

MINERALOGICAL CHARACTERIZATION OF IRON ORES

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INTRODUCTION

Mineralogical characterization of iron ore is a very important and basic aspect that has to get due attention before any attempt for its processing and has become almost inevitable these days because of the increasing demand of the ore. Mineral processing technology is evolved to separate and recover ore minerals from gangue in a commercially viable method and is mainly based on the process of mineral liberation and the process of mineral separation. Therefore, it is important to first get a clear understanding about ore- and gangue minerals. A mineral is a natural inorganic substance having definite chemical composition and atomic structure. If the internal atomic arrangement is lacking, then it is an amorphous substance. A rock is generally composed of various minerals and if the rock contains valuable minerals from which metals can be extracted at a profit, it is called an 'ore'. The unwanted mineral in an ore is called gangue (i.e., generally rock forming minerals). For example, hematite (Fe_2O_3) and magnetite (Fe_3O_4) are oxides of iron and are ore forming minerals. In iron-ore, these minerals generally dominate but other unwanted minerals that occur are quartz (SiO_2), kaolinite (clay) etc., are gangue minerals. The terminologies 'ore' and 'gangue' are subjective. For example in a mine, when the ore/rock is mined with an objective to extract iron, hematite is the desired mineral and is called ore. The same mineral hematite if found in a sulfide ore (that is being mined to extract Pb, Zn or Cu) is called gangue. Therefore, 'ore' and 'gangue' terminologies can broadly be assumed as the desired and 'undesired' material in a rock mass respectively. Even though ore is mined with the highest efficiency of the technology, the excavated ore gets partly contaminated by the surrounding host rock (overburden) and the geological material closely associated with the ore during mining. Both the materials are undesirable and hence form the gangue.

From mineral processing point of view, it is important to identify the ore and gangue minerals (mineralogical study), and their textural relationships (grain size, grain boundary relationship, intergrowths etc.). It is also important to find out the modal distribution of ore and gangue minerals which decides the grade of ore. Grain size of the minerals and their textural relationship helps in deciding the size reduction and liberation in mineral processing. An effective liberation of gangue minerals and ore minerals influences the optimum separation efficiency. Therefore, ores must be characterized in terms of the above parameters before they are subjected to processing and it becomes easier to decide on the process that has to be adopted. Practical processes for the efficient recovery of metals and minerals from ores are a fundamental requirement for the health of any industry. These processes have been developed in response to declining ore grades, the need for new materials, increased environmental awareness, and changing market requirements. Much of this progress was

achieved by scientific inquiry into the nature and behavior of minerals and ores, aided by the development of new instruments and research techniques.

MINERAL CHARACTERIZATION TECHNIQUES

Optical Microscope (OM)

Mineral identification is the most important aspect that is needed to be carried out prior to beneficiation. The basic instrument used for mineral identification is an optical microscope. For this, polished sections of ores and thin sections of rocks are prepared. The ores/ore forming minerals are studied under reflected light microscope while the rocks/ rock forming minerals are studied under transmitted light microscope. The minerals are identified basically from their optical properties which differ from mineral to mineral because the differences in their crystal structure and chemical constituents. But mineral kingdom is so vast that sometimes it becomes difficult to identify all the minerals using only the optical microscope because there are cases where the optical properties of one mineral very closely resemble another. Also the resolution of optical microscopes is in the range of 50 to 1000 times magnification. In such cases the help of advanced characterization techniques such as X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) with micro-chemical analysis by WDS or EDS, and Electron Probe Micro-analysis (EPMA) are followed. The latter techniques help in confirming the mineral phases which is very important.

