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Characterization of Materials by X-Ray Diffraction

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Abstract:- X – rays are electromagnetic radiation of wavelength about 1 A°, which is about same sise of an atom. X-ray diffraction provides information that cannot be obtained any other way. This information relates to the crystal lattice of the material and can characterize the crystalline phases present. This paper provides the background of the use and theory of x-ray powder diffraction. Here polycrystalline ferrite with the chemical composition $Ni_{0.5-x}Zn_{0.5}Ti_xEe_{2-2x}O_4$ system with x = 0.0, 0.1, 0.2, 0.3, 0.4 and 0.5 were prepared by ceramic and characterized by x-ray diffraction.

INTRODUCTION:

X – rays are electromagnetic radiation of wavelength about 1 A°, which is about same sise of an atom. They occur in that portion of the electromagnetic spectrum between gamma rays and ultra violet radiations. Thediscovery of x-rays enabled scientists to probe crystalline structure of the atomic level. X-ray diffraction has been in use , for the finger print characterization of crystalline materials and in determination of their structures. Each crystalline solid has its unique characterics x-ray powder pattern which may be used as a figureprint for its identification. Once the material has been identified, x-ray crystallography may be used to determine its structure. X-ray diffraction is one of the most important characteristics tools used in solid state physics, chemistry and materials science.

Bragg's Equation of X – ray Diffraction:

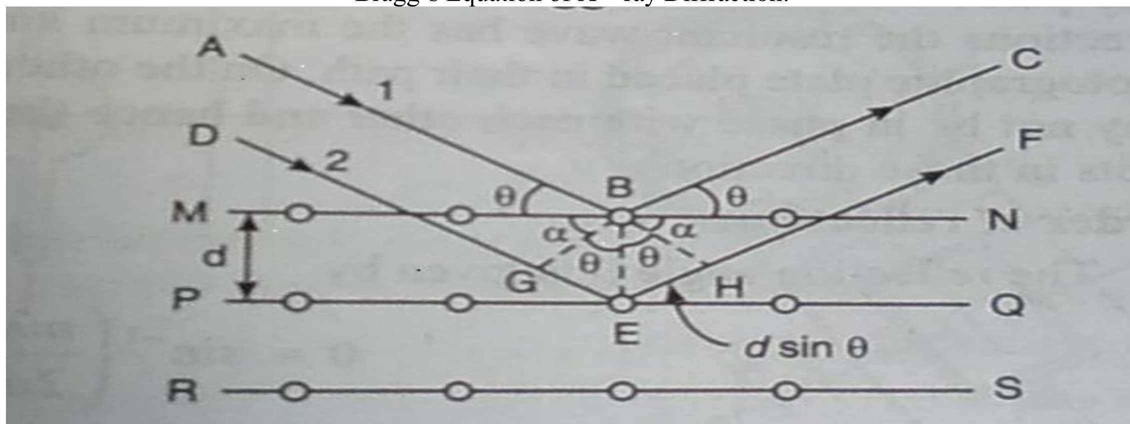

Figure1

Diffraction of an x –ray beam striking a crystal occurs because the wavelength of x –ray beam is similar to the spacing of atoms (1AU). When an x-ray beam encounters the regular three dimensional arrangement of atoms in a crystal. Most of x-rays will destructively interfere with eachother, but in some specific directions they constructively interfere and reinforce the another.

W L Bragg showed that diffracted x-rays act as if they were reflected from a family of planes within crystals. Bragg;s planes are the rows of atoms that make up the crystal structure. These reflections were shown to only occur under certain conditions which satisfy the equation

$2dSin\ \theta = n\lambda$

Where n= is an integer

Λ = wavelength of x – rays

d = distance between atomic planes

θ = the angle of incidence of x-rays beam and atomic planes for a given wavelength of incident x-rays and interplanar spacing in a material, only specific angles will satisfy the Bragg's equation

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INSTRUMENTATION:

