

CHAPTER II

HEXAGONAL FERRITES

HEXAGONAL FERRITES

2.1 Introduction

A magnetic material which can be prepared with an appropriate microstructure (i.e. in the form of fine elongated particles) can be used as a permanent magnet material. Lot of efforts are being continuously taken towards the development of the newer and newer permanent magnet materials. The improvement of properties of existing magnet materials through the control of their chemical composition and microstructure. Permanent magnets find wide applications in the electrical and electronic industry technology. Starting from the use of conventional steel magnets in the early days, lot of advances have been made with regard to finding newer permanent magnet materials like alnico, vicalloy, Cu, Ni, Fe, Pt, Co, R Cos etc. The hexagonal ferrites (hard ferrites) also form the part of this list, because of their relatively inexpensive nature and the ease of fabrication, apart from their characteristic permanent magnet qualities. After discussing what is required of a permanent magnet, account has been given here of the composition, crystal structure and properties of hexagonal ferrites. A brief review of the recent research work on these materials is also included at the end.

2.2 Magnetic considerations of a Permanent Magnet Material.

2.2. a Magnetic hysteresis

The requirements of a good permanent magnet material can well be explained with the help of hysteresis loop. Such a loop for a virgin ferromagnetic sample is shown in Fig. 2.1. It can be seen that, increasing the applied field, H , causes the induction to increase to its saturation value, B_s . Upon decreasing the value of the applied field to zero the induction falls to the value B_r , the remanent induction. Reversing the direction of the magnetic field and increasing its value causes the induction to fall to zero, define the coercive force, H_C . On further increasing the value of $-H$, the induction increases in the $-y$ direction to $-B_s$. By decreasing the applied field to zero, the induction does not return on the same path, but rather decreases to a value equivalent to a negative B_r as shown. On reversing and increasing the applied field, the induction falls to zero at a point equivalent to a positive H_C . By further increasing H , the loop is closed at B_s . Incidentally, the first path taken by the sample from the origin to B_s . Called the virgin curve, can not be reproduced unless the sample is completely demagnetised.

Whenever a permanent magnet forms the part of the circuit in which there is an air gap, it is subjected to a

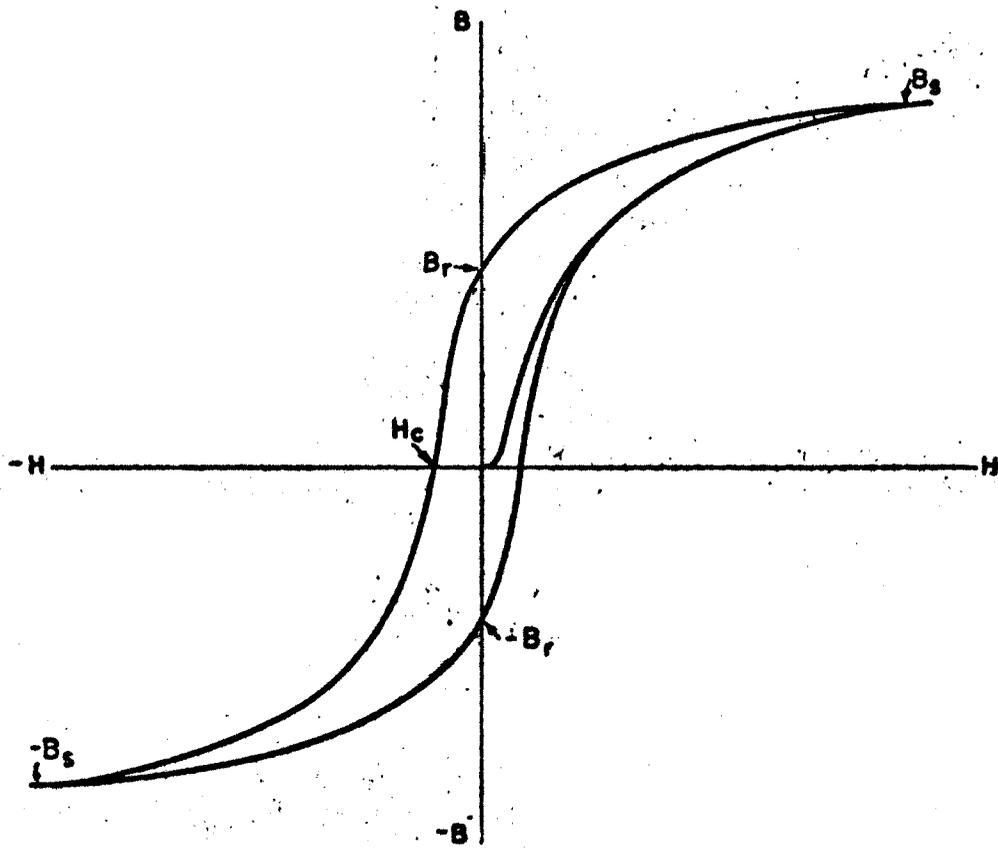


Fig. 2.1 Path of magnetization about a hysteresis loop.

demagnetising field. It is therefore the second quadrant of the hysteresis loop, usually called as the demagnetization curve, which is of interest to the permanent magnet materials. By finding the product of B and H which is maximum for points along the curve the qualifying factor called the (BH) maximum or the energy product is defined. It is this energy product which is used a figure of merit for permanent magnet materials. In so far as magnet design is concerned, it will suffice to say that by arranging the magnet dimensions such that the operating point coincides with the point of (BH) maximum most of the magnetic energy can be desired from the magnet material.