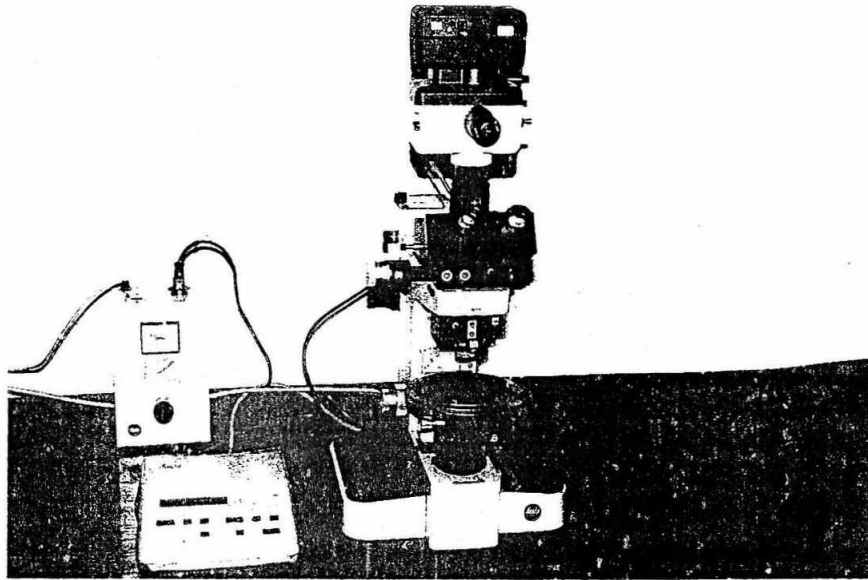


Fig. 2.1: Optical Petrological Microscope

The optical properties of few important iron-bearing minerals are given below. However, the subject will be more appreciated in the class room lecture where numerous case studies shall be discussed.

Hematite (Fe_2O_3)

It is the brightest among all the iron minerals and shows greyish white color with a bluish tint. Hematite can be distinguished from magnetite and wustite from its anisotropic character under crossed nicols. It generally occurs as dimensionally oriented elongated crystals (as it crystallizes in hexagonal system). However, basal sections show polygonal outline and are optically isotropic. Generally, recrystallization of hematite proceeds almost homogeneously and a large number of small crystals are formed by recrystallization. Reddish-brown internal reflection is also a characteristic feature of hematite crystals. Twinning in hematite is occasionally seen.

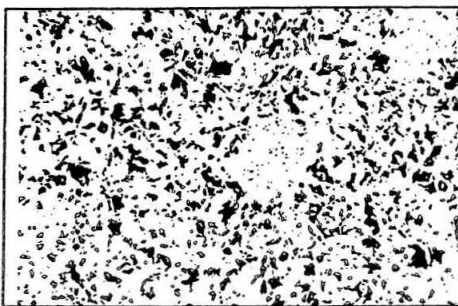


Fig. 2.2: Fine-grained acicular hematite in a massive type of sedimentary iron ore. Longer edge of photo equals to 600 μm .



Fig. 2.3: Coarse-grained magnetite crystals in triple junction arrangement in an igneous magnetite ore. The grain boundaries are filled with goethite (grey) and clay-gangue (black). Scale is same as in Fig. 2.2.



Fig. 2.4: Goethite showing colloform banding in a supergene type of iron ore. In the whitish band goethite has been dehydrated and converted into hematite. Scale is same as in Fig. 2.2.



Fig. 2.5: Martite (oxidized magnetite) crystals within a groundmass of fine crystallites of hematite in a supergene type of iron ore. The black portions are micro-pores (weathering pits). Scale is same as in Fig. 2.2

Magnetite (Fe_3O_4)

The color of magnetite under reflected light is brownish and the reflectivity is comparatively low as compared with hematite. Since it crystallizes in cubic system it shows isotropic behavior under crossed nicols and is easily identifiable. In general, magnetite exists in the form of equidimensional crystals. Massive crystals of irregular shape are also not uncommon. It also crystallizes in dendritic forms in sinters. In natural deposits, if the ores are exposed to oxidative atmosphere or weathering, the magnetite crystals oxidize to hematite called 'martite' (a modified pseudomorph of magnetite). Some magnetite ores contain maghemite ($\gamma\text{-Fe}_2\text{O}_3$) as well.

Goethite ($\text{Fe}_2\text{O}_3 \cdot \text{H}_2\text{O}$)

It is a hydrous iron oxide and commonly found in the lateritic profiles or surfaces of iron ore deposits. Goethite can form in prismatic crystals (orthorhombic) or more commonly as colloform bands. It is grey in plane polarized light and is characteristically anisotropic under crossed nicols that makes it easy to identify. The main constituent of limonite is goethite. The water is variable and it contains clays and other impurities. Therefore, limonite is a rock rather than a mineral.

