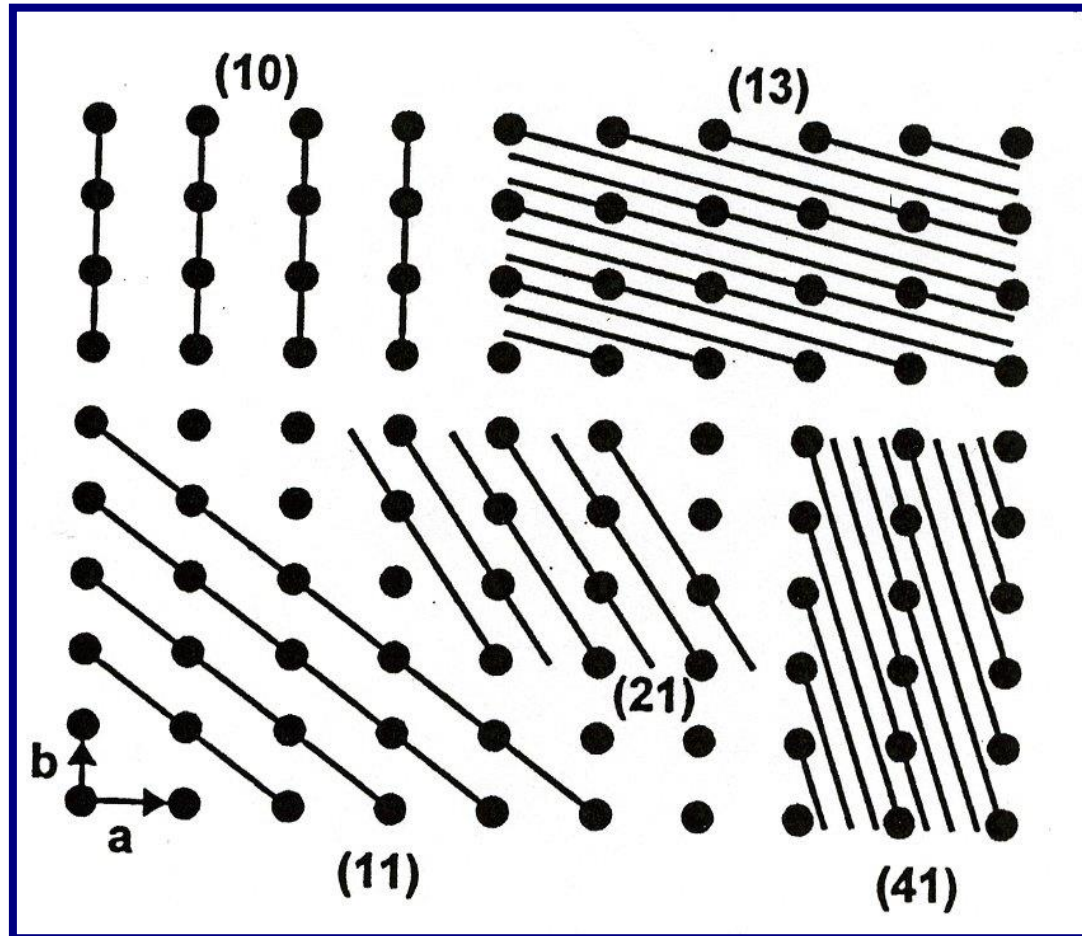
- Hexagonal Metals
 - α Titanium
 - Magnesium
 - Zirconium
 - Zinc



Crystal Spacings, d -spacings

- A crystal consists of lots of unit cells packed together to form a regular, repeated array. From this we can see that there will be planes of atoms, called lattice planes.
- The spacing of these lattice planes are called d -spacings
- d -spacings are measured in Ångstroms, 10^{-10} m
- When using any diffraction method we **measure these d -spacings**. Let's have a look at what some of them look like.

Crystal Spacings, d -spacings



A 2D representation of a simple cubic lattice, the atoms are arranged to form planes

