CHAPTER 2
Experimental Techniques

2.1 Preparation of Ferrites

The ferrite samples are prepared by the standard ceramic method [Fig. 2.1]. The materials are usually oxides or carbonates of the constituent metals. They are weighed accurately in the proper stoichiometric ratios and mixed thoroughly. The mixing improves thermal conductivity.

Pre-sintering or pre-firing in a calcining process is the one in which the temperature of the powder is raised to the region of \(800^\circ\text{C}\). Though this is not essential in the preparation of ferrites, it is a very important means of obtaining the necessary degree of control over the properties of the finished product. According to Wallow and Jordan [1], pre-sintering is carried out for the following reasons:–

- Decomposes the carbonates or higher oxides, thereby reducing the evolution of gases in the final sintering.
- Assists in homogenizing the material.
- Reduces the effects of variations in the raw materials.
- Reduces or controls the shrinkage occurring during the final sintering.

After pre-sintering, the porous lumps of partly formed ferrites are easily crushed and once again made into fine powder. At this stage, the binder and lubricants are added. Commonly used binders are acrylates and polyvinyl alcohol; while waxes and wax emulsions may be used as lubricants to ease powder flow. The quantities used are very small. The correct quantity of granulated powder is then poured into the die and a pressure of about 1.6 to 16 kg/mm\(^2\) is applied to convert the powder to the required pellet or toroid.

These pellets or toroids are then placed in alumina crucibles inside the furnace and sintered at temperatures of \(1000^\circ\text{C}\) or more for 6 to 10 hours. At these high temperatures, the free surface containing the voids decreases; the particles grow together to form crystallites and their density rises.
Fig. 2.1 Typical flow diagram for preparing polycrystalline ferrites by dry ceramic technique
The graph Fig. 2.2 shows a typical sintering cycle for a ferrite. During the heating period, the oxygen pressure is made relatively high. As maximum temperature is reached, oxygen pressure is normally reduced quickly to a low value. For those ferrites whose properties are not critical, this reduction of oxygen is not necessary and the whole sintering process can be carried out in air.

![Graph showing sintering cycle](image)

**Fig 2.2 Typical sintering cycle for a ferrite**

The sintered pellets or toroids are then subjected to the most usual finishing process – the grinding of the surfaces. The mechanical dimensions are checked by conventional techniques.

Some of the other basic methods are:
- Spray Drying of Mixed Oxide or Salts – This is used where high purity and control of composition is required [2].
- Flame Spraying – This is used for microwave ferrites with fine grain size [3].
- Chemical Vapour Deposition – In this, a vapour comprising a mixture of the component oxides produced insitu by chemical reaction is passed over a heated single
crystal substrate where the ferrite is deposited directly from the vapour phase to give epitaxial growth [4].

Chemical Co-Precipitation – In this, the constituents may be precipitated from salt solutions in the form of hydrated oxides or oxalates. However, the drawback in this technique is that the product is susceptible to changes in the conditions of the solution during the preparation of a batch. Also, unwanted products are formed and these are difficult to remove [5].

Sol-Gel Process – Sol-gel techniques can be applied to an extremely wide variety of materials, where they offer the possibility of controlling the size, distribution of particles and their shape. Fibres, films and monoliths can also be obtained by sol-gel methods.

The process involves the preparation of a ‘sol’, which is a dispersion of solid particles, the ‘dispersed phase’, in a liquid, the dispersion medium; at least one dimension of the particle lies between 1 nm and 1 μm. The sol is prepared by mixing concentrated solutions containing the cations of interest, with an organic solvent as the dispersion medium [6]. It is also possible to begin with a colloidal solution, instead of, or together with the solutions. The sol is then destabilized, generally by adding water. The presence of water modifies the pH of the sol and reduces the repulsion between particles. The viscosity of the system increases, leading to the formation of a ‘gel’.

If excess water is used, the gel is a continuous network of aggregates formed by the particles; it is a ‘colloidal gel’ or an ‘aquagel’. If the water is added slowly and in small amounts, the sol particles increase in size by a condensation-polymerisation reaction. This gives a ‘polymeric gel’, also known as ‘alcogel’. For the removal of the dispersion medium, the rate at which the operation is performed is important. Fast removal leads to a powder. For this, fine droplets of the gel is passed through a column of an alcohol. The final ceramic can be obtained by compaction and sintering of the ‘powder xerogel’ at an appropriate temperature. A very slow and controlled removal of the liquid can lead to fabrication of fibres, coatings and monoliths.