Under the optical microscope, the gangue minerals appear dull (in grey or brown colors) in comparison to the ore minerals that reflect light. The interrelations between ore and gangue minerals in different iron ores are described in Figs. 2.12 to 2.15.

Zoom Stereo-Microscope

Zoom stereo-microscope is very useful in getting a three-dimensional view of mineral grains/particles. The magnification is of the order of 5 to 50. Therefore, loose fine-particles can readily be seen and comments can be made on their associational behavior. It is very helpful for studying process intermediate products such as that of tabling, spiraling etc.

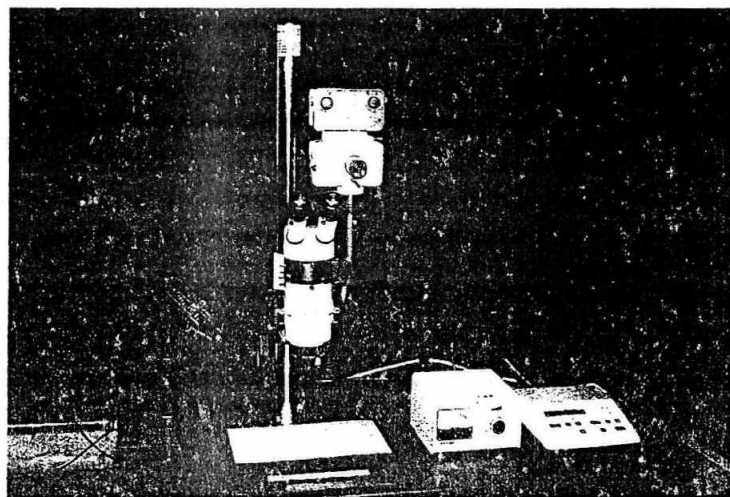


Fig. 2.6: Zoom Stereo-Microscope



Fig. 2.7: Photograph of -10+14# size fraction of an iron ore sample taken in a zoom stereo microscope. The grid in the photograph is 1mm X 1mm

Image Analysis System

Image Analysis System is just an attachment to an optical microscope where the image is captured by a high resolution camera and various parameters (area, shape, size, colour/grey-level) of the image can be quantified manually or by the help of softwares. This is very helpful in generating quantified data with respect to distribution of different minerals.

X-ray Diffraction

One of the most important and conclusive method to identify a crystalline mineral is by X-ray Diffraction. X-rays are somewhat like light waves but have much smaller wave-lengths, these being comparable to the distance between atoms in a crystalline solid. When a beam of X-rays falls on a crystal, it is scattered or diffracted by the parallel layers of atoms within the crystal, in the same way that light waves are diffracted by an optical grating. The powder method is more suitable instead of crystals because crystals are not always available and in a powder all possible crystal planes are represented.

In the powder method a beam of X-rays of one wavelength falls on finely-ground mineral powder. Diffraction takes place on the structural planes in the multitudes of randomly oriented crystal-particles. Certain particles with favourable orientations give reflections that are recorded electronically and are plotted in a paper in a continuous diffraction pattern which is called a diffractogram. Each mineral have its own diffractogram with characteristic

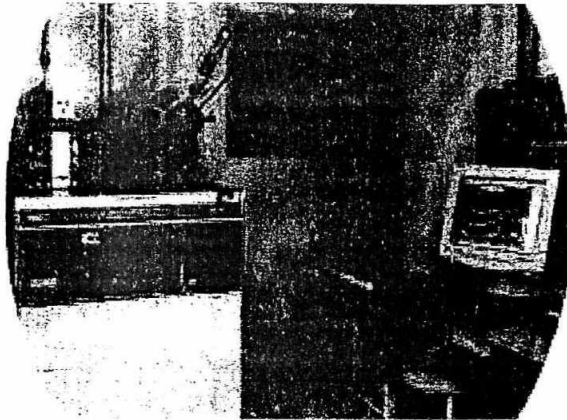
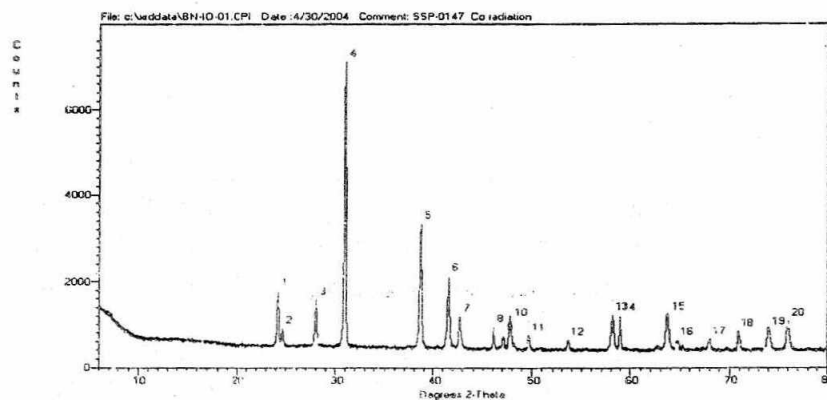