There is another factor called the 'fullness factor' which is used to describe the squareness of the BH curve. It is expressed as

$$K = \frac{(BH) \text{ max}}{B_r H_c} \dots \dots \dots (2.1)$$

The value of K can range from 1 for square loop, to 0.25 for a straight line. A demagnetization curve described by a straight line is indicative of a material which is magnetically isotropic. On the other hand, greater values of fullness factor imply that there exists a magnetic anisotropy.

2.2.b Crystal Anisotropy

The magnitude of the crystal anisotropy energy is expressed either by an anisotropy constant, K , or by an effective anisotropy field, H_a . The anisotropy constant, K , is measure of the energy required to turn the magnetization vector from the C axis into the basal plane. It may be either positive or negative. If K is positive, the C axis is the easy direction of magnetization. If K is negative, the basal plane contains the easy direction(s) of magnetization. The anisotropy field, H_a , is related to the anisotropy constant, K by the relation

$$H_a = 2 K/M_s \dots \dots \dots (2.2)$$

M_s = Saturation magnetization.

In the use of ferrites as magnetic cores in inductive circuits operating at ultrahigh frequencies the initial permeability is of paramount importance. In this frequency range the permeability results from the rotation of electron-spin axes under the influence of the alternating magnetic field. At frequencies below a certain limiting frequency the initial permeability, μ , is given by

$$\mu = C M_s/H_a \dots \dots \dots (2.3)$$

In this approximate relation, C is a constant whose exact value is irrelevant here. The limiting frequency is

that frequency at which a resonance occurs, a resonance involving the mass of the electron-spin system and the attractive force of the anisotropy field. At this frequency the deflection of the spin axis is very large, and the energy loss in the material is correspondingly large. Above the resonance frequency the initial permeability drops to a very low value.

The limiting frequency of a cubic ferrite is

$$2\pi fc = \gamma H_a \dots \dots \dots (2.4)$$

Where r is the gyromagnetic ratio (nominally 2-8 (2π)) if H_a is expressed in oerstedes and fc in megacycles per second) on combining eqns (2.3) and (2.4) it is found that $(\mu fc)/(\gamma Ms)$ is a constant. An increase in fc , then, may be brought about either by decreasing μ or by increasing Ms . The first alternative obviously is undesirable. The second alternative already has been developed as far as seems to be possible. The frequency range in which cubic ferrites have useful permeability values is thus limited by this relation to a few hundred megacycles.

A different situation exists in the hexagonal materials.

If H_{A_1} denotes the anisotropy field for relation within the basal plane and H_{A_2} the anisotropy field corresponding to relation out of the plane, the resonance frequency is

$$2\pi fc = \gamma \sqrt{H_{A_1} H_{A_2}} \dots \dots \dots (2.5)$$

and the initial permeability is proportional to

$$M_s/H_{A_1}$$

In many of the hexagonal compounds the anisotropy field H_{A_1} is of the same order of magnitude as the anisotropy field of cubic ferrites; hence comparable values of initial permeability are found. The other anisotropy field, H_{A_2} , may be very much larger, and the resonance frequency is correspondingly increased. Thus, it should be possible to extend the frequency range within which useful values of permeability are found by a suitable choice of anisotropy constants.

2.3 Hexagonal ferrites

The group of compounds having the general formula $MeO \cdot 6Fe_2O_3$, where Me is Ba, Sr or Pb or a mixture thereof which are commonly known as hexaferrites or hexagonal ferrites have been successfully developed and used as the basic materials for the manufacture of ceramic permanent magnets¹. This is not only because they can be produced from relatively inexpensive raw materials, but also, this type of magnet has several properties that for certain applications, are superior to those of conventional metallic magnets mentioned above. The ceramic permanent magnet is characterised by a high coercive force, high permeability, relatively narrow ferrimagnetic resonance,

high thermal and electrical conductivity line width at wave frequency the maximum energy product and is chemically inert. These properties permit the utilization of hexagonal ferrites in electronic components at microwave and higher frequencies and other equally important areas of application, as in loudspeaker, television, telephones, generators and motor, to name but a few.