The process can be operated on a continuous basis though its present disadvantage is the high cost of the materials used.
By Nanotechnology - Synthesis of nano size (9 – 12 nm) Mn$_{0.65}$Zn$_{0.35}$Fe$_{2.04}$ particles from metal chloride solution through a hydrothermal precipitation route using aqueous ammonia is described here [7].

Stock solution of ferric chloride (120 g/l) is prepared from electrolytic iron powder in the presence of nitric acid as an oxidant. Zinc and manganese chlorides (100 g/l) are prepared from electrolytic zinc granules and manganese chlorides, respectively. Aqueous ammonia, 25% is used as the precipitant. Required quantities of Fe$^{3+}$, Mn$^{2+}$ and Zn$^{2+}$ chloride solutions are mixed and the pH is adjusted to required values varying between 7.6 and 9.

In another set of experiments, ammonia is added to Fe(III) chloride solution to adjust the pH for precipitation of Fe(III) hydroxide, and the chloride from the slurry is removed by washing with distilled water. The iron hydroxide slurry so obtained is transferred quantitatively to the predetermined quantities of manganese and zinc chloride solutions. With continuous stirring, ammonia is added to the suspension to obtain the desired pH between 8.5 and 11.

The slurries so obtained are subjected to hydrothermal treatment in an autoclave having an automatic temperature controller and provisions for sampling, gas inlet, outlet, stirring and cooling. Sampling is done at different time intervals during the course of the reaction and immediately filtered in order to estimate the soluble metal ion present in the reaction mixture. The autoclave is cooled to 75$^\circ$C and the contents are filtered immediately. The precipitated ferrites are made chloride-free by thorough washing with distilled water, treated with acetone and dried in air at 80$^\circ$C for 24 hours. The dried samples can then be analysed for their composition, microstructure and magnetic properties.

2.2 X-ray diffraction

X-ray diffraction (XRD) from a crystal reveals the arrangement of its constituent atoms and thus gives information about the structure of crystals. X-ray diffraction (XRD) is a complementary analytical technique to electron diffraction. One limitation of electron diffraction is small sampling volume. If several electron diffraction patterns are not collected across the entire specimen, it is difficult to make a claim about the crystallinity of the bulk of the sample. X-ray diffraction, however, analyzes milligrams of sample (billions of particles in the 1 to 100 nm range) while electron
diffraction may analyze micrograms (thousands of particles in the 1 to 100 nm range). Using XRD in combination with electron diffraction allows one to corroborate data acquired with both techniques.

In the present study, a Rigaku Miniflex model X-ray diffractometer (60 KV x 300 mA) was used. In this, characteristic Cu-Kα X-rays with wavelength 1.54056 Å (Ni filter) are allowed to fall on the sample and the intensity of the X-rays reflected is recorded as a function of the angle (2θ) by which the incident beam is deviated on reflection. Data were collected over a range of 2θ of 20° - 70° with a scan speed of 2°/min. Strong reflections occur where the Bragg condition (eq. 2.1) is satisfied

\[ 2d \sin \theta = n\lambda \]  

where \( d \) is the interplanar spacing and \( n \) is an integer. Depending on the crystal structure and crystal parameters, intense peaks are developed at various values of \( \theta \) corresponding to different families of atomic planes. The block diagram of the set up used is shown in Fig. 2.3 and a typical XRD pattern of spinel structure is shown in Fig.2.4.

![Fig. 2.3 Schematic diagram of XRD set up](image)
Fig. 2.4 Typical X-ray diffraction pattern of spinel structure

X-ray diffraction can also be used to determine the crystallite size from analysis of peak broadening. This may be understood by assuming the crystal to be a three-dimensional grating. It is well known that width of principal maxima is inversely proportional to the number of slits present on a grating. Thus, as the particle size is decreased, the number of diffraction centres will decrease and the peak under observation will get broadened. Scherrer [8] derived a relationship between the broadening of diffraction peaks and the average crystallite size, which states that the average diameter $t$ of the crystallites composing a powder is related to the X-ray diffraction line broadening $\beta$ as

$$ t = \frac{0.9\lambda}{\beta \cos \theta} $$  \hspace{1cm} [2.2]

where $t$ is the crystallite size, $\lambda$ is the X-ray wavelength, and $\beta$ is the FWHM of the XRD peak at $2\theta$. $\beta$ is the contribution to the XRD peak width due to the small size of crystallites. The broadening parameter $\beta$ is calculated using the relation

$$ \beta^2 = B^2 - b^2 $$  \hspace{1cm} [2.3]

where $B$ is measured linewidth and $b$ is instrumental broadening. This instrumental broadening factor was determined using an X-ray pattern of standard silicon sample under identical conditions.
2.3 Scanning Electron Microscope

Scanning Electron Microscope (JEOL JSM 5600) is used to examine the surface morphology of the solid specimens and it is one of the powerful techniques employed to get the images of the specimen. The properties of ceramic materials are closely associated with its microstructure and hence the analysis of its microstructure can yield useful information like surface defects, cracks, pores, presence of additional phases, grain morphology and sizes etc.