Fig. 2.8: Photograph of an X-ray Diffractometer

peaks. Since XRD pattern of individual minerals/phases are unique, this is considered as finger-print for a particular mineral. Standard diffraction patterns of thousands of minerals have been archived in JCPDS Data Book for reference matching with which an unknown mineral can be identified. In the diffractogram, the x-axis represents the angle (2θ) and the y-axis represents the intensity of the peak. Each angle corresponds to a particular distance (measured in Å) between two atomic planes. However, in a sample, if the volume percentage of a mineral is very low (say $< 5\%$), then XRD is not very helpful. The principle of X-ray diffraction for mineral/phase identification is based on the Bragg's Law (equation): $n\lambda = 2d \sin\theta$; Where, n = an integer; λ = wavelength; d = spacing between atomic planes; θ = reflecting angle, i.e., angle of incidence between the X-ray beam and the varying atomic planes.

Fig. 2.9: X-ray diffraction (XRD) pattern of one iron ore sample showing the peaks of quartz, hematite and



goethite. The prime peaks of the minerals are no. 4, 5 and 2 respectively. Goethite is a minor phase in this case.

Scanning Electron Microscope (SEM) + EDS / WDS

SEM provides a high degree of resolution even less than a micron. The magnification can be of the order of $>1,00,000$. Instead of light the energy source is an electron beam which is generated from a tungsten filament called electron gun. An aligned and finely focused electron beam falling on the sample surface is able to generate secondary electron images (SEI) and back-scattered electron images (BSI) which give morphological contrast and chemical compositional (atomic) contrast respectively. Attaching spectrometers such as EDS or WDS the chemical composition of different phases can be qualitatively or quantitatively measured.

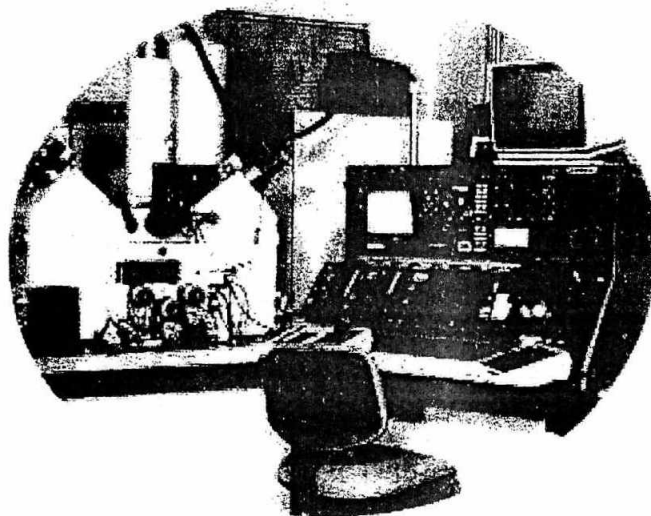


Fig. 2.10: Photograph of a Scanning Electron Microscope with EDS/WDS.

