1. Introduction

Around the turn of the 20th century, many physicists were trying to understand the nature of both cathode-rays and X-rays (discovered during cathode-ray investigations by Rontgen in 1895, who won the first Nobel Prize ever in 1901 for this work) [1]. As had been a theme throughout 20th century physics, there was much debate as to whether these different rays resulted from a flow of particles or the propagation of waves. With a background in optics and an awareness of the contemporary view of crystal structures at the time, Max von Laue realised that the expected wavelength of X-rays (if indeed wave-like in nature) should be comparable to, but less than, the expected periodicity of the atomic arrangements in crystals [2,3]. Hence, crystals might be used as effective diffraction gratings for X-rays and irradiating them might therefore allow distinct interference maxima and minima to be found in the intensity of the diffracted X-ray beams. Initial experiments, by von Laue and his colleagues Friedrich and Knipping, used copper sulphate as the crystal of choice and were unimpressive. However, hints of diffraction spots on the photographic plates prompted them to further optimise their experimental geometry and change from copper sulphate to regular cubic zinc sulphide crystals. With the X-rays passing through one of the square faces of zinc sulphide, a stunningly regular array of diffraction spots was observed which appeared to be entirely consistent with the four-fold symmetry of the macroscopic crystal perpendicular to the beam (figure 1) [4].
Figure 1. One of the original X-ray diffraction patterns taken by von Laue and his colleagues on zinc sulphide [2].

These experiments led to develop further usage of crystals to investigate properties of X-rays, and use of X-rays to determine the periodicity, symmetry and structure of crystals. Diffraction patterns generated from crystals are best rationalised by considering the reciprocal lattice construction. Each set of parallel lattice planes in the crystal is represented by a vector, oriented perpendicular to the planes with which it is associated and with a magnitude equal to the inverse of the perpendicular spacing between adjacent planes in the set [5,6].

2. Methodology

The main purpose of the experimental procedure was to take Laue Photographs. They were taken using incident x-rays with a range of wavelengths. This means that a number of different lattice periodicities can simultaneously satisfy the Bragg's law for the direction of the x-ray beam and crystalline sample orientation. To make sure that the equipment was working correctly, a calibration was to be done to the equipment. The Au anode x-ray tube was set up with X-ray unit. The circular collimator and pin-hole aperture were put in their designed positions accordingly. An energy detector was placed around 10 cm away from pin-hole aperture and was attached to goniometer (figure 2).

Figure 2. Schematic illustration of the experimental geometry needed to measure the emission spectrum from a gold anode. The collimator is labelled as “b”, the attenuating aperture as “c” and the energy detector unit as “d”.

The measurement parameters were set using CASSYLAB software. The parameters were: The tube emission current = 0.1mA, the tube voltage = 35kV, factor=-2.5, measuring time=360s, multichannel measuring = 512 channels and negative pulses parameter was turned on. Overall, the set up was done so that the spectrum collection was at its best and hence the X-ray energy spectrum was recorded. Once the calibration was done, the next step was to take the Laue photographs. To capture Laue Photographs for LiF and NaCl the entire spectrum of different x-ray radiation was to pass through a thin single cubic crystal sample for both Laue pattern photographs. In order for it to happen, the energy detector and associated signal communication electronics and connections were removed, as was the beam attenuation aperture. The solid-state X-ray camera was attached to the optical rail mount that fits into system. It was placed around 2cm from the diffracting crystal.

The CT software was used to capture image. The accelerating voltage on the x-ray tube and current were held the same as for the first part of the experiment. The integration time was set to 3500ms, contrast=30% and intensity=70%. Then offset images using dark frames were created. Next, by inverting the images the Laue photographs were obtained for NaCl and LiF accordingly.

3. Results
Firstly, the recorded X-ray energy spectrum was plotted. The gamma radiation counts were plotted as a function of the photon energy. The error source in the vertical axis was accordingly the statistical square root value, as for the photon energy values, the error source was the equipment uncertainty in spatial resolution.

![X-ray energy spectrum](image)

**Figure 3.** X-ray energy spectrum of gold.

The graph obtained showed the expected presence of Bremsstrahlung Radiation as a large source of noise on the spectra. On the other hand, the spectral lines for the L shell emissions were clearly visible, to identify the sample as the gold. The peaks on the graph were correspondingly identified, and labeled as Lα, Lβ and Lγ transitions. The emitted photon energy for the peaks were in correspondence to the table values for the peaks. The results obtained for the spectrum showed that the equipment was correctly calibrated and was ready to obtain the Laue Photographs for NaCl and LiF (figure 4).
Figure 4. NaCl and LiF Laue photographs respectively.

The black dots on the Laue photographs identify the spatial distribution of the crystal’s atomic planes due to destructive and constructive interference. The spatial orientation of the patterns shows that the symmetry of the crystals has formed the black dots due to maxima intensity because of the constructive interference and that the symmetry is a four-fold symmetry. The four-fold symmetry in the crystals was formed due to cubic forms of the crystals and that the atomic planes of the crystals were perpendicular to the incoming beam in 001 direction (in Miller index notation).

4. Conclusion
To sum up, the nature of the x-ray scattering from crystal lattice planes and the pattern they form due to the scattering was investigated using two cubic crystals, namely NaCl and LiF. In order to so, an experiment was designed using an x-ray source and an x-ray camera. This allowed to investigate the internal structure of the crystals and even the reciprocal lattice itself. Prior to that, the experimental set up was to be calibrated and using a gold anode and a gamma detector instead of the x-ray camera the energy spectrum of the gold was recorded. Identifying the spectral lines and their corresponding energies and checking their obtained values with the tabulated data, the calibration was finalized. Lastly, the Laue photographs were obtained of the crystals. Both crystal’s atomic planes created a four-fold symmetry under the x-ray beam. The reason for the created patterns was identified as the constructive interference of the x-ray beam interacting with the crystal parallel to the lattice planes in the 001 direction.

References
[1] Bamatov I M and Bamatov M 2020 IOP Conf. Ser.: Earth Environ. Sci. 548 062076
[2] Eckert M 2012 Max von Laue and the discovery of X-ray diffraction in 1912 Ann. Phys. 524 A83-5 doi:10.1002/andp.201200724
[3] Bamatov I M, Rumyantsev E V and Zanilov A K 2019 The influence of biopolymer modification of mineral fertilizers on main agrochemical parameters of soil IOP Conference Series: Earth and Environmental Science 52059-64 DOI:10.1088/1755-1315/315/5/052059
[4] Von Laue M 1920 Concerning the detection of X-ray Interference Nobel Lectures The Nobel Foundation online: Available at: https://www.nobelprize.org/uploads/2018/06/laue-lecture.pdf (Last accessed: 15 September 2020)
[5] Oeckler O 2012 100th anniversary of the x-ray diffraction experiment Royal Society of Chemistry ChemComm 16
[6] Bamatov I M and Bamatov D M 2020 J. Phys.: Conf. Ser. 1515 022025