ELEMENTAL ANALYSIS OF GNIESS ROCK (METAMORPHIC ROCK) FROM KYAIKHTO TOWNSHIP BY XRD AND EDXRF METHOD

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MRes THESIS MAY, 2003

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THESIS

Submitted in fulfilment of the requirements

for the degree of

MASTER OF RESEARCH

in

PHYSICS

of the

University of Yangon

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ACKNOWLEDGEMENT

I wish to express my sincere thanks to Professor Dr Sein Htoon, PhD *Sur* CPhy FInstP *London*, Head of Department of Physics (IT) and Director of IAEA Nuclear Physics Laboratory, University of Yangon, for his kind permission to carry out this research.

I would like to acknowledge my gratitude to supervisor Dr Kay Thi Thin MSc PhD *Ygn*, Lecturer in Physics Department, University of Yangon and co supervisor, Daw Yin Pyone Chit MSc, Demonstrator, Department of Physics, University of Yangon for all their valuable guidance, helpful advice and encouragement in preparing this work.

Thanks are also due to Dr Htun Hlaing, MSc DCSc PhD *Ygn*, Lecturer in Physics Department, University of Yangon and Daw Ohmar Aung MSc, Demonstrator, Department of Physics, University of Yangon for their help and guidance with the thesis research.

Finally, I am deeply grateful to my parents for their support and encouragement without which this dissertation will not be possible.

ABSTRACT

Gneiss rock (Metamorphic rock) sample is analyzed by energy dispersive x-rays florescence method and x-rays diffraction method. The contamination and elements in two methods are compared.

CHAPTER 1
INTRODUCTION

Nuclear Physics techniques often have a great advantage over traditional chemical technique for the determination of elemental composition of minerals. X-rays discovered by Roentgen in 1895 have been widely used in the scientific researches and the medical purposes. The x-ray fluorescence spectrometry is one of the well known nuclear analytical techniques and it can be applied to the determination of elements in materials. Two different approaches have been developed, known as Wavelength Dispersive X-ray Florescence (WDXRF) and Energy Dispersive X-rays Fluorescence (EDXRF) methods.

In the Wavelength Dispersive Florescence (WDXRF) spectrometry, the characteristic secondary (fluorescent) x-rays induced in a sample are diffracted on a crystal and separately detected as a function of their wavelength (or) energy at a certain angle according to Bragg's Law.

Energy Dispersive X-ray Fluorescence (EDXRF) is a non-destructive testing methods and a powerful analytical tool for the qualitative and quantitative determination of almost all chemical elements in a sample. The application of x-rays for elemental analysis was possible due to the observation of characteristic x-ray emission spectra by Barkla and the establishment of the relationship between x-ray frequency and atomic number by Moseley x-ray fluorescence analysis (XRF) was introduced into elemental analysis around 1950.

Initially all XRF apparatuses were equipped with wavelength dispersive (WD) detector system in which x-rays are separated by a selective single crystal and recorded by a proportional photon counter. For the solid state Si (Li) detectors of sufficient resolution become available EDXRF came into existence in 1966. In this type of detectors, a voltage pulse whose amplitude is proportional to the energy of the incoming x-ray photon is produced. There are no fundamental differences between WD and EDXRF apart from the detection system.

EDXRF is well established technique for the elemental analysis. The qualitative element analysis is easy due to the simplicity of the x-rays spectra in which the characteristic lines of elements are hardly influenced by chemical bonding except only for the very low atomic number elements. Moreover, the interference between the lines can be overcome with the help of spectral fitting. The analyzed sample can be presented in various forms i.e., solid, liquid, powder, gas, slurry. The amount of sample material required for an accurate quantitative analysis can be less than a few milligrams with conventional excitation.

X-ray fluorescence covers nearly all the elements (Z>5) in the periodic table. The technique also provides a nearly uniform sensitive for all elements. EDXRF is also used for analysis of small quantities of elements. In this technique, a thin sample of the material is placed in the secondary target area of x-rays tube and bombarded with secondary x-rays. A semiconductor detector, scintillation detector and proportional counter can detect the secondary characteristic x-rays. Identification of the element in carry out in spectrum analysis where different energies are shown for different elements and their concentration are determined from the intensity of the line. In the present work, the Gneiss rock (metamorphic rock) sample, containing the substance seemed to be the small seam of gold is analyzed with EDXRF and XRD methods, to know about the containing elements.

Gneiss rocks are metamorphic rocks. This gneiss rock is applied to banded rocks formed during high-grade regional metamorphism. Included under this heading are a number of rock types having different origins. Gneissose banding consists of the more- or-less regular alternation of schistose. The schistose layer consist of mica and or amphiboles, pyrroxene is rather rare constituent. The granulose bands are essentially quartzo-feldspathic and may or may not show a preferred orientation. Gneiss is generally fairly coarse gained rocks. The thickness of the band may vary from a millimeter or so up to several centimeters and may or may not be consistent. Various types of gneiss are recognized according to the presumed original rock or the mode of formation, e.g., paragneiss (from a sedimentary

parent), orthogneiss (from an igneous parent), Lit-par-Lit gneiss, injection gneiss and sugregation gneiss and migmartic gneiss. The term "gneissose" is essentially applied to granite in which the biotite crystals although dispersed are aligned. These gneiss rock can be use to construct the building but it is expensive because it is hard and difficult to make this rock to be plane and it is mostly use in the foundation of the road. These rocks may have been granite, which is an igneous rock, but heat and pressure and arranged alternating patterns. It contains many mineral grains. Now, we analyzed the Gneiss rock sample from the Kyautkalut village group, Kyaik Hto Township, Mon State and to know about the containing minerals.

CHAPTER 2
EMISSION OF CHARACTERISTIC X-RAY SPECTRA

2.1 X-Ray Emission

When a sufficiently energetic x-rays photon interacts with an atom several phenomena take place. One interaction involves the transfer of the total energy of the photon to one of the electrons of the atom (for example, a K shell electron), which is ejected, from its shell Fig.2.1. The ejected electron is called a photoelectron and the interaction is called the photoelectric effect.

The distribution of electrons in the ionized atom is then out of equilibrium and within an extremely short time returns to the normal state, by transition of electrons from outer shells to inner shells. Each such electron transfer, for example from the L shell to K shell, represents a lost in the potential energy of the atom. The difference between the binding energies of the two shells is given off in the form of x-rays photon. One of two processes can then take place, namely (a) the x-rays photon escapes from the atom and contributes to a characteristic radiation of the atom; or (b) the photon is absorbed within the atom itself on its way out and ionizes the atom in an outer shell; for example a K_{α} photon can eject an L,M, or N electron; this phenomenon is called the Auger effect. In this case, no characteristic x ray is emitted. Again, the ionized atom becomes a source of radiation as explained above. The emitted radiation actually follow a number of rules that are better understood by referring to the atomic structure theory.