We measure d -spacings using **Bragg's Law**

How do we label the planes? We use their **Miller Indices** $\{hkl\}$

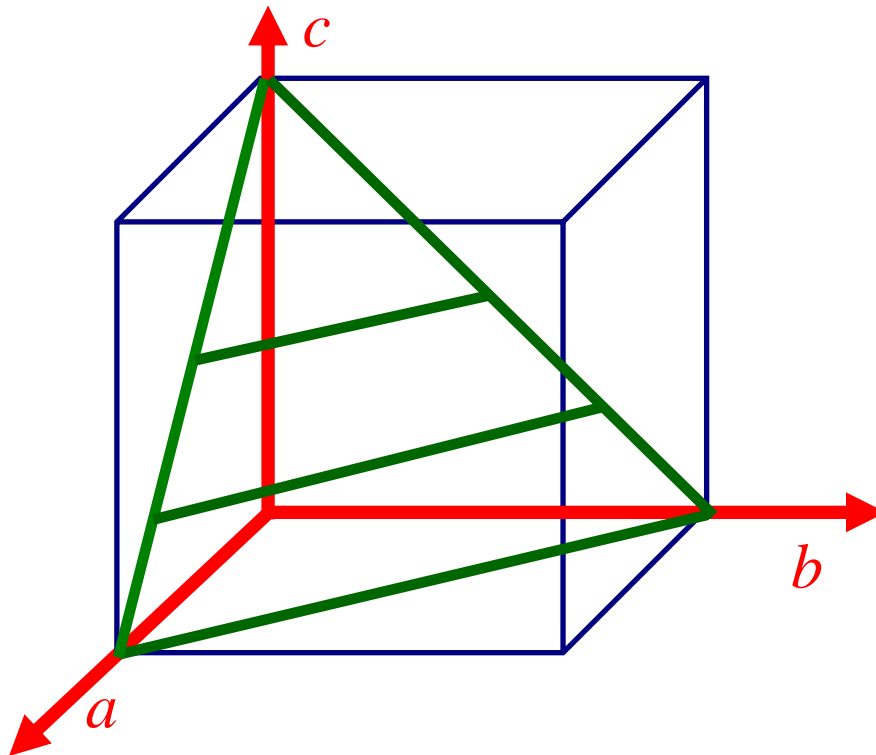


Firstly, Labelling the planes in a Crystal Structure/Unit Cell : Miller Indices

- Miller indices are very easy!
- They allow us to specify the location of a plane or, more commonly, a set of planes in a crystal structure.
- We need to know the basics of Miller indices so that we can describe our methods precisely
 - The trick is to remember that they are small, whole numbers
 - There are 3 Miller indices $\{hkl\}$, though in hexagonal systems 4 are sometimes used $\{hkil\}$.
 - They specify the points at which a plane in the crystal intercepts the axes of the unit cell.

Labelling the Planes in a Unit Cell : Miller Indices

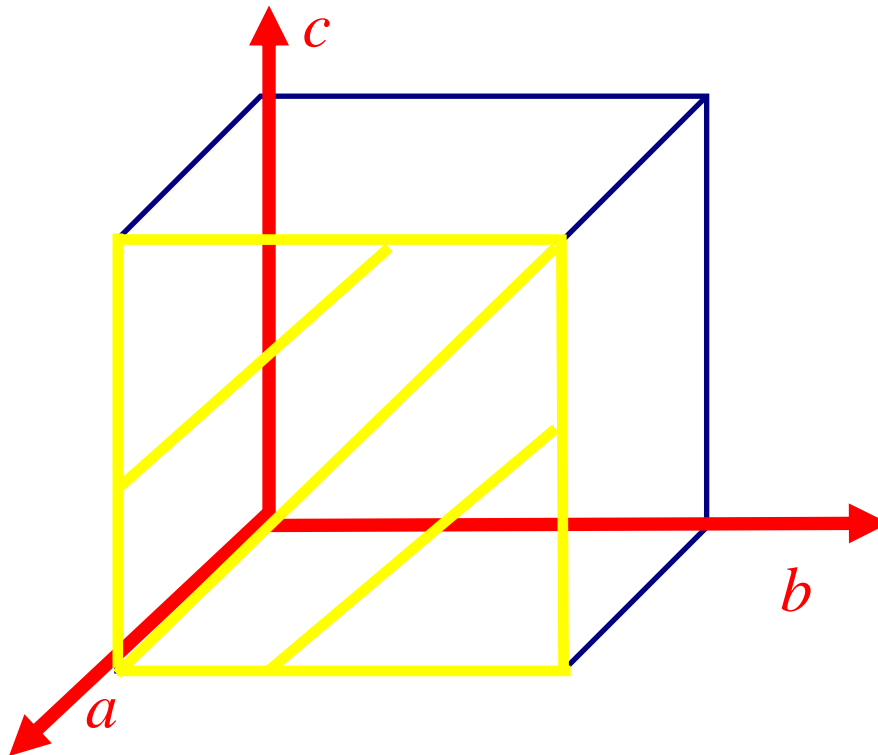
Let's draw an example of Miller Indices, using a cubic unit cell



- This is a cubic unit cell, the axes are a, b & c.
 - The plane labelled in yellow intersects each of the axes at one unit cell length.
 - The Miller indices are {111}.
- This is easy! However...