In Scanning Electron Microscope the electrons emitted by a heated filament is accelerated and then collimated into a narrow beam. It is then allowed to fall upon a small portion of the specimen surface, which then releases secondary electrons, backscattered electrons, characteristic X-rays and several other types of radiation from the irradiated portion of the sample. The intensity of these signals will depend on the shape, chemical composition and crystal orientation of the irradiated volume. The secondary electron current reaching the detector is recorded and the microscope image consists of plots of this current against probe position on the surface. The contrast in the micrograph arises from several mechanisms. Since the image contrast is varied according to the shape or composition of the specimen, the scanning electron microscope provides useful information about the specimen.

SEM facilitates the observation of very fine details (high resolution) of materials and good focus over a wide range of specimen surface (large depth of field). It also produces clear image of specimen ranging from object visible to the naked eye to a structure spanning few nanometers. Besides its use in studying soils, sedimentary particles, rock materials, synthesized compounds, and biological materials, it also helps to elucidate the architecture and evolution of microfossils.

2.4 Conductivity Measurements

The four-probe method is one of the standard and most widely used method for measuring conductivity.
Fig 2.5 Four probe

The probes are collinear and equally spaced. The probes are mounted in a teflon bush, which ensures good electrical insulation between the probes. A teflon spacer near the tips is also provided to keep the probes at equal distance. The probe arrangement is mounted in a suitable stand, which also holds the sample plate and temperature sensor. This stand also serves as the lid of the PID controlled oven. Proper leads are provided for the current and voltage measurements.

The PID controlled oven is a high quality temperature controlled oven suitable for four-probe set up. The oven has been designed for fast heating and cooling rates, which enhances the effectiveness of the controller. Platinum RTD has been used for sensing the temperature. A Wheatstone’s Bridge and an instrumentation amplifier are used for signal conditioning. The set and measured temperature are displayed on a $3\frac{1}{2}$ digit display panel meter through a selectors switch.

A battery operated, low current source is used to measure the current. The battery operated source ensures that the noise pick up from the mains and elsewhere is kept a minimum even though the sample resistance is high. An internal voltage reference of 2.5 volt ensures reliable operation even when the battery voltage falls and a ten turn potentiometer makes the current adjustment very easy. The actual current is read on a $3\frac{1}{2}$ digit LCD display.
The digital microvoltmeter used for the measurement of low d.c. voltage is a very versatile multipurpose instrument. It uses a very well designed chopper stabilized IC amplifier. This amplifier offers exceptionally low offset voltage and input bias parameters along with excellent speed characteristics. A filter circuit is provided to reduce the line pickups of 50 Hz. All internal power supplies are IC regulated.

The given sample is placed on the base plate of the four-probe arrangement. The pipe holding the four probes is unscrewed so that the four-probes rest in the middle of the sample. The pipe is tightened at the position in which a very gentle pressure is applied in the probes.

\[ \rho = \rho_0/G(W/S) \]  

Fig 2.6 Correction divisor for four probe method

The outer pair of probes is connected to the constant current source through a three-pin socket and the inner pair to the d.c. microvoltmeter. The microvoltmeter is switched on and about five minutes time is given to attain thermal stability. The current source is then switched on. The power to the oven is also switched on. The voltage (V) and the current developed (I) at different temperatures are noted. All the probes are equally spaced.

Resistivity is calculated using the relation

\[ \rho = \rho_0/G(W/S) \]  

[2.4]
where
\[ \rho_0 = \frac{V}{I} 2\pi S \] \hfill [2.5]

and \( G_\gamma(W/S) \) is a function obtained from the graph for appropriate values of \( W/S \). Where \( S \) is the distance between the probes and \( W \) is the thickness of the sample.