2.2 X-Ray Tube Source

Modern x-ray generators are still built around the Coolidge type tube (1913), i.e. a vacuum tube in which electrons are emitted by an incandescent W Filament (negative electrode or cathode), accelerated by a high voltage Vand focused on a

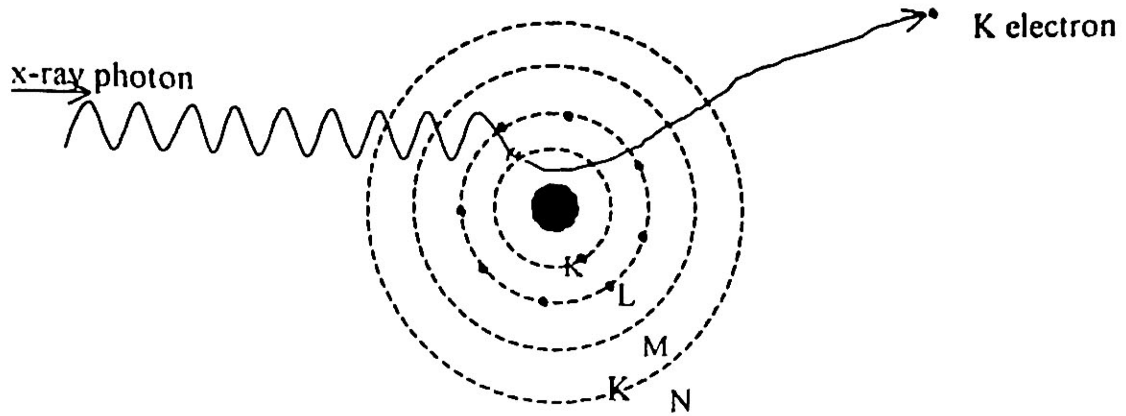

Fig 2.1 Ionization of the K Shell by an incident x-rays photon

water-cooled metal target (positive electrode or anode). A typical spectrum is shown in Fig.2.2; it consists of a continuous x-rays spectrum or white radiation on which are superposed a few characteristic lines of the target element, in the case W. The characteristic spectrum results from the direct ionization of the W atoms by the impinging electrons and is only observed if the voltage is higher than the critical voltage; in the case of the K lines, the characteristic spectrum is observed. If

$$V \geq V_k \qquad (2.1)$$

The critical excitation voltage of W is 69.5 kV for the K spectrum and 10-12 kV for the L spectrum. Consequently the emission spectrum of a W target tube operated at 50 kV consists of white radiation and L line of W Fig.2.3, but at 100 kV both the K and L spectra are observed. On account of their monochromatic character it is relatively easy to measure the intensity of the characteristic lines as a function of applied voltage. This relationship can be expressed by

$$I = A_i (V - V_k)^n \qquad (2.2)$$

where A is a constant, i is the current through the tube and the exponent n is a constant that has a value between 1.5 and 2 depending on the anode material and analyte element. The rapid increase in intensity predicted by eqn (2.2) does not materialize when V exceeds three or four times the critical voltage V_k or V_L.

The continuous spectrum is the result of loss of energy by the electrons as they collide at random with the loosely bound electrons of the target, hence the name of Bremsstrahlung also given to this radiation. The properties of the continuum that concern the analyst can be expressed by three criteria:

(1) The short wavelength limit is given by λ_o i.e the spectrum starts abruptly at a wavelength that does not depend on the target material but follows the Duane-Hunt relation.

$$h\mu_o = hc / \lambda_o = eV \qquad (2.3)$$

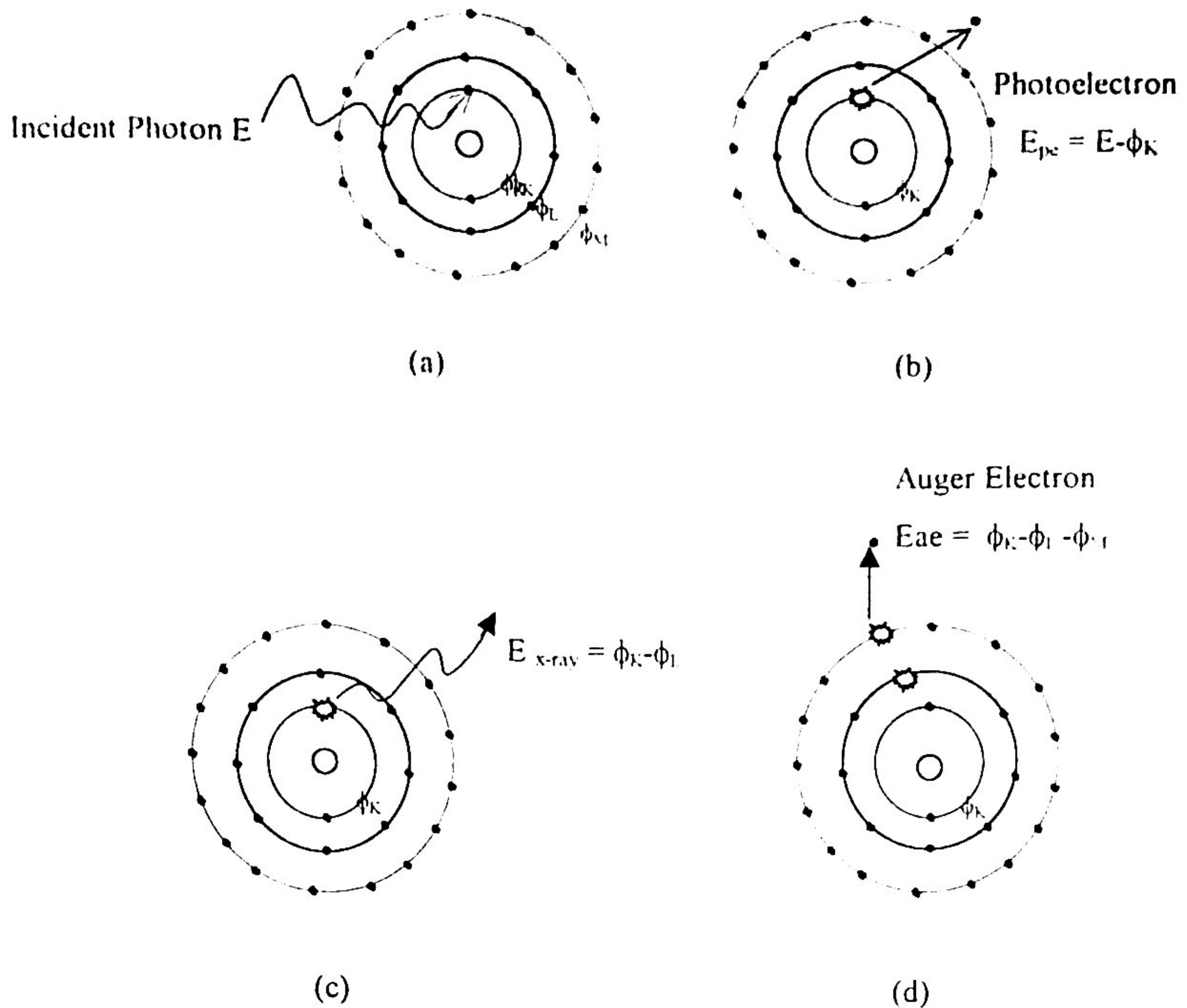

Fig 2.2 The photoelectric interaction

(a) Before photoelectric interaction a photon of energy E encounters the atom.