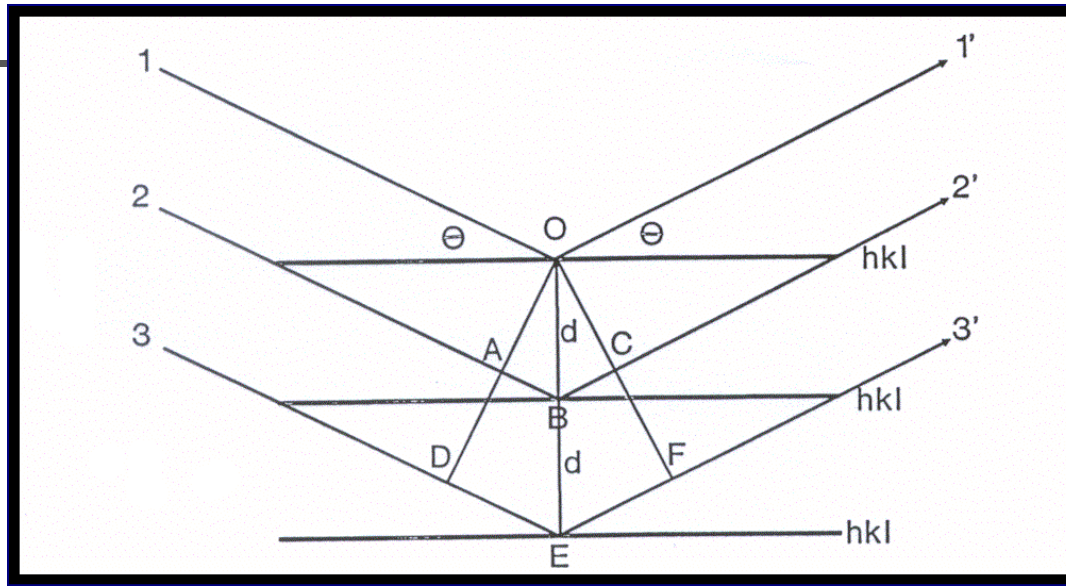
Miller Indices

Another example, again a cubic unit cell



- What do we do here?
- The plane labelled in yellow intersects the a axis at one, and b and c at infinity.
- Miller indices are small whole numbers. What do we do?
- Take reciprocals!
- The Miller indices are $\{100\}$.

Bragg's Law



$$2d_{\{hkl\}} \sin\theta = n\lambda$$

- $d_{\{hkl\}}$ spacing of the set of lattice planes with Miller indices $\{hkl\}$ (Å)
- θ Bragg Angle, angle of incidence & of reflection (degrees)
- n Order number, usually omitted
- λ Wavelength of incident X-rays (Å)

If the d -spacing changes (due to stress), the position of the Bragg reflection (2θ) will change (**very slightly!!**) and we can measure this.

Normally diffraction angles are expressed as 2 x the Bragg Angle, 2θ for reasons of diffraction geometry & tradition

Which Materials Can We Measure?



- Works on any poly-crystalline solid which gives a **high angle Bragg reflection**.
 - Metals
 - Ceramics (not easy!)
 - Multi-phase materials
- Not usually applied to polymers, as no suitable reflections. Can add a metallic powder, reported in the literature... never tried it... there are chemical methods.

Why use the $\sin^2\Psi$ Method?

😊😊😊, The Advantages

- *Most Important*

- A stress free d -spacing is NOT required for the bi-axial case which is almost always used

- *Other advantages*

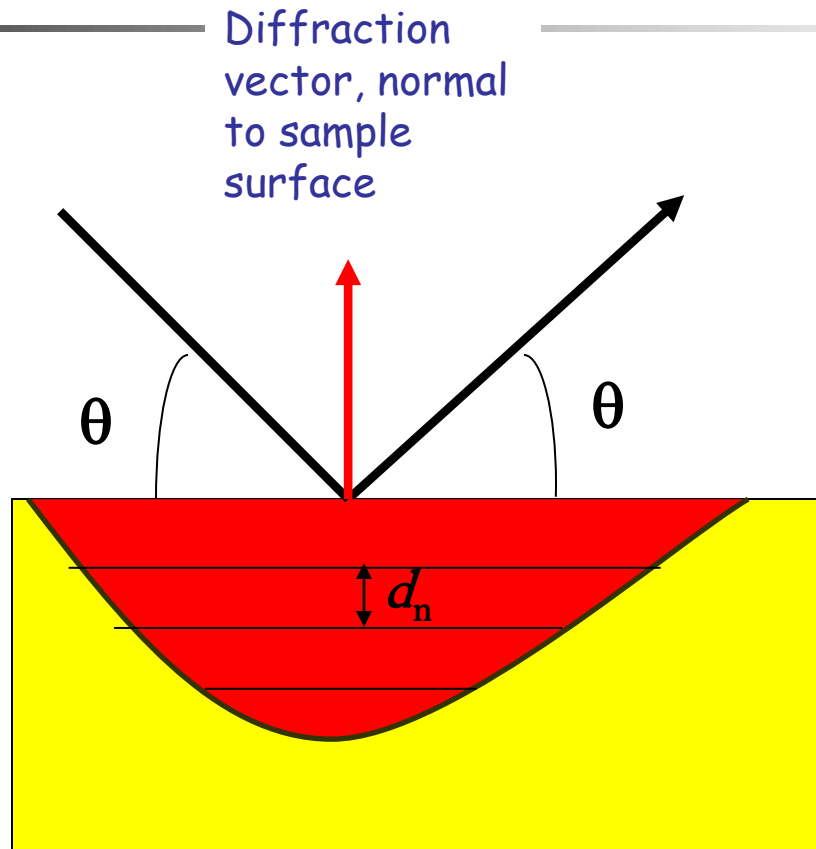
- Low cost (compared with neutrons and synchrotrons, but not hole drilling).
- Non-destructive, unlike hole drilling.
- Easy to do & fairly fool proof (if you are careful!!).