### 2.5 Thermoelectric Measurements

When two dissimilar metals are joined at the two ends only and if these two junctions are maintained at two different temperatures, a thermoemf develops across the two junctions. This is Seebeck effect. Experiments have shown that the Seebeck voltage \( (V_S) \) is proportional to the temperature difference \( (T_2 - T_1) \) where \( T_1 \) and \( T_2 \) are the temperatures of the cold junction and hot junction.

![Fig 2.7 Experimental arrangement to determine Seebeck coefficient](image)

\[ V_S = S(T_2 - T_1) \] \hfill [2.6]

where the constant of proportionality \( S \) is the thermoelectric power or Seebeck Coefficient and has the dimensions of voltage per degree.

#### 2.5.1 Experiment

The thermoelectric measurements of the ferrite samples has been measured by the hot probe method [9,10]. The experimental arrangement is shown in fig. 2.7. A point contact probe touching the upper surface of the ferrite sample acts as the hot junction (A). The ferrite sample (C) is placed on a metal base (D) which acts as the cold junction.
The bottom surface of the sample is coated with silver paint to provide good electrical contact with the base. The temperature of the probe can be raised to about 200°C with the help of an electric heater coil wound round it.

The pointed hot probe ensures that the upper and lower surface of the sample have a difference in temperature between them. If the pointed probe is not used, both the surfaces would attain almost the same temperature, and there would practically be no temperature difference between them. The hot probe and base are connected to a microvoltmeter for measuring the thermoemf. The ferrite specimens used in the experiment are in the shape of discs of approximately 13mm diameter and thickness 0.2cm.

2.6 Dielectric Measurements

The dielectric measurements were made from 40 to 110 MHz by an Agilent 4294A Precision Impedance Analyzer. From the capacitance and phase angle between the current and the voltage across the sample for different frequencies the dielectric constant and loss factor are calculated by using the equations

\[
\varepsilon = \frac{C \cdot d}{\varepsilon_0 \cdot A}
\]

[2.7]

and

\[
\tan\delta = \frac{1}{\tan\Phi}
\]

[2.8]

where C is the capacitance due to the presence of the sample between the two electrodes of the sample holder, d is the thickness of the sample, a is its cross sectional area, \(\varepsilon_0\) is the free space permittivity and \(\Phi\) is the phase angle between current and voltage across the sample.

2.7 Vibrating Sample Magnetometer

The VSM, first developed by Foner [1956, 1996] in the late 1950s, is a basic research tool for determining magnetic properties in a variety of studies of the structure of paramagnetic, ferromagnetic, antiferromagnetic, diamagnetic and ferrimagnetic materials. VSM has been used in such areas as the study of amorphous and magnetic
domain materials and investigations of the nature of the metastable magnetic materials like superparamagnetic or spin glass materials.

The force method and induction method are used commonly to measure dc magnetization. In force method, one measures the force experienced by a magnetic dipole moment in a known uniform field gradient while the induction method involves the measurement of change in flux due to the relative motion between the sample and the detection system as in Vibrating Sample Magnetometer (VSM) or SQUID Magnetometers. The detection systems in the induction methods include coils, Hall probe etc. The induction methods are performed in a uniform magnetic field hence they are free from field gradients that are a natural outcome of the force methods. In general, all induction methods involve a measurement of induced voltage produced in a detection coil by a flux change when the applied magnetic field, coil position or sample position is changed. In moving the detection coil as done in vibrating coil magnetometer, extremely uniform magnetic fields are required over the whole coil, therefore moving the sample with respect to stationary coils is a better alternative.

If any material is placed in a uniform magnetic field, a dipole moment will be induced in the sample proportional to the product of the sample susceptibility and the applied field. If the sample is made to undergo sinusoidal motion, the resulting magnetic flux changes near the sample will induce an electrical signal in suitable placed stationary coils. This signal will be proportional to the moment, amplitude, and frequency of vibration.