(b) In the photoelectric interaction the photon is absorbed by a K shell electron and the electron is ejected with energy equal to the photon energy less the K shell electron binding energy.

(c) The K shell vacancy is filled by an L shell electron and the different in binding energies is given off as either a characteristic x ray photon or

(d) as and Auger electron.

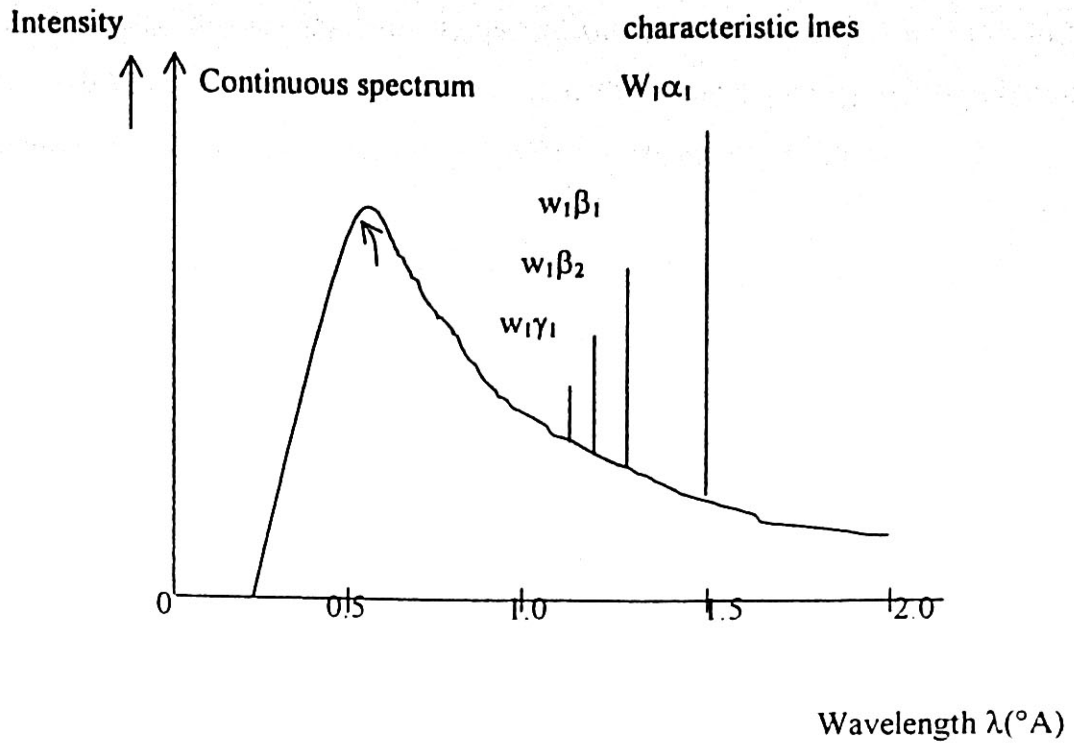

Fig 2.3 Typical spectrum from a W target x-rays tube at 50 kV

This expression signifies that the energy of any electromagnetic radiation in the emission spectrum can never be higher than the kinetic energy of the electrons striking the target. Following equation is more conveniently written.

$$\lambda_o = 12.398/V \qquad (2.4)$$

where λ_o is in Å and V in kV, the fact that the short wavelength limit varies as the reciprocal of the applied voltage is clearly shown in Fig 2.3.

(2) The total or integrated intensity, the area under the curve in Fig 2.4 an increasing function of applied voltage V and atomic number A of the target.

$$I = KZV^2 \qquad (2.5)$$

where K is a constant that includes the electron current I in the tube.

(3) The spectral distribution of the continuum can be approximated by empirical relation.

$$I_\lambda = CZ\frac{1}{\lambda^2}\left(\frac{1}{\lambda_0} - \frac{1}{\lambda}\right) \qquad (2.6)$$

where C is a constant that includes the current i. This is a remarkable equation because it describes the intensity distribution rather well, it predicts the observed maximum at $\lambda_m = 1.5\ \lambda_0$ and it is in agreement with eqn (2.5) for the integrated intensity. However, this analytical expression for the continuous spectrum is not sufficiently accurate for present requirement is petrochemical analysis and so led to newer developments along both theoretical and experimental lines.

2.3 Radioactive Source

Radioisotopes Table 2.1 are commonly used because of their stability and small size when continuous and monochromatic sources are required. Safely regulations require that x-rays emission from these sources is limited to about 10^7 photons s^{-1} streadian^{-1} compared with 10^{12} or 10^{13} for x-rays tube; the difference is only partly compensated for by the small size of source, which allows very compact source-specimen-detector assemblies to be constructed that are very convenient due to

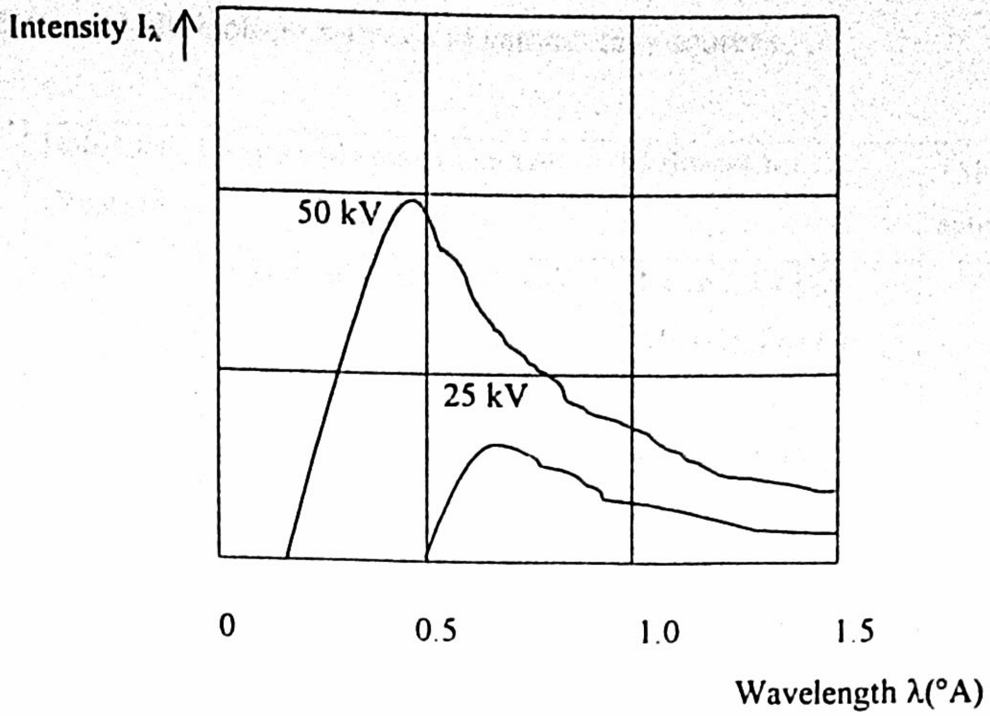

Fig 2.4 Effect of voltage on the continuous spectrum

Table 2.1 Radioisotope x-rays and gamma rays sources

Isotope	Half-Life (Years)	Type and energy emitted	Efficient for	Typical activity(Ci)
^{3}H	12.3	White radiation, 3-10 keV	Ca (K), Cr (K) Mn (K), Fe (K)	4.5
^{55}Fe	2.7	Mn K x-ray, 5.9 keV	Ca (K), V (K)	0.001
^{57}Co	0.74	γ-rays, mainly 122 keV Fe K x-rays, 6.4 keV	W (K)	0.001
^{109}Cd	1.3	Ag K x-rays, 22 keV	Fe (K), Cu (K) W (L), Mo (K)	0.001
^{153}Gd	0.65	γ-rays, 100 keV Eu K x-rays, 42 keV	W (K)	0.001
^{147}Pm/Al	2.6	White radiation, 10-100 keV	Mo(K), Ag (K) W (K)	0.5
^{241}Am	460	γ-rays, 60 keV N_pL x-rays, 11-22 keV	Mo(K), Ag (K)	0.001

their portability. On other hand, the low intensities preclude crystal dispersion so that these sources are used almost exclusively energy dispersion techniques.