These compounds are found within the ternary compositional diagram $\text{BaO} - \text{MeO} - \text{Fe}_2\text{O}_3$ shown in fig. 2.2. The well known spinel ferrites can also be seen in the same diagram. The symbol Me represents the divalent metal ions like Co, Zn, Cu, Ni, Mg, Fe or Mn. As in the spinel ferrites the trivalent Fe ions may be partially replaced by other like Al^{3+} , Cr^{3+} , Mn^{3+} or a trivalent ion combination like Ti^{4+} , Co^{2+} . Of the compositions shown in fig. 2.2. only the ferrite is of the cubic crystallographic system. The permanent magnet barium ferrite as well as other compounds are however hexagonal. Associated with this hexagonal crystal structure hexagonal compounds have a high crystalline magnetic anisotropy which varies in sign and in magnitude according to the composition. The anisotropy is such that the permeability along the unique axis may be very much larger or smaller than the permeability in the basal plane. It is this large difference in permeability which makes it possible to prepare polycrystalline permanent magnet specimens which are magnetically oriented so that the

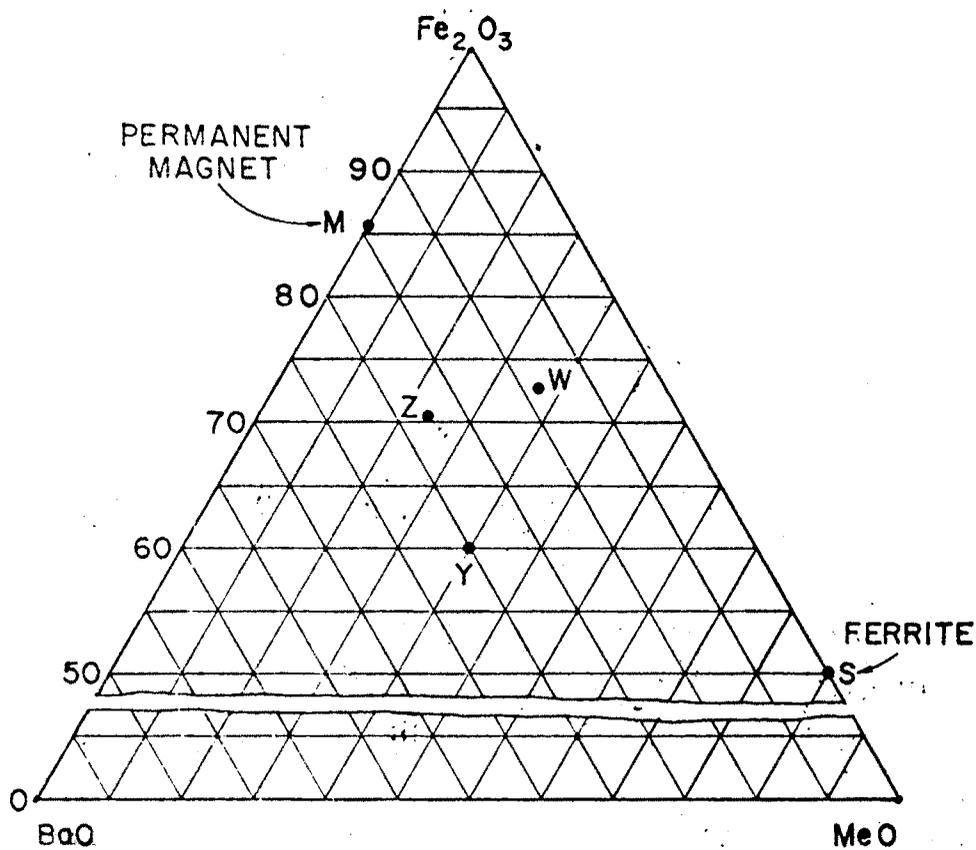


Fig 2.2: The system $\text{BaO}-\text{MeO}-\text{Fe}_2\text{O}_3$. $M = \text{BaFe}_{12}\text{O}_{19}$, $Y = \text{Ba}_2\text{Me}_2\text{Fe}_{12}\text{O}_{22}$, $Z = \text{Ba}_2\text{Me}_2\text{Fe}_{24}\text{O}_{41}$, $W = \text{BaMe}_2\text{Fe}_{16}\text{O}_{27}$, and $S = \text{MeFe}_2\text{O}_4$. $\text{Me} = \text{Co, Zn, Cu, Ni, Mg, Fe, or Mn}$.

C axes of all the individual crystallites are parallel.

The direction of high permeability is obviously a direction of easy magnetization and the direction of low permeability is a direction of hard magnetization.

The magnetic properties of the hexagonal ferrites which distinguish them from cubic spinel ferrites are found in those compositions in which the C-axis is hard direction and the basal plane, a plane of easy magnetization. Although there is a small anisotropy in the basal plane of crystallites in the finished polycrystalline specimen the crystal axes in the basal plane are randomly oriented.

