X-Ray Diffraction and Crystallography

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Abstract

- Since the middle of the 20th century, X-Ray diffraction has been a popular tool used by researchers to gather data on many types of materials that exhibit both crystalline and semi-crystalline behavior.

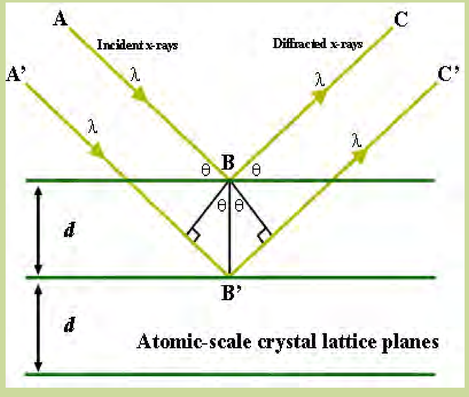
- The main objective was to explore the capabilities of the X-Ray Diffractometer by learning how to operate the machine and then interpret collected data.

- The diffractometer can perform scans on epitaxial thin films, crystalline powders, polymers, single crystals, and other forms of materials. Scans were performed on many of these types of materials.

- Researchers have many tools available to them to make this process easier. We used Rietveld Refinement, a popular technique used for curve fitting, to model different phases of Tantalum Boride, a material most well known for its remarkable hardness.

- We use these models to determine important information about the phases present, such as lattice parameters, phase composition, and abundance.

Introduction



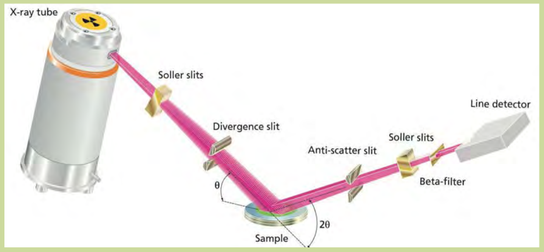
$$d\sin\theta = n\,\lambda$$

- X-Rays, like other types of radiation, obey Bragg's Law.

- When x-rays of a fixed wavelength strike the crystal at very precise angles, strong reflected x-rays are produced. This may result in constructive interference occurring.

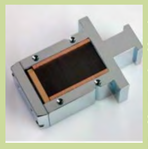
- The differences in the travel path must be equal to integer multiples of the wavelength.

Methodology

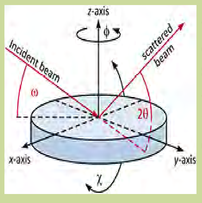


- X-Ray diffraction devices are composed of three main parts: An X-Ray tube and accompanying incident optics, a detector and accompanying diffracted optics, and a stage upon which the sample to be scanned is placed.

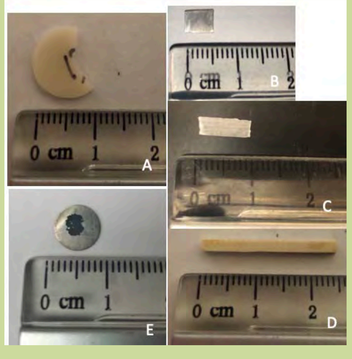
- Divergence slits narrow the beam and determine the irradiated length on the sample.
- Two kinds: Fixed and Variable
  - Fixed: Constant irradiated volume
  - Variable: Constant irradiated area



- Soller slits control the irradiated width on the sample and reduce background.
- Stray photons are absorbed by the copper plates, and so only photons that are traveling at shallow angles with respect to the slits pass through

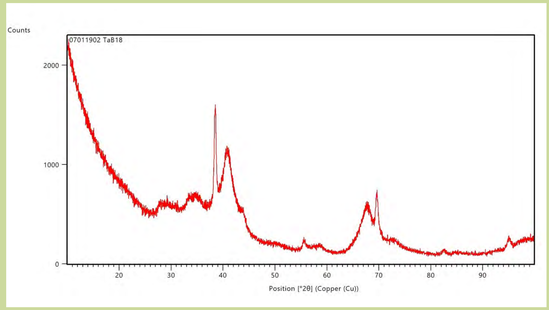


- Most stages use finely tuned motors to allow the sample to rotate through several different axes
- Stages can rotate so that different planes of reflection in the crystal lattice can be examined .