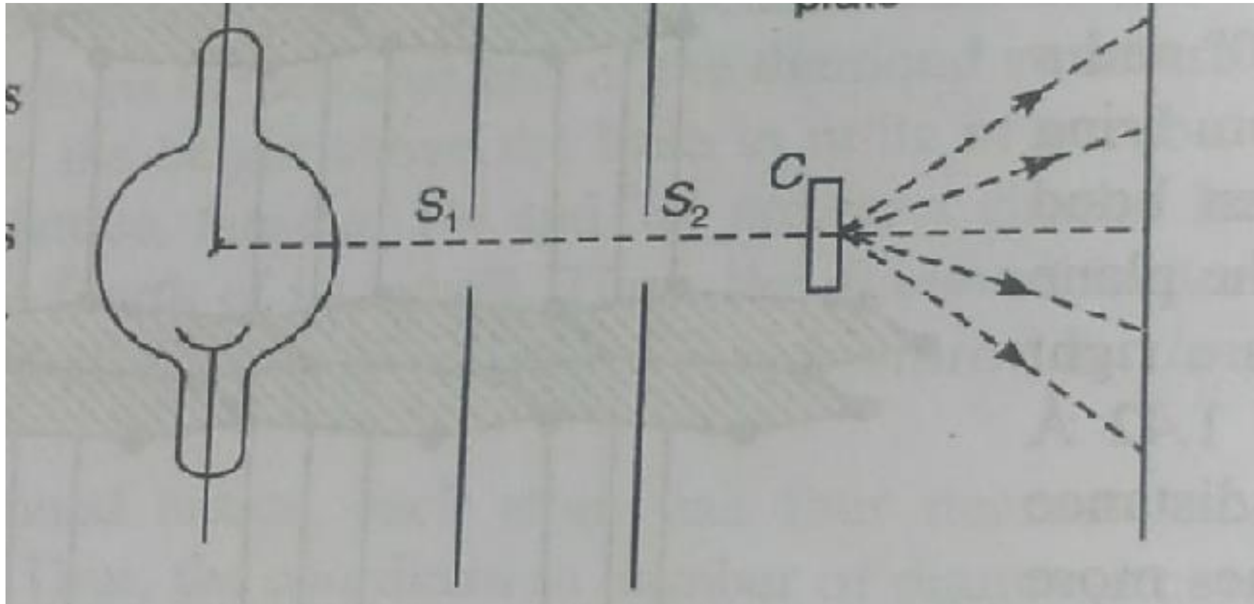

Figure2

The x-ray diffraction experiment requires an x-ray source, the sample under investigation and a detector to pick up the diffracted x-rays.

The schematic diagram of powder x-ray diffractometer is as shown in figure. The x-ray radiation most commonly used is that emitted by copper, whose characteristic wavelength of the k radiation is 1.514 AU. When the incident beam strikes a powder sample, diffraction occurs in every possible orientation of 2θ . The diffracted beam may be detected by using a movable detector. In normal use, the counter is set to scan over a range of 2θ at a constant angular velocity. Routinely the angular range of 5 degree to 79 degree is sufficient to cover the most useful part of the powder pattern. The scanning speed of the counter is usually of 2 degree per minute

APPLICATIONS:

x-ray diffraction has wide range of applications in geology, material science, environmental science, chemistry, forensic science and the pharmaceutical industry among others. Researchers use x-ray diffraction to characterize materials from a wide veriety of settings. Some examples are

1. Many plastic polymers have sme order and can be identified and studied by x-ray diffraction methods
2. Many structures such as polymers, drawn fibers, rolled and forged metals or thin films deposited by various methods show varying degree of anisotrophy. This information relating to preferred crystalline orientation textures can be quantified and evaluated by x ray diffraction methods.
3. In studies of areas affected by acid mine drainage, the identification of secondary materials and fine grained precipitates is a critical element
4. Minerals form under specific ranges of pressure and temperature. Mineralogical identification of ore minerals and associated minerals including fine grained hydrothermal alteration minerals provides evidence used to deduce the condition under which ore deposites formed and the conditions under which in many cases they were subsequently altered
5. X-ray diffraction is one of the primary tools used to evaluate the lateral and vertical variation in mineral matter amd majoe, minor and trace elements in coal beds

ANALYSIS OF X-RAY DIFFRACTION:

In the days of digital x-ray diffraction patterns, Rietveld Analysis(Rietveld 1969) has played a very important role. This a whole pattern treatment rather than a limited number of reflections of the x-ray data and it gives the type of structural analysis. Normally obtained by a single crystal diffractometer. It is originally conceived as a refinement method for crystal structures using neutron diffraction data. But today it is also used for x-ray diffraction. Briefly, the Reitveld method requires a knowledge of the approximate crystal structure of all phases of interest. The input data obtained to calculate a synthetic pattern includes the space group symmetry, number of atoms. Atomic positions, temperature factor, site occupancies and lattice parameters. The refinement is conducted by minimizing the sum of the weighted, squared differences of this calculated pattern and the observed intensities every step in a digital powder pattern. In a typical refinement, individual scale factors (related to the weight percent of each phase) and profile, background and lattice parameters are varied. Since the method uses all lines, severally overlapping reflections are not a problem. The method can obtain the following crystallographic information.