There are numerous factors affecting the final magnetic properties of a ceramic ferrite permanent magnet body. These factors can be divided into two major groups. Firstly, there are chemical factors; these include the purities and relative proportions of the major constituent oxides and the amounts of the various possible doping elements. Secondly, technological factors; these include the parameters governing the methods of preparation. The latter of these have a profound effect on the microstructure of ferrite magnets.

To increase the merit of a permanent magnet (viz. the energy product) one must increase the remanent magnetization while keeping the coercive force sufficiently high to retain its permanent magnet quality. The metal magnets have a greater value of saturation (and remanence) while generally showing a

lower coercive force and incidently, a lower electrical resistivity. The remanent magnetization of a ceramic magnet depends upon its density for a given degree of particle orientation. The coercive force on the other hand, depends strongly on the grain size of the ceramic specimen. In general smaller the grain size, the higher will be the coercive force for these reasons, the aims in making a good ferrite permanent magnet are to produce a well oriented material in order to take the maximum advantage of its anisotropic property and to attain highest possible density and the smallest possible grain size.

2.3.1. Crystal Structure of Hexagonal Ferrite

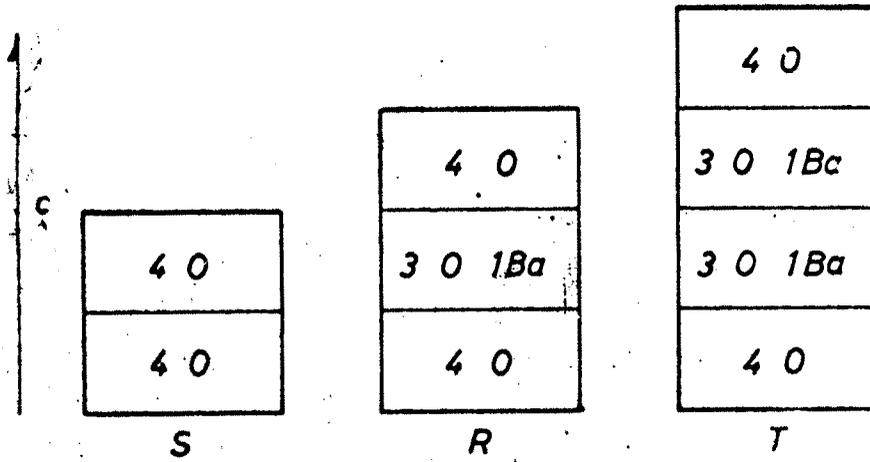
The unit cell is hexagonal and contains two molecules of $\text{Me Fe}_{12}\text{O}_{19}$. The unit cell parameters of some important compounds of this class are given in table 2.1

It is easy to describe the structure in terms two blocks, namely R and S blocks (fig. 2.3). The S block is a spinel block with its cubic $\langle 111 \rangle$ axis oriented parallel to the C axis of the hexagonal cell. One such block consists of two oxygen layers, each layer containing four oxygen ions. The corresponding six Fe^{+3} ions are distributed over two tetrahedral and four octahedral sites. The R block is made up of three oxygen layers. The two terminal layers contains four oxygen ions each, while the central layer has three oxygen ions and one Me ion. In this block we have five metal ions at the octahedral sites and one at trigonal bipyramidal site (five fold co-ordination).

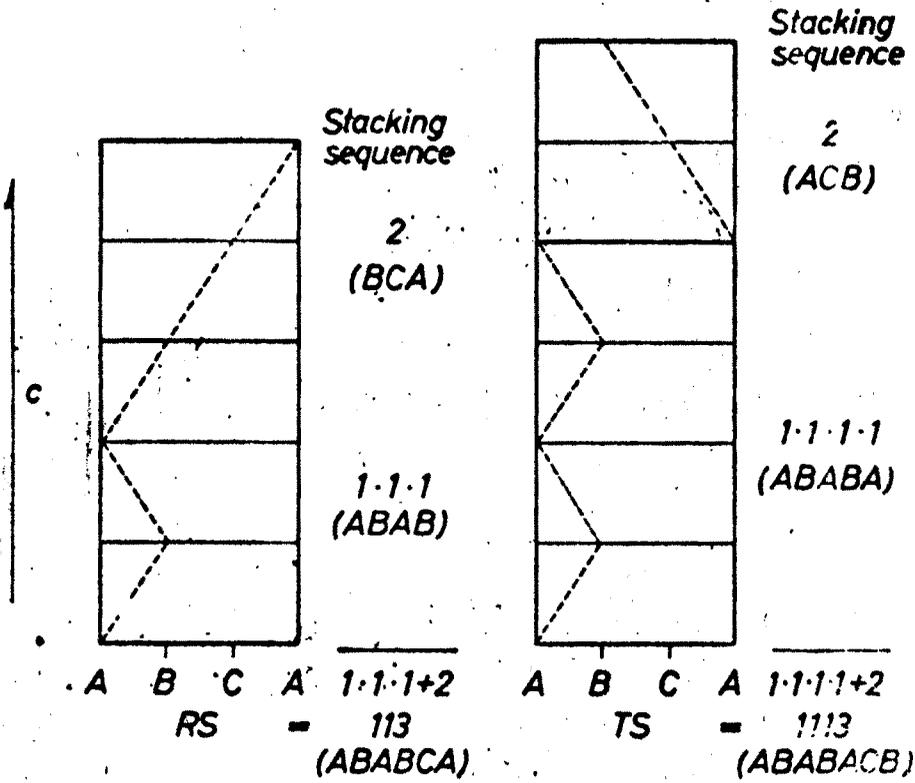
Table 2.1 : Magnetic Properties of Hexagonal Ferrites (1,2,3,4)