Fig. 2.8 shows a typical Vibrating Sample Magnetometer (VSM) setup. The material under study in the VSM is inserted in the sample holder so that it rests centered in a pair of pickup coils between the poles of an electro magnet. The sample holder is mounted using a sample rod in a transducer assembly that passes through the center of a driving coil. The transducer is driven by a power amplifier which itself is driven by an oscillator. The magnetic sample under study is constrained to vibrate only along the vertical axis. As it does so, it induces a signal in the sample pickup coils as described above. The magnitude of this signal is dependent on the magnetic properties of the sample itself.
Attached to the sample holder rod is a permanent magnet. A pair of stationary coils picks up the induced ac signal, which is proportional to the amplitude and frequency of the vibration and is used as a control signal for modulation of the transducer. The output of the sample coil is fed to the differential input of a lock-in amplifier. The reference input of the lock-in comes from the sine wave oscillator used to drive the sample holder. The output of the lock-in goes to the data acquisition computer as well as the magnitude of the applied magnetic field coming from a gaussmeter. The signal from the lock-in amplifier is directly proportional to the magnetic moment of the sample. The computer is now able to graph the magnetic moment of the sample against the applied magnetic field. The set up used for present case was calibrated using standard paramagnetic salts Gd$_2$O$_3$ and Er$_2$O$_3$. Sensitivity was found to be better than $5 \times 10^{-6}$ emu in the measured field range. The system used for the present study is Lakeshore Model 7404.

The magnetic moment measurements of the samples are made with this machine. This is a nondestructive technique. The magnetic susceptibility value is helpful in the determination of the oxidation state of paramagnetic ions.

![Vibrating Sample Magnetometer system Overview](image)
2.8 LCR Meter

The inductance measurements of the ferrite samples has been carried out by an LCR meter working at a frequency of 1 KHz. The inductance of a 28 gauge coil wound round the toroidal samples were measured at 1 KHz frequency, for different temperatures from room temperature to a temperature well beyond the curie temperature.

2.9 Curie Temperature Measuring Device

The device consists of an iron rod attached to the bottom of a powerful magnet is placed inside a transparent furnace whose temperature can be varied by controlling the current with the help of a rheostat. The entire arrangement is then placed inside a glass chamber. The sample in the form of pellet is placed at the bottom of the iron rod. The pellet being ferrimagnetic at room temperature is well attracted by the magnetic rod. On slowly increasing the temperature, the sample suffers a change of phase from ferrimagnetic to paramagnetic, the pellet falls down at a particular temperature. This temperature is recorded as curie temperature.

Fig 2.9 Experimental arrangement to determine Curie temperature
2.10 Mössbauer Spectrometer

The Mossbauer experiment can be done in both transmission and back scattering geometry. While the transmitted photons through the absorber are counted in the former case, in the later the conversion electrons from the surface of the sample are counted (known as conversion electron Mossbauer spectroscopy, CEMS). Due to the finite range of the emitted electrons, the information obtained in the CEMS is limited to the surface region. Hence, for this reason, for the study of thin films CEMS is used. For both the cases, the components of the set-up are same except the detector. The Mossbauer spectrometer essentially consists of source, absorber, and velocity drive system for moving the source relative to an absorber, detector with counting and sorting arrangement. In most cases the source is moved with desired velocity range by an electromechanical drive.

![Block diagram of a typical Mossbauer spectrometer](image)

Figure 2.10: Block diagram of a typical Mossbauer spectrometer. M.C.A. and S.C.A. stand for multichannel analyser and single channel analyser, respectively
2.10.1 Source

The decay of a long lived radioactive element like $^{57}$Co embedded, generally in a metallic matrix populates the 14.4 keV excited state and transition to the ground state with a recoil free emission becomes the ideal source for Mossbauer experiments. This process combined with the reverse process in an absorber is the resonance phenomenon indicated by the bold arrows connecting the ground and excited states. The radioactive source nuclei are usually embedded in a matrix which provides the necessary solid environment to prevent recoil. The source used for $^{57}$Fe Mössbauer Spectroscopy is $^{57}$Co in Rhodium matrix, with a mean lifetime of about 270 days and 25 mCi in strength.

![Decay scheme of $^{57}$Co](image)

Fig 2.11 The decay scheme of $^{57}$Co.

2.10.2 Absorbers

The absorber in Mossbauer experiments consists of a solid matrix containing resonating absorbing nuclei in which the gamma rays are scattered or transmitted through towards the detector. If the natural abundance of the Mossbauer isotope is large and if it is a part of the lattice under investigation, then there are no complications. If, however, the natural abundance of the Mossbauer isotope is low then sometimes it becomes necessary to enrich the absorbers. If the Mossbauer isotope is not a part of the lattice under investigation then it is necessary to incorporate it in the lattice as an impurity. It is necessary to see that the concentration
of resonating nuclei is kept as low as possible also to see that the impurity occupies
the lattice site in the material substitutionally.