2.4 The Secondary Fluorescence Method

The technique for improving trace-element detection limits is to concentrate all the excitation energy in a narrow band just above the absorption edge energy of the highest energy line to be excited. Radioactive sources such as ^{55} Fe, ^{109} Cd, ^{57} Co, etc have been used to achieve the desired monochromatic excitation but the source strengths are usually too low to achieve detectable limits competitive with optimize x-rays tube excited systems.

In the secondary florescence method, a high power x-rays tube is used to excite a secondary fluorescence target. The anode material is generally chosen to have a high bremsstrahlung output so that a wide range of secondary fluorescence materials can be efficiently excited. A fraction of the characteristic lines fluorescence in the secondary target, reach the specimen to excite the desired element lines. For light elements, secondary florescence excitation efficiency is limited by available x-rays tube current.

2.5 Instruments used in EDXRF System

An EDXRF system consists of several basic functional components. These components are an x-rays excitation source sample chamber, Si(Li) detector, preamplifier, main amplifier and the multichannel pulse height analyzer. The performance of an EDXRF system differs upon the electronics and the enhancements from the computer.

The different types of excitation sources can be used in an EDXRF system. The excitation source produces the x-rays beam that is used to generate x-ray fluorescence in the sample target. A Si(Li) detector normally consists of a protective Be window, a gold contact layer, a Si dead layer and the Si(Li) active crystal. When an x-rays photon from the sample is collected in the Si(Li) detector

diode. electron hole pairs are generated and are swept to the terminal the detector by the high voltage applied across it. The number of the electron hole pair is proportional to the energy of the photon. The pair is accumulated by a capacitor and the pulse is generated with voltage height proportional to the number of the pairs. The pulse is sent to a feedback preamplifier and then to main amplifier. The amplified pulse is then processed by a multichannel analyzer within which it is transformed into a digital signal by an analog to digital converter (ADC). The output of the ADC becomes the address of a channel in the memory unit where an add one operation is performed. The detection procedure mentioned above continues and a spectrum is then formed as counts (intensity) versus channel number (energy) and is displayed on a monitor. The spectrum suffers quite a lot of deviations due to the imperfections of the excitation, detection and signal processes. These imperfections cause peak broadening distortion fluorescence peak, silicon and gold absorption edges, pile up effects, incomplete charge collection dead time effects, non-linearity, stray radiations, diffraction from the sample target, etc. They need to be considered and improved by used of high performing electronics design measuring conditions.

2.6 XRD Analysis Technique

The diffraction of x rays phenomenon appears, when an electromagnetic waves passes through a slit whose size is of the same order as the wavelength or smaller. Since x rays have wavelength of the order of an angstrom, it is rather difficult to make a slit of this size to observe the diffraction of x rays. Von Laue first suggested the used of the planes of atoms in a crystal as the natural grating (set of slit) to observe the diffraction of x rays as shown in Fig 2.5, an incident x rays makes an angle θ with the crystal plane. If this crystal plane (that is, the incident angle is equal to the reflected angle, this assumption is unnecessary), it is easy to show that the path difference between ray 1 and ray 2, AC+CD is $2d\sin\theta$. When it is an integral multiple n of the wavelength,

we have

$$2dSin\theta=n\lambda \qquad (2.7)$$

$$n= 1,2,3,......$$

where,

d=lattice spacing

λ=the incident x ray wavelength

θ= the incident angle and reflected angle

The x rays reflected in the direction θ will have a maximum in their diffraction intensity. The above formula is called the Bragg Law. Where d is assumed that the distance between the two adjacent ions.

When a beam of x rays strikes a crystal, it must satisfy the Bragg Law for its reflected rays to be intensified by diffraction from the formula we know that the wavelength must be smaller than 2d, and if either x or d is known, the other can be calculated after measuring θ in a diffraction experiment.

A standard crystal with a known d can be used to measure an unknown x rays wavelength λ, and then this x rays can be used to measure the lattice spacing of an unknown crystal. For a cubic crystal, if d is measured from a diffraction experiment. Avogadro's constant N_A can be calculated. This is one of the experimental methods for measuring Avogadro's constant. Atoms in a crystal can form many sets of parallel planes in different directions shown in Fig 2.3 with varying values of d. Also as seen in figure, the densities of the numbers of atoms vary on the different planes, so the measured intensities of the reflected x rays also vary.

We consider the Lau film method. In 1912, Laue's suggestions, W.Friedrich and P Knipping used continuous-wavelength x rays diffraction experiment on a single