Compound	C, Å	a, Å	20°C		0°K		
			I_S	$K_1 \text{ or } K_1 + 2K_2$ $\times 10^{-6} \text{ erg/cm}^3$	HK (KOe)	n_B μ_B	θ_C °C
BaM	23.18	5.889	380	3.3	17.0	20	450
SrM	23.03	5.864	370	3.5	20.0	20.6	460
Fe ₂ M	32.84	5.88	320	3.0	19.0	27.4	455
Zn ₂ M	43.56	5.88	227	(-1.0)	9.0	18.4	130
Cu ₂ M	52.30	5.88	267	(-1.8)	13.0	31.2	410
Zn ₂ U	113.2	5.88	295	1.4	9.6	60.5	400





Derivation of the three building block types, *R*, *S* and *T*, from the two framework layers



The *RS* and *TS* structural units, showing the framework layer stacking along *c*

Fig. 2.3



The complete M unit cell is built up by alternate stacking of R and S units in the sequence of RSR*S* where * indicates that these blocks are rotated by 180° around the C axis with respect to R and S blocks. The unit cell thus contains ten oxygen layers with a Me ion replacing an O^{2-} ion every fifth layer. The distribution of Fe^{3+} ions and spin orientations are as follows (in half unit cell only)

S Section : 2 tet \uparrow 4 oct \downarrow

R Section : 1 five fold \downarrow 2 oct \uparrow + 3 oct \downarrow

The net magnetic moment is $4 \times 5 u_B = 20 u_B$ per formula unit. This calculated value agrees with extrapolated experimental value of magnetic moment at $0^\circ K$.

The other important hexagonal ferrites, which have structure related to that of the magnetoplumbites are W, Y, Z, X and U compounds. (fig. 2.2 and figs. 2.4, 2.5, 2.6). Their chemical formulas and structures are given in table 2.2

These structures are described in terms of S, R and T blocks. The T block is made up of four oxygen layers with a Me ion substituted for an oxygen ion in each of the two middle layers.