Disadvantages ☹️☹️☹️

■ Most Important

- Surface method only, X-ray beam penetration depth 10 to 20 microns, at best.
- For depth profiling must electro-polish, gives 1-1.5mm (messy and destructive)
- *Other Disadvantages*
 - Affected by grain size (>~80 to 100 μ problem in welds!!), texture (preferred orientation) and surface roughness.
 - Doesn't work on amorphous materials (obviously!!).

How the $\text{Sin}^2\Psi$ Method Works Sample in "Bragg Condition"

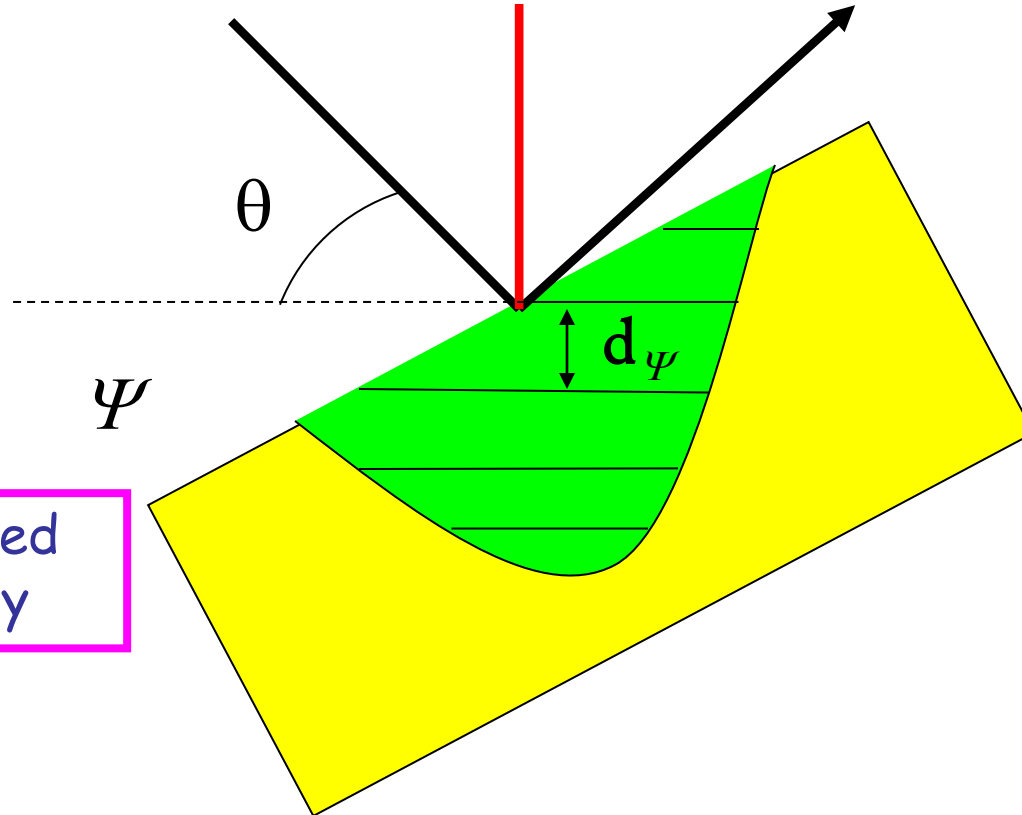


We measure the d -spacing with the angle of incidence (θ) and the angle of reflection of the X-ray beam (with respect to the sample surface) equal. These planes are parallel to the free surface & unstressed, but not unstrained.

Also called focussed geometry

How The $\text{Sin}^2\Psi$ Method Works

Diffraction vector, titled with respect to sample surface



Tilt the sample through an angle Ψ and measure the d -spacing again. These planes are not parallel to the free surface. Their d -spacing is changed by the stress in the sample.