Electron Probe Micro-Analyzer (EPMA)

The basic unit of an EPMA is an SEM and the analytical spectrometer is in-built. Indeed, modern microprobe instrument is a hybrid between an SEM allowing measurement of high-resolution scanning electron photomicrograph as well as incorporating a fully quantitative x-ray analysis capability. It involves the excitation and chemical analysis of selected areas of diameter as small as a few microns on the surface of the sample. It has ability to determine the composition of individual minerals or even concentration variations within a single grain. An electron beam, accelerated to a selected voltage, normally in the range 15 to 30 kV, is focused on the surface of a sample. Interactions between this primary electron beam and the sample cause a number of phenomena, including the generation of X-rays characteristic of the atoms of the excited sample. The intensity of these X-rays is measured using WDS or EDS. After corrections of matrix effects, count data are compared with data from minerals of standard composition in order to quantify the analysis.

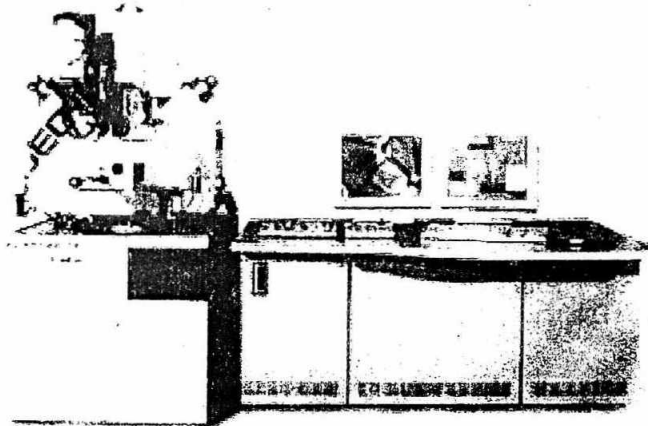


Fig. 2.11: Photograph of an Electron Probe Micro Analyser.

MODE OF OCCURRENCE AND LIBERATION STUDIES

Ore Texture- Complexities and Liberation of Minerals

It has commonly been asserted that mineral processing consists of two steps, liberation and separation, and the second step is impracticable if the first has not been successfully accomplished. One of the principal objectives of comminution is to liberate dissimilar minerals from attachment to each other. The liberation characteristics of minerals in an ore primarily depend on the ore-texture, i.e., the mutual relationships between grains. The parameters that guide the liberation are shape and size of the grain, their inter-locking nature, hard- or softness of the adjacent minerals, mineral compositions etc. There are varieties of ore textures such as exsolutions, inclusions, intergrowths, myrmekitic intergrowths, colloform, dendritic, framboidal etc. to name a few. Each type of texture will affect differently in the process of liberation of minerals. Therefore, textural studies play a very vital role in liberation studies.

One of the primary roles of comminution in mineral beneficiation is the liberation of valuable minerals. The extent of liberation achieved during comminution can play an important role in determining the final concentrate grades and recoveries obtainable in a mineral processing plant. In froth flotation, for example, a low concentrate grade can be attributed to the recovery of composite particles, or the non-selective recovery of liberated gangue material. In either case, the corrective action needed to improve the grade is highly dependent upon knowing the manner in which free and composite particles are distributed in size. Furthermore, by knowing this information as material leaves the mill, it may be possible to optimize the grinding circuit on the basis of both size consist and mineral content distribution.

Quantification

The description of multiphase particles has always been of special interest to the mineral processing engineers. In its most abbreviated form this description, liberation analysis, is simply a measure of the fraction of a particular phase that is free from all other phases, i.e., liberated, without regard to distribution to grade or particle size for that matter. In its most complete form liberation analysis involves the determination of the volumetric abundance or volumetric grade distribution of a particular phase for each particle size. Data necessary for liberation analysis is frequently obtained as one or two dimensional microscopic information from the polished sections of mineral particles of specified size. This data overestimates the extent of liberation and in the past an empirical correction (locking) factor has been used to give a better estimate of the extent of dispersed phase liberation. The one or two dimensional information can be taken either as linear intercept measurements or as projected aerial measurements, each of which is influenced to some extent by all values of the volumetric grade distribution of the dispersed mineral phase. For detailed liberation analysis, the volumetric grade distribution of multiphase mineral particles can be estimated from one or two dimensional information obtained by image analysis of polished sections by establishing transformation matrix and computer simulation.

The degree of liberation of a certain mineral or phase is the percentage of that mineral or phase occurring as free particles in relation to the total of that mineral occurring in the free and locked forms. Conversely, the degree of locking of a mineral is the percentage occurring in locked particles in relation to the total occurring in the free and locked forms.