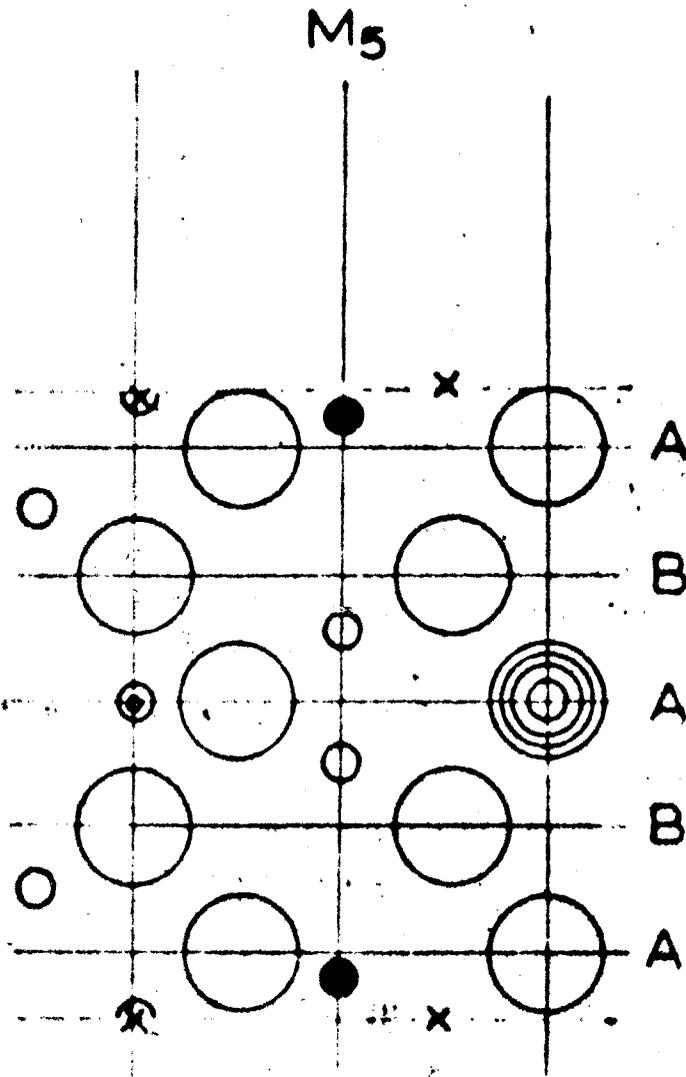
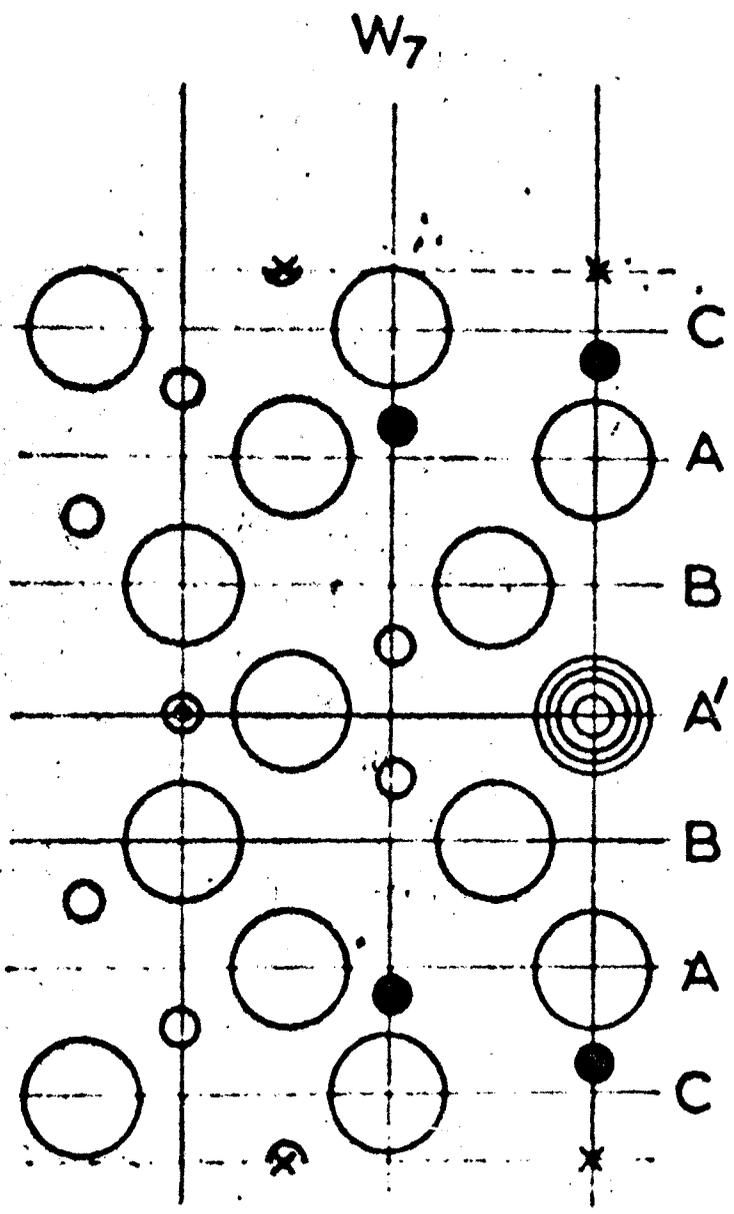
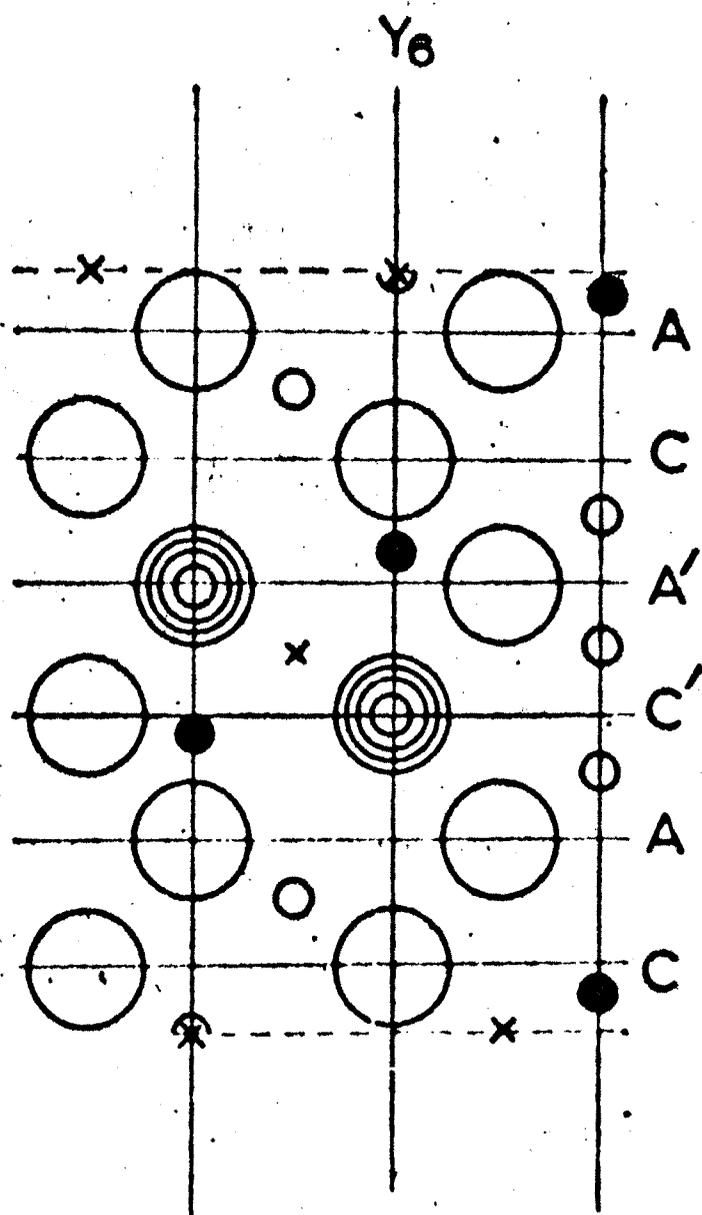