Defocused geometry

How the $\text{Sin}^2\Psi$ Method Works

- We incline/tilt the sample by a series of steps in the angle, ψ .
- For each new value of ψ we measure the d -spacing, d_ψ

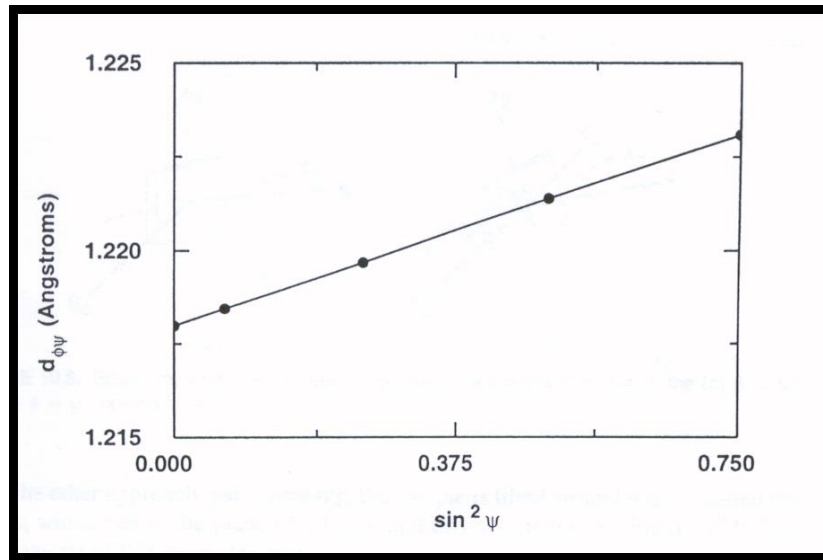
We use a range of values of Ψ (called offsets).
for example, from 0 to 45° in steps of 5°

- We tilt the sample through a series of values of the angle, Ψ , to measure the magnitude of the normal & shear stresses
- We **rotate** the sample through an **angle**, ϕ , to determine the directions of the principal stresses.
- We use the results to plot a graph, the $\text{sin}^2\psi$ plot

The $\sin^2\Psi$ Plot, We Plot $d\{hkl\}$ Against $\sin^2\psi$

We can plot d_ψ against $\sin^2\psi$ & again obtain the stress from the gradient, where

$$m = d_n \frac{(1+\nu)}{E} \sigma_\phi$$



- In this example the d -spacing decreases with $\sin^2\psi$ the stress is **compressive (planes pushed together)**
- When we are using a laboratory X-ray source a stress free standard is not required as the "approximate" value of d_0 is obtained from the intercept of the fitted line on the vertical axis of the graph
- Note the points on the graph are usually much more scattered than in this "ideal" example!



No Stress Free d -spacing Needed, The Approximation

- The depth of penetration of the X-ray beam in the sample is small, typically $< 20\mu$ & the absorption is exponential
- We can say that there is no stress component perpendicular to the sample surface, which is a free surface.
- Effectively, we can use the d -spacing, measured at $\psi = 0$ as the stress free d -spacing, d_0 .
 - This is the d -spacing of the planes parallel to the sample surface & is the intercept on our $\sin^2\psi$ plot more about this later
- A reasonable approximation!! The error is $< 2\%$, certainly less than trying to make a stress-free standard!!!

Radiation Selection

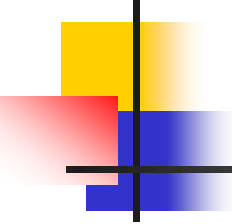
Choice of X-Ray Tube (Wavelength)

- **ALWAYS** check what other people have done in the past as, generally, measurements on different planes with different wavelengths are not comparable.
- 3 Considerations
 - (1) Dispersion
 - (2) Fluorescence
 - (3) Choice of crystallographic plane

Radiation Selection

Choice of X-Ray Tube (Wavelength)

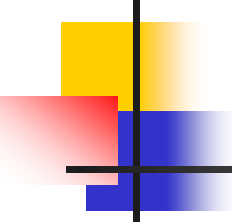
- We can measure the stress in a **variety** of materials (i.e. ferrite, austenite, nickel, aluminium, corundum, etc) using the same diffractometer, by **changing the X-ray tube & consequently the wavelength of the X-rays**.
 - Most residual stress diffractometers will have a selection of X-ray tubes available.
 - How do we choose?????



Choice of X-Ray Tube

(1) Dispersion

- We need a 2θ angle, ideally $> 140^\circ$
 - The change in d -spacing due to strain is very small, typically in the third decimal place.
The dispersion of the diffraction pattern is much greater, at high 2θ angles.
 - The small changes in d -spacing can only be detected easily at angles $> 125^\circ$.