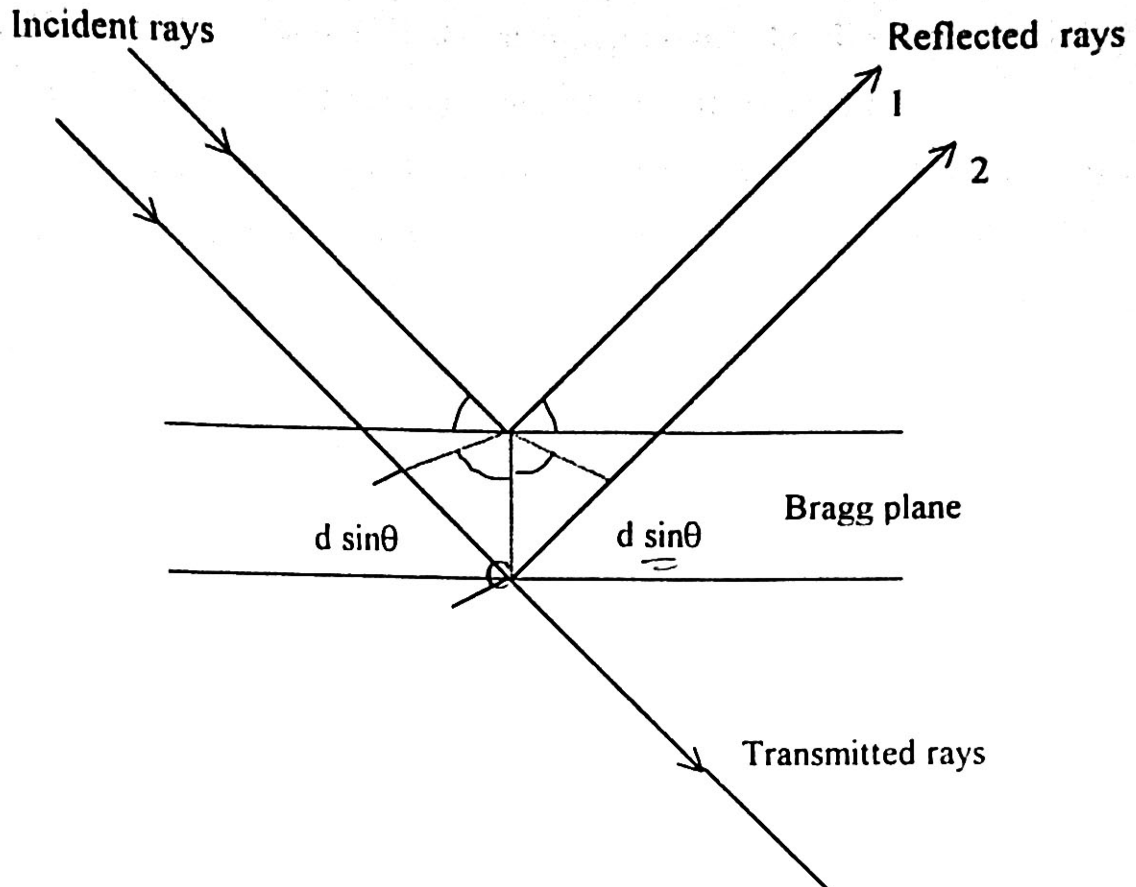

Fig 2.5 Derivation of Bragg's formula.

crystal. The experimental result on a sapphire single crystal is were occurred each dark spot in the photograph is called a Laue spot, which is correspond to one set of crystal planes. The position of the spot represents the direction of the related planes. Bragg's Law gave a correct explanation of these results.

It is not easy for a single-crystal to satisfy Bragg's law, but, fortunately, x rays are a continuous wavelength distribution were applied.W.H. Bragg and his son W.L Bragg not only gave the reflection relative for x rays but also invented the crystal reflection (diffraction) x rays spectrometer and applied it in a series of structural analysis of crystals.

The method more commonly used than Laue film method is the polycrystalline powder method invented by P.J.W. Debye and J.A Scherrer. The advantage of this method is that it does not need a sample crystal but rather only polycrystalline powder or metal foil. The preparation of sample is simplified greatly and generally monochromatic x rays are used.

CHAPTER 3

EXPERIMENTAL SET UP

3.1 Sample Preparation

The x-rays fluorescence analysis system and XRD method consists of Si(Li) detector and NaI scintillation detector with personal computer and x-ray analysis software and also used preamplifier, amplifier and ADC are used in experimental measurement. The powder of rock sample was prepared by pressing into pallets in two tons Hydraulic press. The pallet weight is 1g and the diameter of each sample is 2.54cm.

3.2 Experimental Procedure

The experimental work was done at Nuclear Physics Laboratory, Department of Physics, Yangon University, Department of Atomic Energy and Universities' Research Centre. In EDXRF measurement the samples are measured by the two methods: source excitation method and tube excitation method. Source excitation system used Cd-109 source. The spectrometer was operation voltage is 0.5kV. The detection unit is Si(Li) detector system. To minimize the electronic noise added to the signal during its use, the Si(Li) detectors, along with the first stage and feedback elements of the preamplifier, is mounted in a light tight, vacuum cryostat and operated at the liquid nitrogen at 77K. The block diagram of the experimental setup was shown in Fig 3.1.

The source excitation method can be detected the atomic number between 20 and 40. The atomic number of Sb has 51, and Pb has 82. So that Sb and Pb cannot detect. Pulse pile up rejection and baseline restoration circuits are included in the amplifier to eliminate resolution circuits are loss and pulse height (gain) shift with count rate. The collected x-ray spectra were stored in PCA card with 486 computer. Afterwards the concentrations of the elements were analyzed using

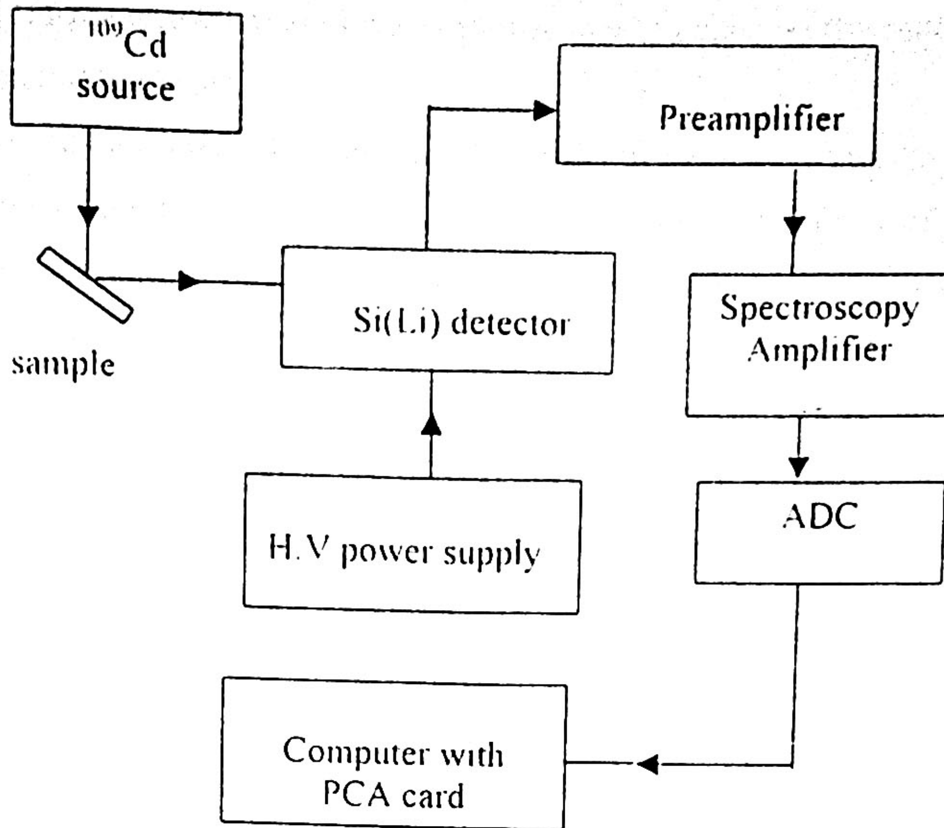

Fig 3.1 Block diagram of the x-rays fluorescence analysis technique

AXIL analysis software. The quantitative analysis is based on the fact that the intensity of the characteristic lines is proportional to mass on the concentration of the respective element.

In tube excitation method, a high-resolution semiconductor x rays detector EDX-700 software and EDX-700 spectrometer with an operation voltage of 50 kV were used in present work. The x-rays fluorescence analysis system consists of Si(Li) detector. The crystal is equipped with a thin beryllium (Be) window to minimize absorption of low energy x-ray. Rhodium (Rh) target was used in detection system. The EDX-700 can automatically identify all elements in a sample based upon a library of x rays data. In addition to this standard feature, quantitative information is provided.