Fig 2.4 : M - Structure



(b)

Fig. 2.5 : W - Structure



(c)

Fig. 2.6: Y-Structure

TABLE 2.2

CHEMICAL FORMULAS AND STRUCTURES OF THE
MORE COMPLEX HEXAGONAL FERRITES

Compound	Chemical Composition	Unit cell	Number of formulas per unit cell
M	Ba Fe ₁₂ O ₁₉	RS R* S*	2M
W	Ba Me ₂ Fe ₁₂ O ₂₇	RSSR* S* S*	2MeW
Y	Ba ₂ Me ₂ Fe ₁₂ O ₃₈	3 (ST)	3MeY
Z	Ba ₃ Me ₂ Fe ₂₄ O ₄₁	RSTSR*S*T*S*	ZMeZ
X	Ba ₂ Me ₂ F ₂₈ O ₄₆	3 (RSR* SS)	3MeX
V	Ba ₄ Me ₂ Fe ₃₆ O ₆₀	RSR*S* T* S*	MeV

2.3.2 Magnetization

Barium ferrite has been used commercially as permanent magnet material, as a major component of Ferroxdure and Magnadure. A figure of merit for such materials is often given as the maximum value of (BH) . This can be seen in the following simple manner. Suppose it is required to produce a total flux ϕ in an air gap of reluctance R and to do this a length l of magnetic material of uniform cross section A is used in fig. 2.7. Suppose the magnet is first magnetized to saturation with the air gap closed by high-permeability material and the magnetizing field is then removed. The flux density in the material will then fall to the remanent value corresponding to the point C in fig. 2.7. If the permeable material is now removed from the air gap, free poles will appear on the pole faces and set up in the magnetic material a reverse demagnetizing field such that the working point falls to D. At this point the flux in the gap must be the required ϕ , with H the reverse field.

Thus the magnetomotive force $= \phi R = Hl$. Since the total m.m.f. in the complete magnetic circuit is zero. There will be some leakage of flux around the magnetic circuit, particularly in the region of the poles and a leakage factor K may be defined as the ratio of total flux to useful flux. Thus $K = BA/\phi$, where B is the magnetic induction in the magnetic material.