Choice of X-Ray Tube

(2) Sample Fluorescence

- If the K- α 1 component of the incident X-ray beam causes the sample to emit its own fluorescent X-rays, **DO NOT USE IT!**
 - X-ray penetration depth will be **very small** <5 microns & \therefore **NOT** representative of the bulk.
 - Peak to background ratio will be terrible
 - May damage sensitive X-ray detectors

Choice of X-Ray Tube

(3) Choice of Crystallographic Plane

- If the sample is textured (preferred orientation) select a set of planes with a **high multiplicity**.
- However, most importantly....

Choice of X-Ray Tube (Most Importantly)

- For **ACCURATE COMPARISON WITH OTHER PEOPLES DATA** check which planes have been used historically!!
 - Measurements made on planes with different Miller {hkl} indices are not usually comparable.
 - Measurements made with different X-ray tubes are also not usually comparable.
- It's good to copy what others have done in the past 😊, plagiarism is a good thing in XRD stress measurement! 😊 😊 😊 😊



Conclusions: Choice of X-Ray Tube, Summary

- *Ask!!!!*
 - Find out what others have done
 - Avoid X-ray tubes where the $K-\alpha_1$ line causes fluorescence.
 - Select a reflection at an angle greater than $125^\circ 2\theta$... if possible.



Sample Preparation

- The main **DISADVANTAGE** of the $\sin^2 \Psi$ method is that it is so surface sensitive!! We must not introduce additional stresses!!
- Do as **LITTLE AS POSSIBLE**,
 - **DON'T**
 - Saw samples
 - Grind
 - Polish
 - Wire Brush
 - Can measure through thin paint & oxidised layers, but beware interfering reflections
 - Better than trying to remove it.
 - Can sometimes measure the coating itself!

Depth Profiling/Layer Removal




- We cannot remove material mechanically as this totally changes the stress state We have to depth profile, chemically by **electro-polishing or chemical immersion etching**
 - **Electro-polishing**
 - Damages the surface.
 - Uneven layer removal.
 - Must be calibrated to determine the layer removal rate for your material and the removal depth must be measured directly... can be difficult.
 - Messy, may be explosive, careful with the temperature! Bit of an acquired art
 - Don't blow yourself up!
 - We can correct for the change in stress due to layer removal

How Precise are the Results????

- Generally, there's a lot of scatter on $\sin^2\psi$ plots!
 - The error bars printed out by most PC's are just the standard deviation of the points from the line fitted by least squares and tend to under estimate the errors.
- Large error bars are not necessarily unacceptable and are due to
 - Texture, large grain size, poor peak fitting etc
 - For example, $200 \pm 50\text{MPa}$ is quite normal
 - Check the peaks on the PC screen!
- A couple of quick tricks,
 - If you change the peak fitting model; the results should not change significantly if it's a "good measurement"
 - If you remove some of the points from the $\sin^2\psi$ plot & the result changes significantly (by more than $\sim 50\text{MPa}$) the result is probably unreliable
- Check the quality of peaks!!!...more on this later

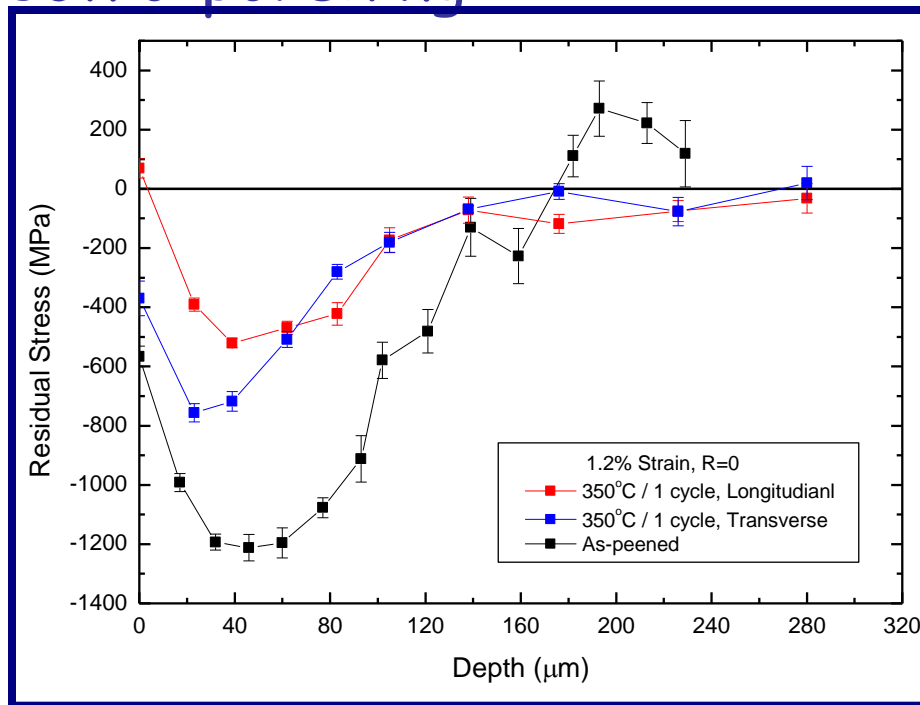
What is the Smallest Stress which we can Measure?