Pulse pile up rejection and baseline restoration circuits are included in the preamplifier to eliminate resolution loss and pulse height (gain) shift with concentrate. The collected x rays spectra were stored in measurement software with computer. Afterwards the concentrations of the elements were analyzed using analyzed software. The quantitative analysis is added on the fact that the intensity of the characteristic lines is proportional to mass on the concentration of the respective element.

The x-rays diffractometer used throughout this investigation is a computer-controlled Rigaku, the model number is D/max-2200/-PC and Cat number G 402. The operation conditions of the x-rays diffractometer using copper target. The operation tube voltage 40kV and 30mA tube current. The detection unit is NaI scintillation detector. The block diagram of the experimental set up was shown in Fig 3.2.

3.3 Source Excitation Method

The excitation source produces the x-rays beam that is used to generate x-rays fluorescence in the sample target. When an x-rays photon from the sample is collected in the Si(Li) detector diode. The x-rays spectra displayed on the monitor

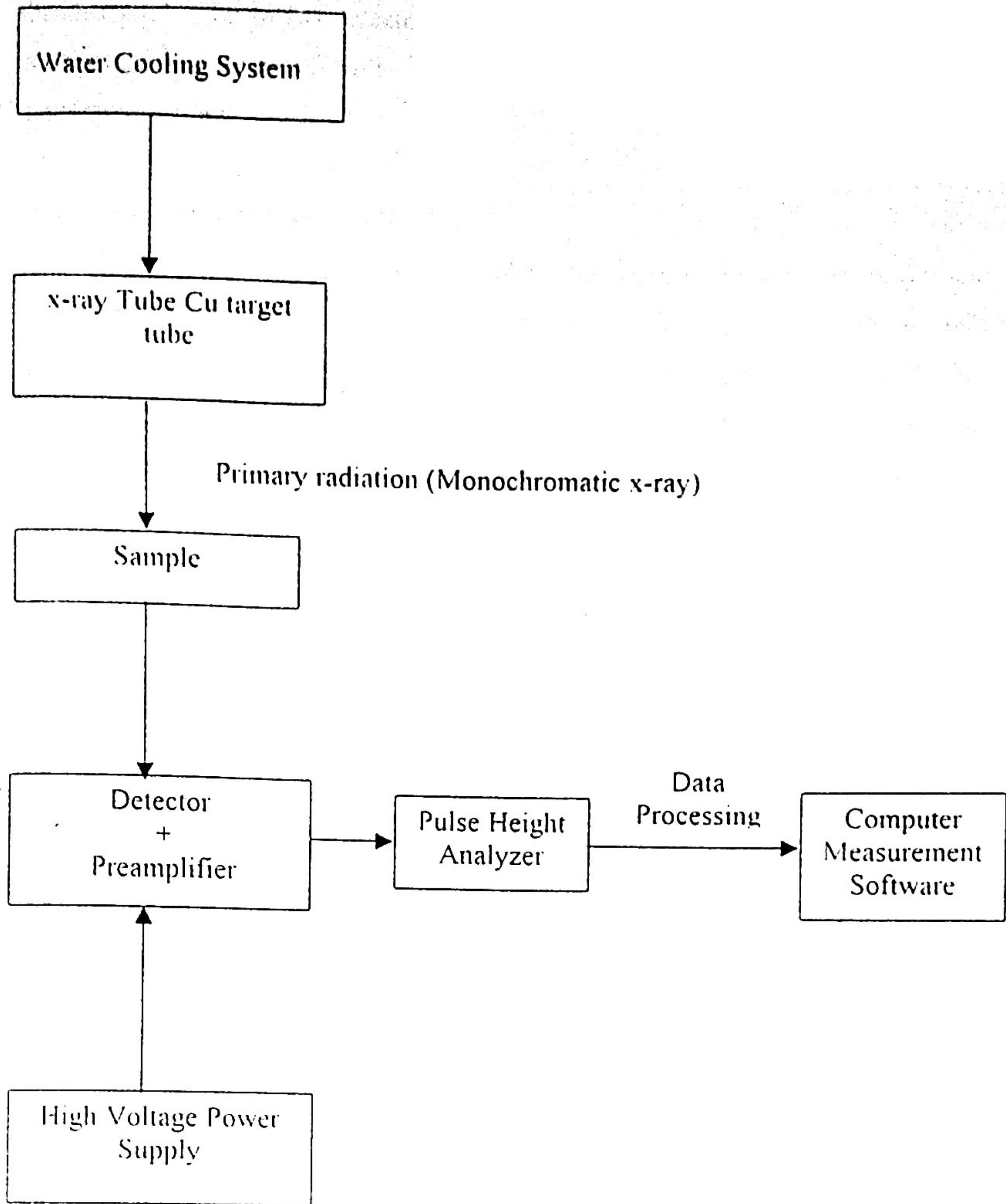

Fig 3.2 Block Diagram of x-rays diffractometer system

of computer with x-ray Analysis Software (AXIL Programme and QAES Programme). The Si(Li) detector system with Cd-109 source, with an operating voltage of negative 500 volts was used in the present work. The sample was irradiated for 500s.

3.4 Tube Excitation Method

The sample was mounted on sample holder. The x-rays beam from the x-rays tube of rhodium (Rh) target was set to fall on the sample for exciting. The individual's elements comprising the sample re-emit their own "characteristic " x-rays. A high resolution semiconductor detector, EDX-700 software and EDX-700 spectrometer with an operating voltage of 50 kV were used. The EDX-700 (Energy Depressive XRF system) detects these x-rays and quantitative determines which elements are present in the material. The characteristic x-rays of each elements contained in each sample was detected by Silicon Lithium Si(Li) detector. The x-ray spectra displayed on the monitor of computer were analyzed by using FP (Fundamental Parameter) software. The detection unit is Si(Li) detector with a resolution of 170 eV at 5.9 keV x-ray spectra were acquired on a PC based MCA, Canberra S 100. Analyzed energy is 40 keV. The sample was thin irradiated for 100s. The detector can be stored at room temperature but the working temperature of the detector must be kept at liquid nitrogen (-170°C). The x-rays tube was operated at its optimized condition of 50kV and 15mA (Auto). The x-rays spectrum was then recorded.

CHAPTER 4

RESULTS AND CONCLUSION

4.1 Results and Conclusion

The concentrations of the elements contained in the sample were calculated. The observed concentration data were shown in Table 4.1. The concentration of the elements content in the rock sample Gneiss of source excitation method was shown in Fig 4.1. The Fig 4.2 was shown the elements content in the rock sample of Gneiss of tube excitation method. The comparison of the concentration Fe content to rock sample of two methods was shown in Fig 4.3. The concentration of the elements (ppm) content in the rock sample Gneiss of tube excitation method was shown in Fig 4.4.

It is calculated that the XRF method can be successfully applied to the determination of some elements in different types of sample. The mechanical stability of XRF Spectrometer and optimization of the excitation source led to the versatile application. The AXIL x-rays analysis software available permits the quantitative determination of the major, minor and trace elements within a short time. The whole procedure is directly applicable to any analytical case when multielement characters have to be determined.