Lattice parameters: since systemetic errors (caused by sample displacement) are corrected during refinement, accurate values up to one part in 1000 can be obtained on solid samples without an internal standard. Additionally accurate cell dimensions can be computed on low symmetry materials.

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Accurate Phase quantification: scale factors are refined and are related to weight percent of each phase. Complex mixtures with overlapping reflections are quantified with high degree of accuracy (about 1 %)

Crystallite size and strain: A mathematical function is used to model the profiles and to separatr diffraction peak broadening due to size from that due to strain. Size and microstrain values are derived simultaneously from x-ray diffraction pattern.

Site Occupancies: This yields quantitative information as to the extent of solid solution or isomorphous substitution.

Atom positions: positions of selected cations in the unit cell can be computed.

X-ray Diffraction of Fewrrite System: X-ray diffraction study is carried out for ferrites $Ni_{0.5-x}Zn_{0.5}Ti_xEe_{2-2x}O_4$ with x = 0.0, 0.1, 0.2, 0.3, 0.4 and 0.5 were obtained at inter University Consortium Indore. All the samples are prepared by ceramic method and all the samples were in the form of powder and copper k radiations were used. The diffraction patterns were taken with the angle range btween 10 degree to 70 degree.

Table 1: Observed and calculated values for $Ni_{0.5}Zn_{0.5}Ee_2O_4$

hkl	220	311	222	400	422	333/511	440
d- observed	2.9737	2.5337	2.4183	2.0924	1.7138	1.6164	1.4838
D calculated	2.9695	2.5323	2.4224	2.0997	1.7144	1.6163	1.4847

Table 2: Physical constants of $Ni_{0.5-x}Zn_{0.5}Ti_xEe_{2-2x}O_4$

Composition	Moleculat wt Gm-mol	Measured density gm/cc	x-ray density gm/cc	Porosity %	Lattice parameter a A°
$Ni_{0.5}Zn_{0.5}Ee_2O_4$	237.73	4.24	5.342	20.6	8.390
$Ni_{0.56}Zn_{0.5}$Ti0.1Ee1.8O_4	237.22	4.21	5.331	21.03	8.392
$Ni_{0.7x}Zn_{0.5}$Ti0.2Ee1.6O_4	236.71	4.16	5.305	21.58	8.399
$Ni_{0.8}Zn_{0.5}$Ti0.3Ee1.4O_4	236.21	4.16	5.332	21.83	8.395
$Ni_{0.9}Zn_{0.5}$Ti0.4Ee1.2O_4	235.69	4.05	5.283	23.33	8.399
$Ni_{01.0}Zn_{0.5}$Ti0.5Ee1.0O_4	235.19	4.04	5.277	23.44	8.396