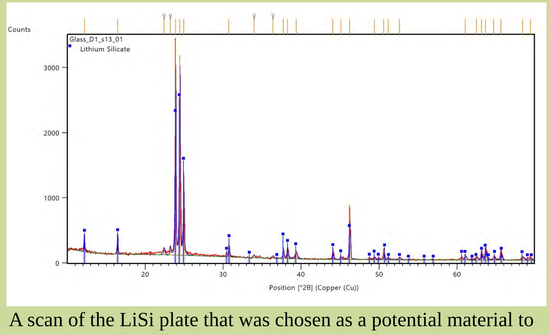


- Some examples of materials scanned using XRD. A: Fragment of LiSi dental material. B: Substrate on which crystalline material is grown. C: Small piece of electrospun polymer. D: Block of TiO_2 dental material. E: TaB and TaB_2 phases formed on Ta substrate.

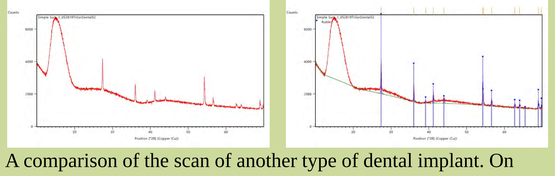
- After the CCD converts collected X-Rays to electrical signals, they are sent to a computer for recording. A diagram is created that records the peak intensity of reflected X-Rays at certain positions. The position (in terms of 2θ), width, and height of these reflections can be used to determine key information about the material's structure and composition



An image depicting the raw data of a scan of different phases of Tantalum Boride that were created using CVD on a Tantalum substrate. The peaks indicate the presence of crystal planes oriented such that many of the incident X-Rays are reflected into the detector. Further work can be done to determine the exact positions and orientations of the crystal planes in space.

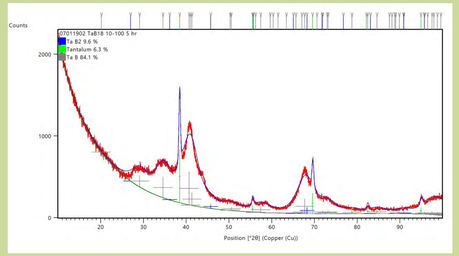


A scan of the LiSi plate that was chosen as a potential material to be used as a dental implant. HighScore Plus was used to treat the data for pattern assignment. Before any assignments can be made, the background (green line) is determined, and peaks are defined according to specifications made by the user.



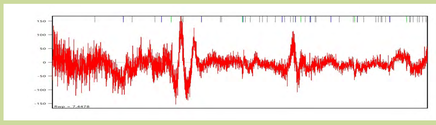
A comparison of the scan of another type of dental implant. On the left is the raw data, and on the right is the same scan after treatment and pattern assignment.

Results and Discussion



- A depiction the scan acquired from TaB18, only now the data has been refined using the Rietveld method. Rietveld refinement is a process that allows researchers to model and characterize their materials.
- After a material is characterized and patterns are assigned, the user can run the refinement program.
  - This varies multiple parameters such as background, unit cell dimensions, peak shape functions and coefficients for each phase identified. After the refinement process is completed, HighScore will display each identified phase along with its relative abundance, lattice constants, and other information

TaB phase Unit Cell: a = 3.266 Å, b = 8.786 Å, and c = 3.204 Å.
TaB_2 phase Unit Cell: a = b = 2.863 Å, and c = 3.294 Å.



RWP for this particular sample is approximately 7.4478

Conclusions and Future Work

- X-Ray diffraction makes use of Bragg's Law to examine crystal lattice structures.
- Collected data is sent to HighScore Plus for interpretation.
- Rietveld refinement allows researchers to model their materials.
- Future work involves learning more about the different types of scans
- Single Crystal work and Reciprocal Space Maps
- Improve refinement process.

Acknowledgements

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