The raw materials employed in the formulation of Gneiss rock sample Quartz SiO_2 was studied by thermal gravimetric-differential thermal analysis confirm their identity. In order to do so it was firstly confirmed by the x-ray powder diffraction method for its exact structure. In Fig.4.5 was shown the XRD spectrum of the rock sample Gneiss.

The diffraction patterns of the samples were recorded using copper radiation as source. The scan is essentially a plot of intensity as a function of $°2\theta$. It is a chart

with a line that changes its position every 0.5°2θ. To interpret the x-ray diffraction, pattern or spectrum, peaks with an indexing system based on the use of there 'd' spacing giving the strongest intensities were chosen.

In conclusion, confirmation of raw materials used in the formulation Gneiss rock sample was carried out by quantitative x-rays diffraction method. The major compositions of SiO_2 in rock sample was determined to be purity implying that the compounds are the major constituents of the respective raw materials. We selected elements cannot be detected significantly because of these are natural ore samples. Unless otherwise, it can be detected by XRF method after metallurgical processing.

This result is not real quantitative results and some minerals are constituted as of oxide form and some are as of sulphide form. The definite result has to obtained for quantitative analysis after standardized calibration. According to the measurement of Gneiss rock by using EDXRF method, the amount of SiO_2 contained in these rock is very high. According to the measurement of Gneiss rock by using XRD method, the amount of SiO_2 also contained in these rocks is very high.

Table 4.1 The Concentration of Elements in Rock Samples by EDXRF Analysis

Sr No	Sample Name	Element Concentration (%)			
		Method (1) (Source Excitation Method)		Method (2) (Tube Excitation Method)	
	Gneiss	Fe	54.0%	Si	10.882%
		Cu	0.3%	Fe	3.506%
		Zn	0.1%	S	3.120%
				Ca	1.668%
				K	1.429%
				Ti	1988.481 ppm
				Sr	1477.860 ppm
				Sb	856.480 ppm
				Sc	586.160 ppm
				Mn	449.934 ppm
				Pd	444.409 ppm
				As	345.980 ppm
				Pb	336.083 ppm
				Cu	317.970 ppm
				Zr	186.953 ppm

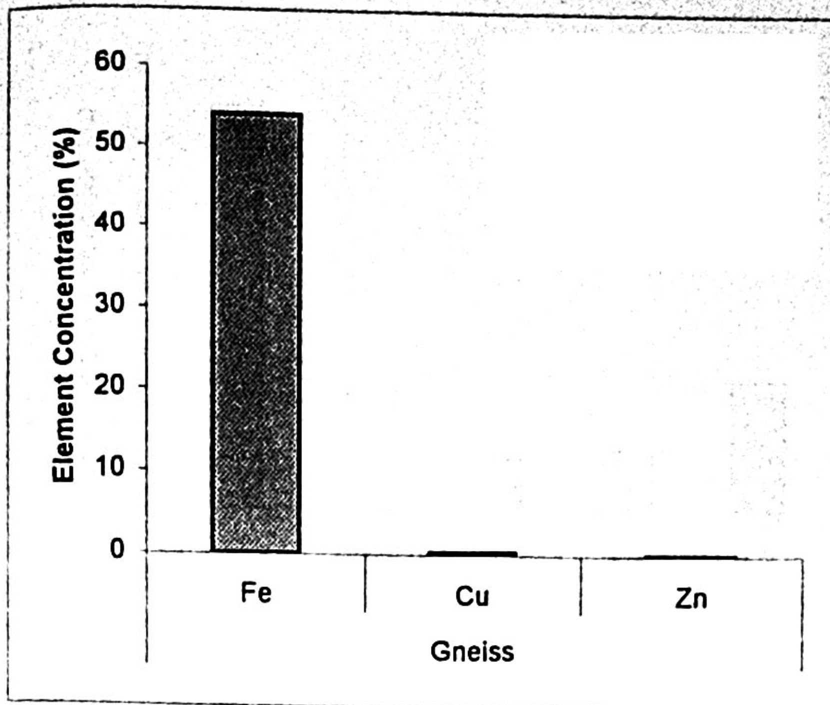

Fig 4.1 The concentration of the elements content in the rock sample Gneiss of source excitation method

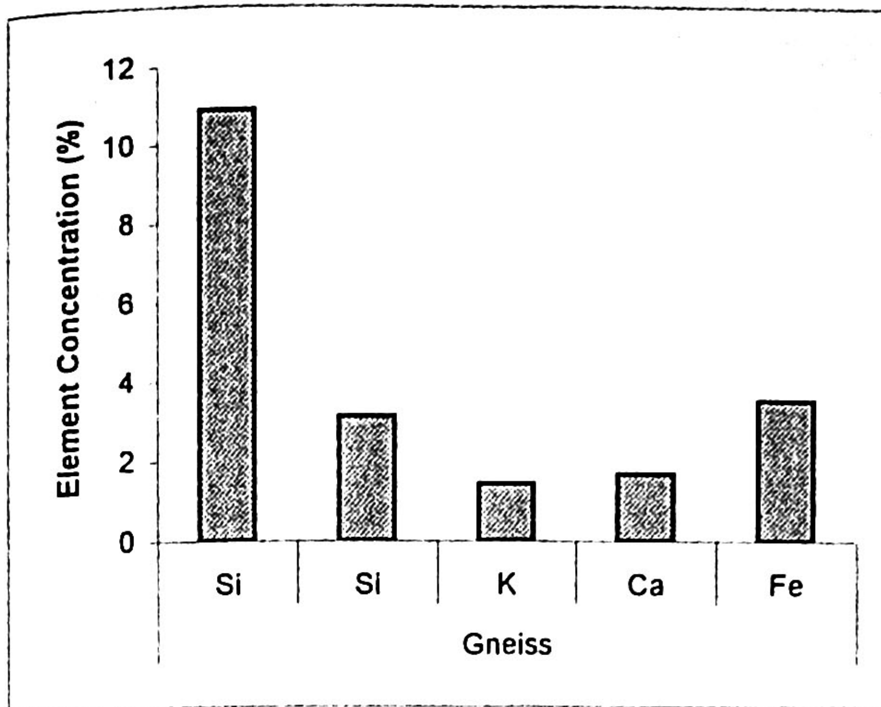

Fig 4.2 The concentration of the elements content in the rock sample Gneiss of tube excitation method