- Depends on the material
 - We are measuring strain so it's easier with softer materials which have higher strains for a given stress
- Values of less than $\pm 50\text{MPa}$ can usually be thought of as zero...
 - However, this does depend on your XRD machine
 - To confirm such low readings, make several measurements on the same point, in the same direction & see if they all come out with the same sign (i.e. all compressive).

A Typical Example of a Stress Profile in a Shot Peened Sample

- A shot peened surface, depth profiled by Electro-polishing





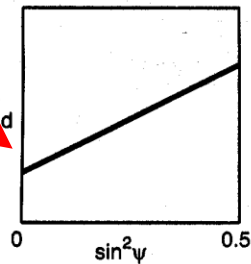
Problems with Materials

- *The $\sin^2\psi$ method works on isotropic materials where the grains are small & randomly orientated!!!! There are not many of these about in the Engineering Industry ☹️☹️☹️☹️*
 - Most components have been rolled, forged etc. which affects the orientation of the grains in the material, this is referred to as "preferred orientation" or "texture"
 - The method is also affected by grain size. If the grains are $>\sim 80-100\mu\text{m}$, the results may be unreliable
 - Rough surfaces (visible topography) can also be a problem
- Usually, these can be overcome by careful data collection & processing...but not always, sometimes you just have to try it

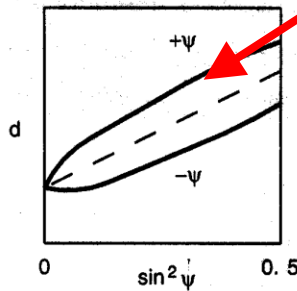
Problems...with the Material!!!

Shear Stress,
the "splitting"

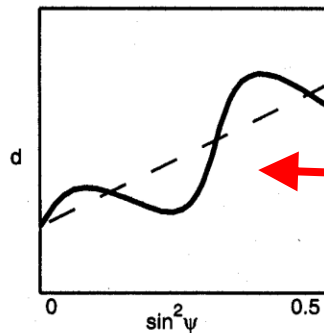
Good
one



(a)



(b)

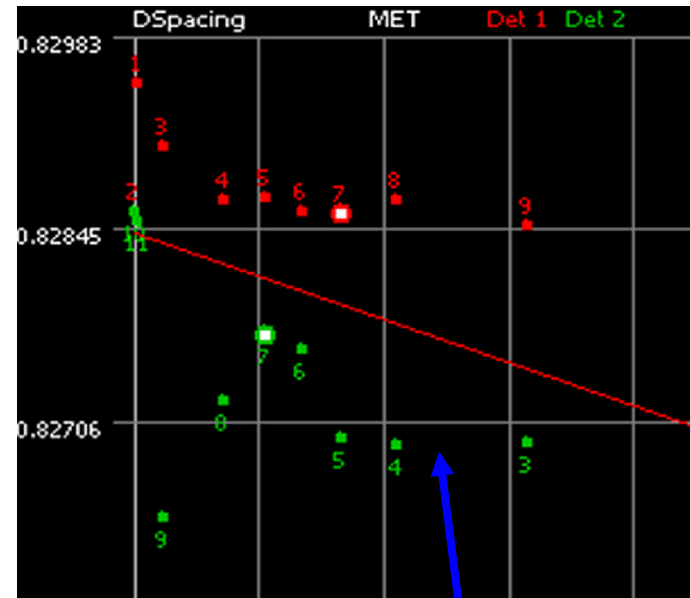
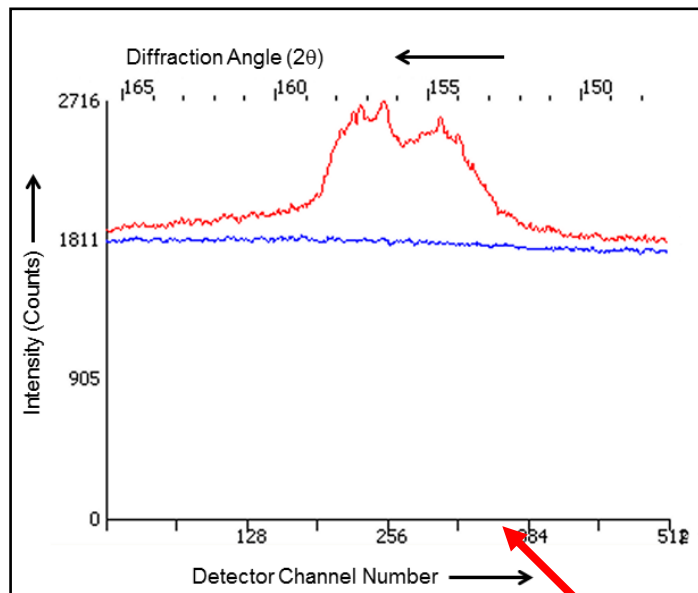