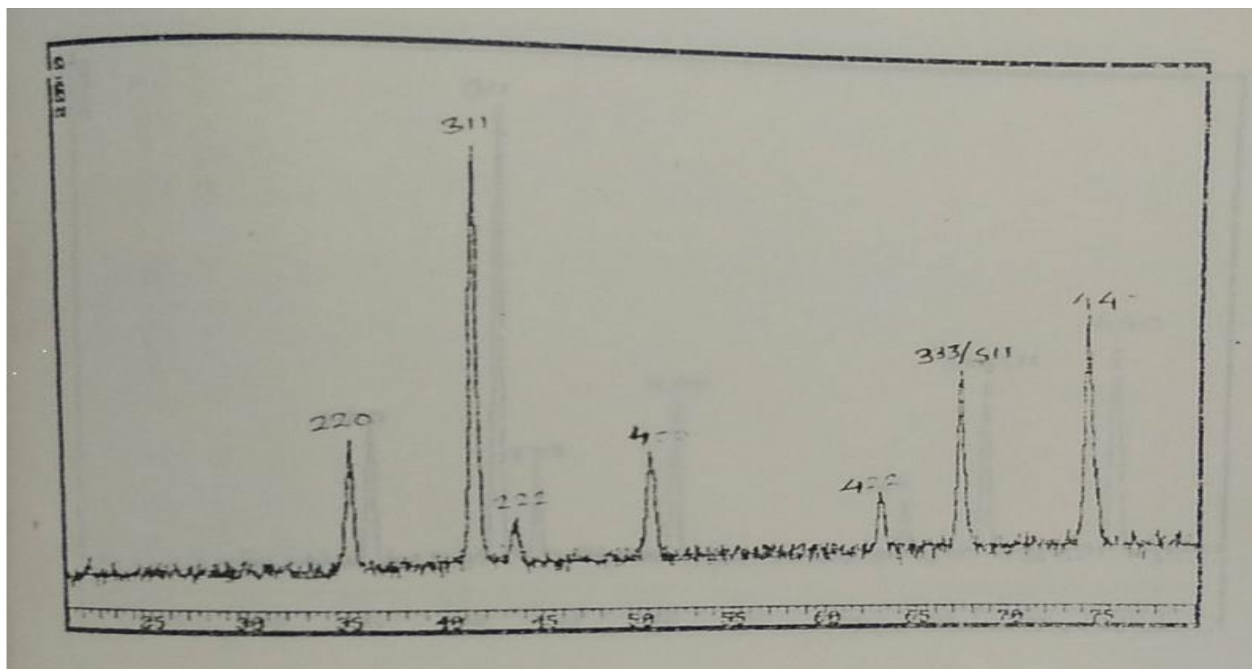

Figure3: X – ray diffraction pattern for $Ni_{0.5}Zn_{0.5}Ee_2O_4$

For the case of $Ni_{0.5-x}Zn_{0.5}Ti_xEe_{2-2x}O_4$ system. From the study it is seen that, for all the samples, the lattice constant remained constant. Here in this case the two ions of Fe^{2+} are substituted by Ni^{2+} and Ti^{4+}ions. The ionic radii of all these ions are nearly same (0.67 AU)/ \\. Therefore the constancy in lattice constant may be because of constant ionic radii. The x-ray density, actual density and porosity of samples have been determined and presented in table.

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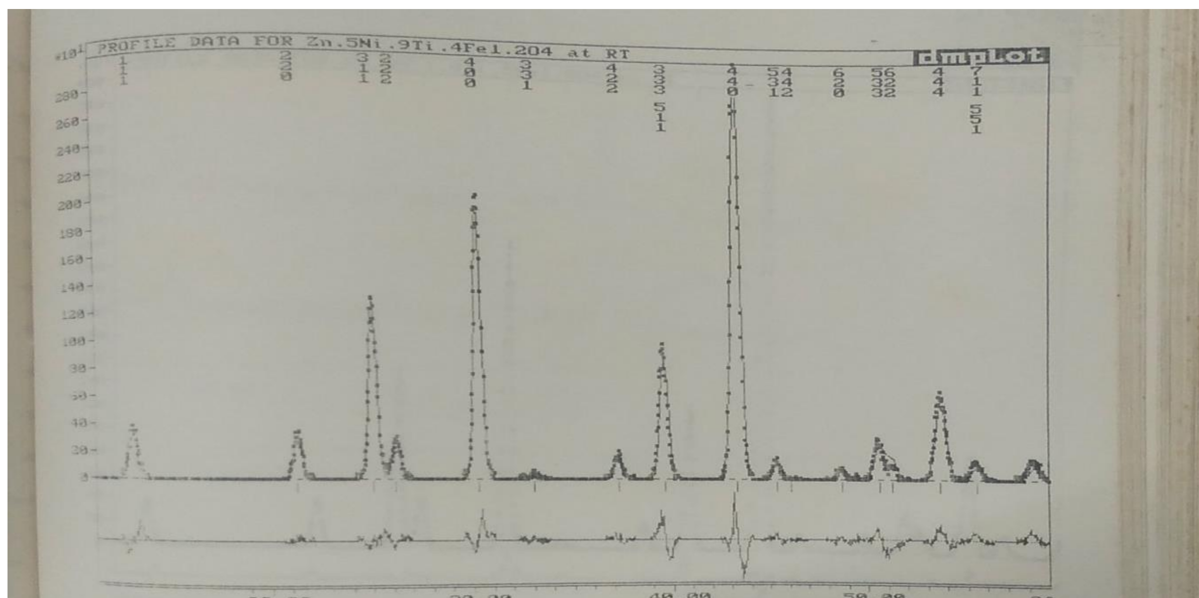

Figure4. Profile fitted pattern with magnetic reflections for $Ni_{0.5}\,Zn_{0.5}Ee_2\,O_4$

CONCLUSION:

/It is seen from above discussion that x-ray diffraction is a powerful tool for characterization of materials and much useful information can be obtained frpm it.

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