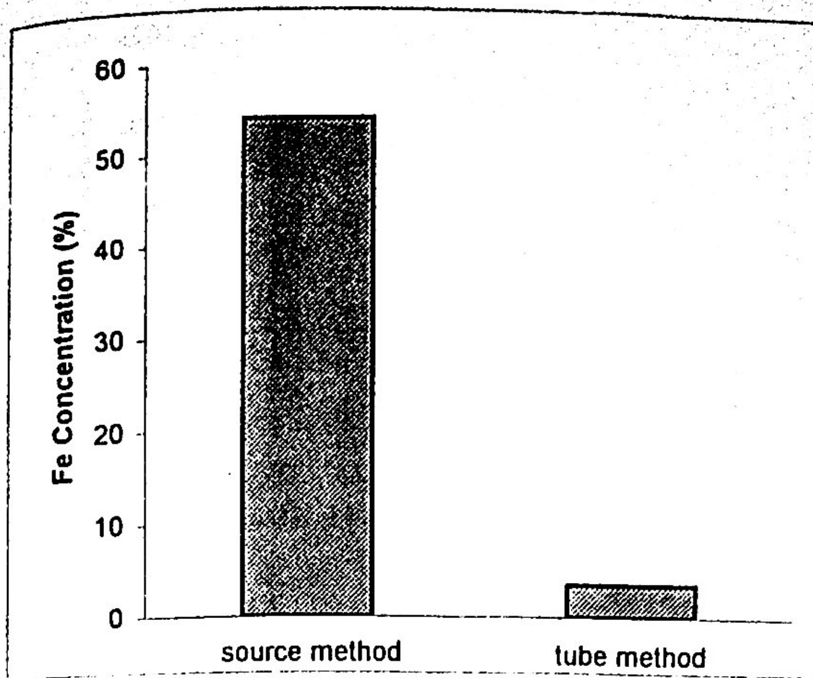

Fig 4.3 The concentration of the element Fe content in the rock sample Gneiss

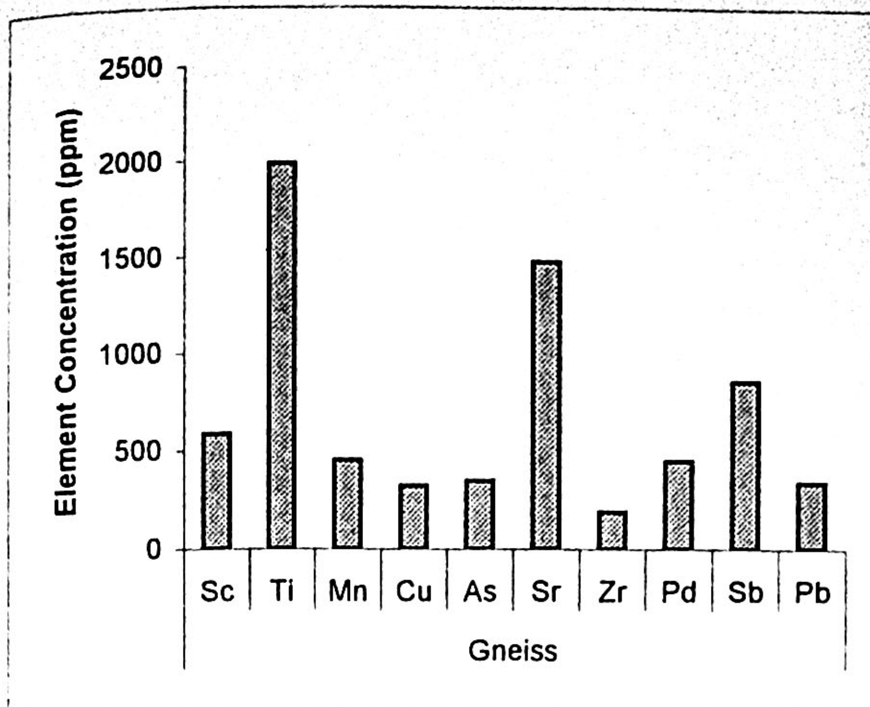

Fig 4.4 The concentration of the elements content in the rock sample Gneiss of tube excitation method

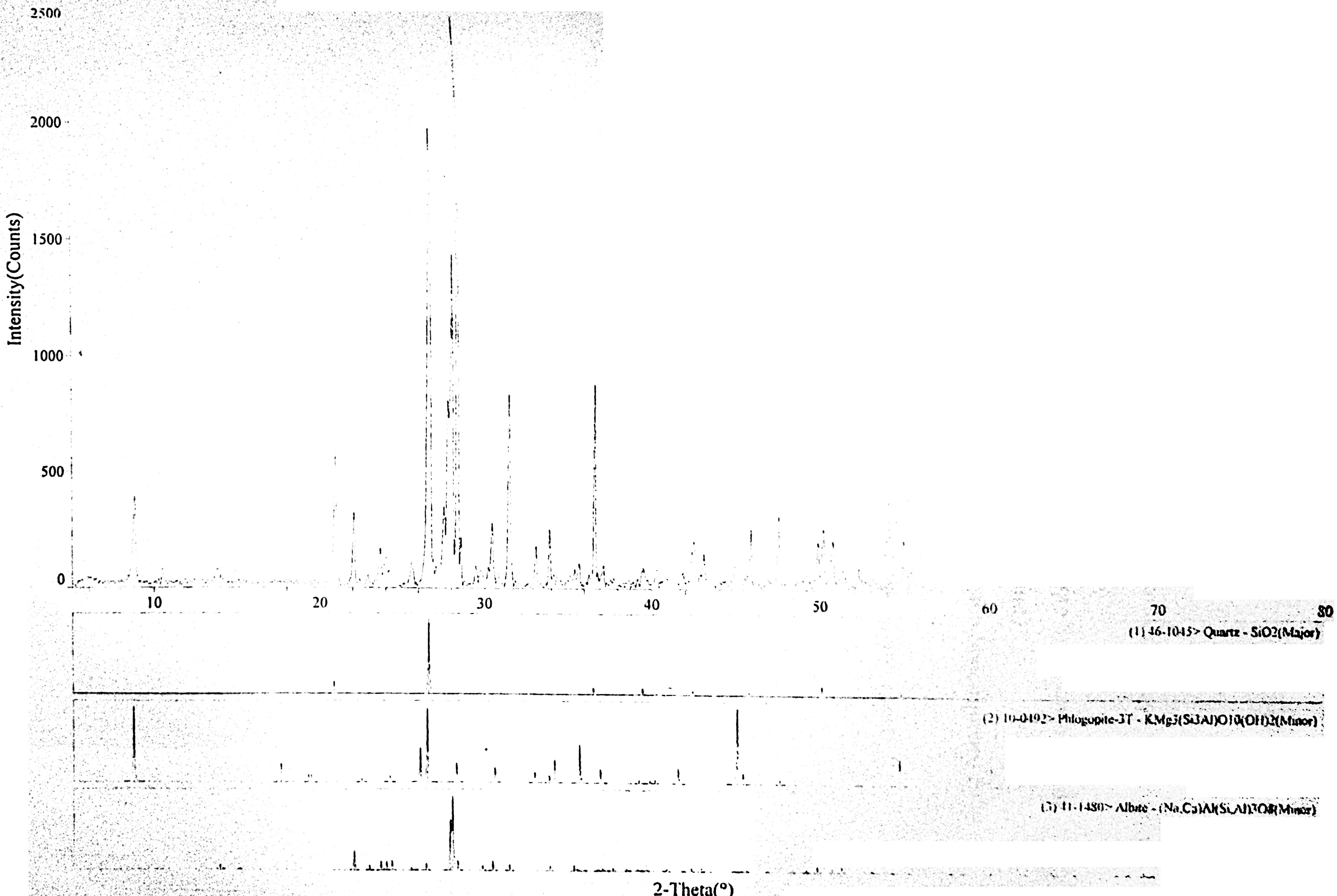

Fig 4.5 The XRD Spectrum of the Rock Sample Gneiss

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