Also, when the grain size is large $> \sim 80-100\mu$ the points are scattered randomly on the $\sin^2 \psi$ plot

Texture, the
"wiggle"

A few more details

Samples with a Large Grain Size



If the grain size is $\gg 80\text{-}100\mu$ the reflections are distorted & the points on the $\sin^2\psi$ plot are scattered randomly due to inter-granular strain of type 2 strain

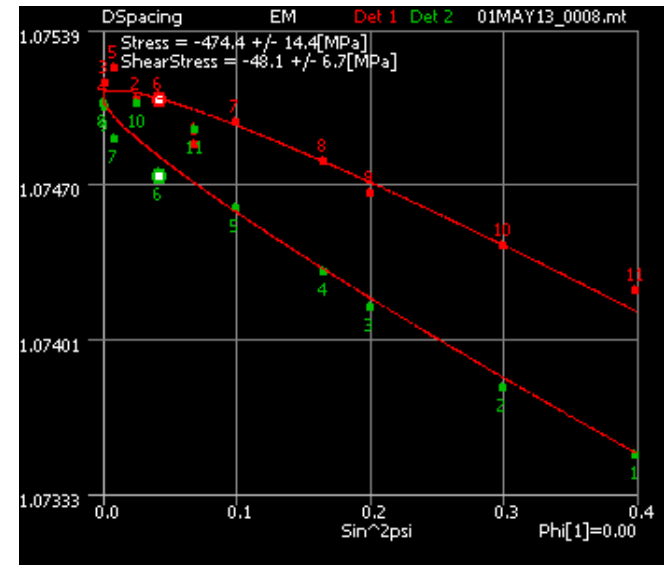
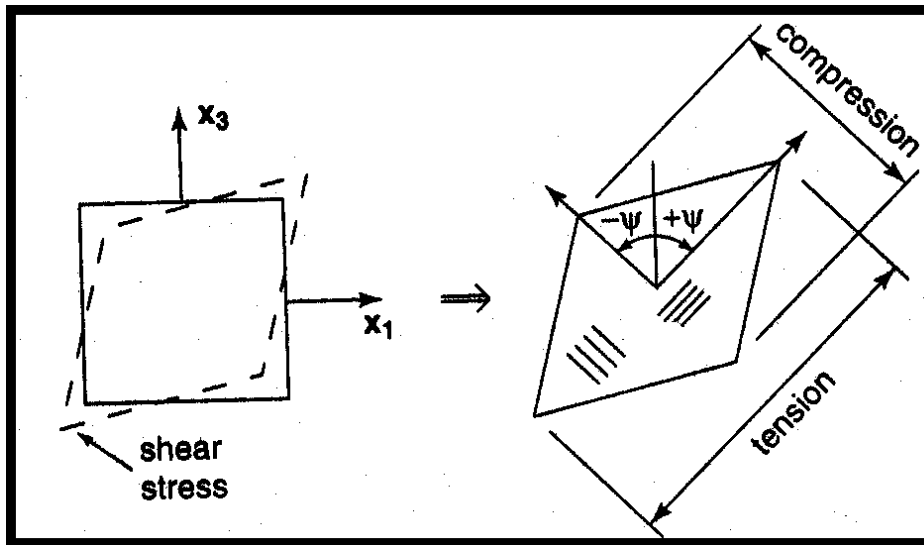


Geometric Problems; Curved Samples

- Samples which are not flat, curved samples etc.
- The measurements made with positive and negative ψ offsets will split on the $\sin^2\psi$ plot
 - Looks like a shear stress
- Solution
 - Restrict the irradiated area, if possible or remove the error mathematically; it's a function of $\sin^2\psi$

Shear Stresses, $\sin^2\Psi$ Splitting

- Positive and negative Ψ give different results when a shear stress is present (or sample is curved or not correctly positioned, always check!).



σ_{13} & σ_{23} are no longer zero



Conclusions

The $\sin^2 \Psi$ method works well if you are careful.

- Don't scrape or scratch the sample.
- Check to see what's been done by others,
 - *Don't reinvent the wheel.*
- Choose you X-ray tube with care.
- Position the sample carefully,
 - Think about the directions you wish to measure.
- Check you peak fitting.

■ ***Most importantly***

Do the results make sense??????

Thank you for your
Attention
Happy Landings! Questions???



This one is
made of
wood!!!