Today, liberation is the hottest field in mineral processing, because of the measurement techniques available, and, more significant, the growing understanding of the physical process of locked particle formation. With the development of image analysis for the assaying of locked particles, large amounts of increasingly accurate data is becoming available for interpretation. Studies have shown that not only are locked particles impossible to eliminate, but the problem of making good cuts between the waste mineral and the locked particle is more severe than realized. A disproportionate amount of the locked particles possess only trace of the valuable constituent. In a deeper sense, it is possible to compute for the ideal case the maximum possible recovery of a given mineral, to formulate the optimum strategy for recovering the target mineral and, in a wider sense, understand how locked particles affect recovery processes.

TYPICAL EXAMPLES

Few examples on textures and liberation of iron ore are presented in Figs. 2.12 to 2.15.



Fig. 2.12: Optical photo-micrograph of martite/hematite (whitish bigger grains) with inclusions/intergrowths of quartz (grey). Black patches are clay. Plane polarized reflected light X 200. The longer edge of the photo equals 600 μ m and average grain size of hematite/martite is slightly more than 100 μ m.

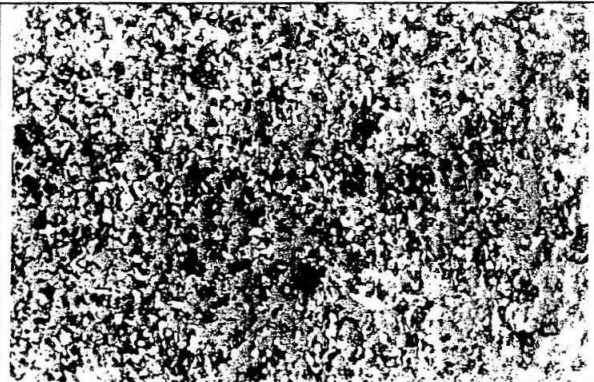


Fig. 2.13: Optical photo-micrograph showing dispersion of fine crystallites of hematite (white) along with quartz (grey). Black patches are micro-pores sometimes filled with clay. Plane polarized reflected light X 200. The scale is same as in Fig. 12.

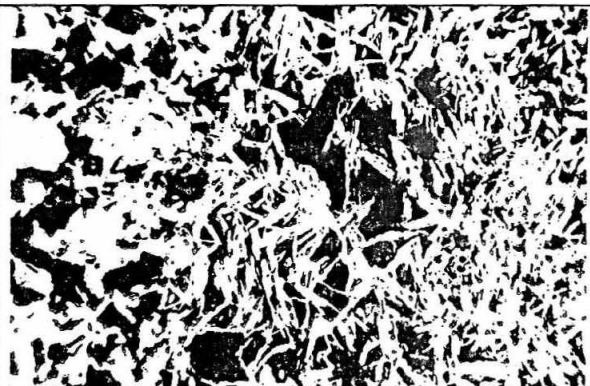


Fig. 2.14: Optical photo-micrograph showing patches of clay (dark grey) within a matrix of acicular/micro-platy hematite (white) in a massive variety of iron ore. The clay is difficult to remove by normal washing. The scale is same as in Fig. 12.

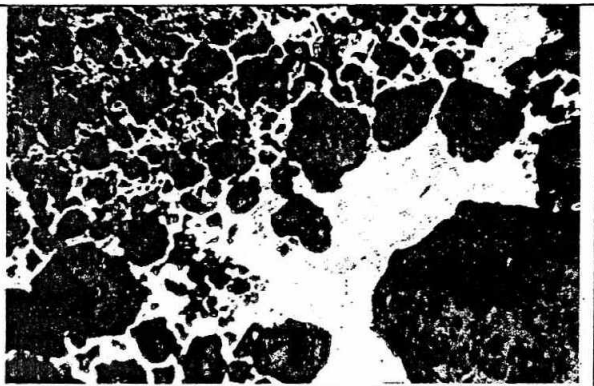


Fig. 2.15: Optical photo-micrograph, showing occurrence of gibbsitic clay (brown) that is bounded by goethite (grayish white), a typical characteristic of the lateritic iron ore. This type of clay is difficult to remove. The scale is same as in Fig. 12.