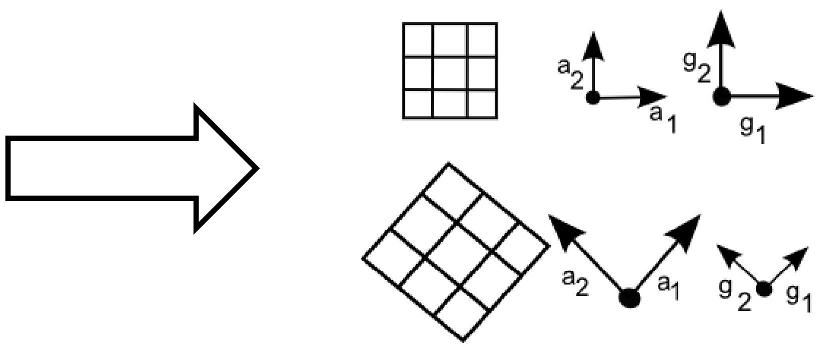
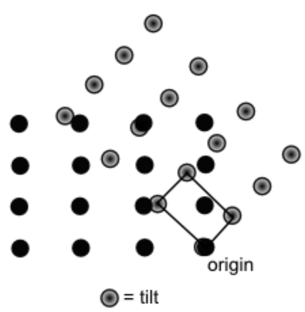
Topic 4-2: Powder Diffraction

Summary: In this video we discuss powder diffraction as if the incoming wave interacts with all powder grains at once. We then can treat reciprocal space as a set of concentric spheres. In order to scan through and locate these spheres we use an ω-2θ scan which grows $\Delta\vec{k}$ radially by moving the source and detector together.

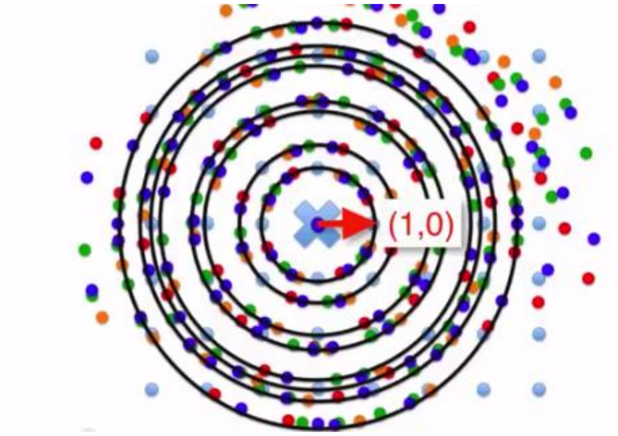
- Moving on to polycrystalline materials
- Start with 2 single crystals



- Can treat the reciprocal lattice of these as a superposition of each individual reciprocal lattice since the incoming wave interacts with both crystals at the same time



- Instead of just two grains, powders are made up of many grains
- All these randomly oriented grains interact with the incoming wave together
- So we move to effective reciprocal space made up of concentric spheres (OK, they're circles here) which we can label using reciprocal lattice vectors

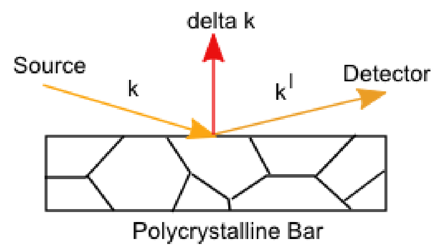


- Can determine the distance to each sphere using h and k values
  - $d = \sqrt{h^2 + k^2}|g_1|$ for a square in 2D

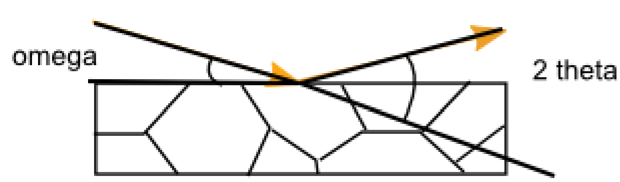
- **Example 1:** Now try a tetragonal crystal where $|\vec{a}_2| < |\vec{a}_1|$
- In reciprocal space $|\vec{g}_1| < |\vec{g}_2|$
- Moving the source and detector we will first see the (1,0) reflection, since $|\vec{g}_1| < |\vec{g}_2|$, then the (0,1), (1,1), (2,0) and (0,2) peaks in that order, all at separate peaks and therefore progressively larger spheres

Develop a sketch of reciprocal space for this 2D tetragonal system

- When dealing with nested spheres, the most efficient way to scan through the spheres is to just move $\Delta\vec{k}$ in the radial direction (away from the origin)
    - Any $\Delta\vec{k}$ angle in reciprocal space will have the same peak information, provided the sample is truly a random powder.
- Not all crystal grains will have reciprocal lattice points that satisfy constructive interference at each sphere, but some will. Thus, the intensity of the diffracted beam is much smaller than from a single crystal illuminated with the same source power.
- Need to figure out how to move $\Delta\vec{k}$ radially
- Start with below configuration



- If we move the source and the detector together this gives radial growth of $\Delta\vec{k}$
- Usually define angles ω and 2θ as below so when the source and detector move you change ω-2θ
    - Call this an ω-2θ scan



- Distance between reciprocal lattice spheres depends on crystal system
    - Gives rise to different intensity patterns

Questions to Ponder

1. We assumed that all grains were randomly oriented. What happens if a significant portion of the grains are oriented in one direction? How would this impact the intensity pattern?

2. For a powder with a 2D hexagonal structure, identify the first 4 peaks to occur in a ω-2θ scan.

3. Compare a square crystal to a hexagonal crystal where both $\vec{a}_1$ lengths are equal. What in the intensity pattern would indicate which is which?

4. You scan an unknown powder with x-rays of wavelength 1.5 angstroms. You see peaks at 10^o 2θ. What can you deduce about the structure?

5. When we use an x-ray source it produces incoherent radiation. This means that the photons are not in phase with one another. We are really hitting a chunk of copper with electrons and having photons emitted. So how do we get interference from an incoherent source?