In the normal transmission experiment the gamma rays emitted by the source pass
through the absorber, where they may be partially absorbed. In most cases the absorber
is the material to be investigated. The absorber thickness plays a significant role in the
quantitative analysis of the spectra. The effective absorber thickness \( t_A \) (for a single line
absorber) is usually defined by

\[
t_A = \sigma_0 f_A n_a d_A a_a
\]  

[2.9]

where, \( \sigma_0 \) is the maximum absorption cross section, \( f_A \) is the recoil-free fraction in the
absorber, \( n_a \) the number of atoms/cm\(^3\) of the particular element, \( d_A \) the physical thickness
of the absorber in cm and \( a_a \) the isotopic abundance of the resonance isotope. If \( t_A \ll 1 \)
the intensities of the resonance lines are small. If \( t_A \gg 1 \) the lines are broadened by
saturation effects. Usually experiments are carried out with a thickness of \( t_A \approx 1 \).

For good absorption and without broadening the line unduly, one should have about
1mg/cm\(^2\) of \(^{57}\text{Fe}\) in the absorber.

2.10.3 Detectors

In Mossbauer spectroscopy, one encounters gamma ray energies ranging from a few
keV to 150 keV. Consequently, the need for energy discrimination i.e. to separate out
the desirable gamma ray from the complicated emission spectrum calls for the use of
proportional counters, scintillation counters or semiconductor detectors and parallel
plate avalanche counters. The type of detector used determines the quality of the
gamma spectrum. For \(^{57}\text{Fe}\), the most common isotope in use, we need a detector with
good energy resolution. Gas filled proportional counters are the appropriate detectors
for reducing noise which have a resolution of \( \pm 15\% \) in the region of 14keV region
under ideal conditions.

The choice of gas is a compromise between the efficiency and signal to noise ratio.
Xenon gives the best efficiency and Argon gives the best signal to noise (S/N) ratio.
The noise level in scintillation counters with a very thin (0.22 mm) NaI(Tl) crystal is
larger than that of the proportional counters. Because of poorer resolution but lesser
efficiency for higher energy gamma rays, it works on the whole at an acceptable level
and one gets a good overall efficiency and long life. PIN diodes are also used as gamma ray detectors.

For the study of thin films using conversion electron Mossbauer effect, gas flow proportional counters are used.

2.10.4 Mossbauer Drives and Data Acquisition

In order to investigate the energy levels of the Mössbauer nucleus in the absorber it is necessary to modify the energy of the gamma rays emitted by the source so that they can have the correct energy for resonant absorption. This is accomplished by moving the source relative to a stationary absorber, and hence giving the gamma rays an energy shift as a result of the first order Doppler effect. The conversion has been adopted to plot the spectrum as a function of the velocity \( v \) (relative velocity of the source with respect to the absorber). Positive velocity is defined as the motion of source and absorber toward each other, and negative velocity as the motion of source and absorber away from each other.

The Mossbauer velocity drive imparts the source a precisely known and controlled velocity with respect to the stationary absorber and the experiment requires very small relative motion. The preferred Mossbauer drive is an electromechanical drive, sometimes called the velocity transducer. The most common and convenient one is the constant acceleration drive. In the constant acceleration mode, as the name implies the velocity of the source varies linearly with time. The constant acceleration requires the multichannel analyzer for data processing. The multichannel analyzer can be used either in 'Pulse Height Modulation mode' or in 'Multi Channel Scaling/Time mode'. In "Time mode" or Multi Channel Scale mode (MCS), each channel of the analyzer is opened and closed sequentially by a clock at controlled rate. The time spent per channel can be set very accurately. In our experiments, all the Mossbauer spectra were taken by using a constant acceleration spectrometer operated in the time mode.

2.10.5 Velocity Calibration

To obtain reliable Mossbauer parameters the drive velocity should be precisely known. Different velocity per channel can be set by using the amplitude knob of the drive unit. A maximum of \( \pm 25 \text{ mm/s} \) can be obtained with a high degree of linearity in maximum
Fig. 2.12 Typical calibration spectrum of $^{57}\text{Fe}$ for data analysis

dial settings. The calibration is made by a hyperfine spectrum of $^{57}\text{Fe}$ (Fig. 2.12 ) where the line positions are known accurately

2.10.6 Data Analysis

A lorentzian curve fitting program is used to fit the raw data. The minimum in $\chi^2$ is tested by specifying a number of random parameter displacements, after each of which a new fitting cycle is initiated, retaining the optimum parameters. The computer analysis of the Mossbauer data yielded the line width, Intensity, isomer shift, quadrupole splitting, hyperfine field and percentage area of each spectrum.
## References


4. J.E. Mee et al., Trans IEEE, MAG-5, 717 (1969)