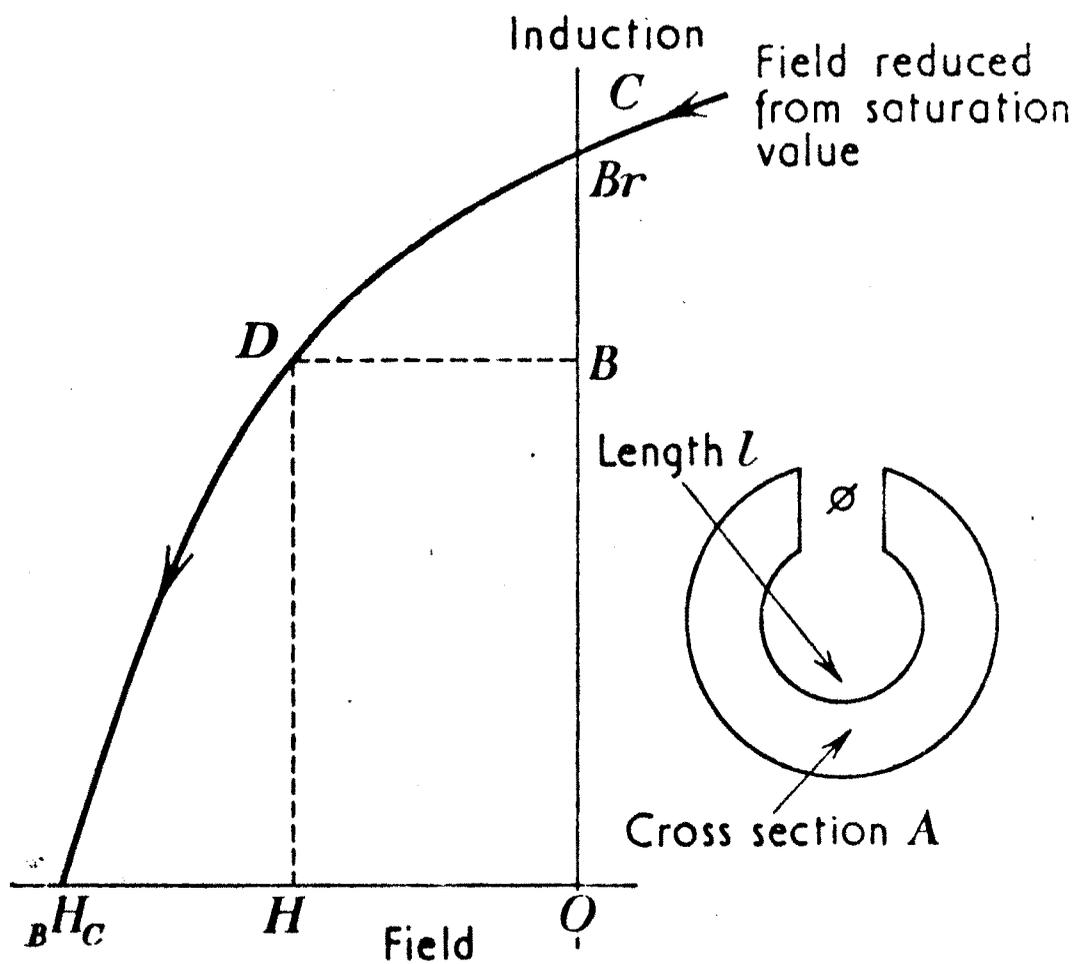


FIG. 2.7 Demagnetization curve of a magnetic material.

Hence to produce the useful flux ϕ we require the total volume of magnetic material A , where

$$\lambda A = \frac{R}{H} \times \frac{K \phi}{B} = \frac{K R \phi^2}{(BH)} \dots \dots \dots (2.6)$$

This will be minimum when (BH) has a maximum value; hence the use of $(BH)_{\max}$ for a permanent magnet material as a figure of merit.

Of course, high remanence and high coercive force are necessary as well as high value of BH . If a material is magnetized to saturation, and the magnetizing field is removed, the remanent magnetization M_r remains and the remanent induction $B_r = 4\pi M_r$. If the magnetizing field is reversed and increased in the reverse direction, the magnetization and the induction become zero though not in the same applied field. The fields are the coercive fields M_c and H_c respectively.

2.3.3 Magnetocrystalline - Anisotropy

The most interesting ferrites of this group are the Y ferrites and the cobalt-substituted 'W' and 'Z' compounds. In these ferrites the anisotropy is predominant in the basal ab plane instead of the C-axis. Therefore, these ferrites are called Ferroplana to differentiate them from the Ferrordure permanent magnets. It should be noted that cobalt substitutions

in the M structure also lead to Ferroplana properties. The anisotropy constants K_1 and K_2 in a hexagonal crystals are derived from the magnetocrystalline anisotropy energy relationship which has following relation.

$$E_a = K_1 \sin^2 \theta + K_2 \sin^4 \theta + K_3^1 \sin^6 \theta + K_3 \sin^6 \theta \cos 6(\theta + \phi) \dots (2.7)$$

where θ is the angle between the magnetic moment M and the anisotropy field H_a . The angles θ and ϕ are the polar angles where the C axis coincides with the z-axis and ϕ is a phase angle. The direction cosines of the magnetization are

$$\alpha_1 = \sin \theta \cos \phi, \alpha_2 = \sin \theta \sin \phi, \alpha_3 = \cos \theta \dots (2.8).$$

The phase angle can be made zero for certain chosen axis.

The anisotropy fields are

$$\begin{aligned} H_a &= 2 K_1 / M & (\theta = 0^\circ) \\ H_a^1 &= 2 (K_1 + 2K_2) / M & (\theta = 90^\circ) \dots (2.9) \end{aligned}$$

If the c - axis is the preferred direction of magnetization the anisotropy constant K_1 will be measured. In the case that the base plane is the preferred direction of magnetization $K_1 + 2K_2$ is measured.

2.4 A brief review of Recent Research work on hexagonal Ferrites.

After their discovery in 1956-57 by Jonker et al.,⁵ the hexagonal ferrites have been extensively studied. The studies are mainly directed towards the understanding of formation mechanism, reaction kinetics, formations of crystal structure, observation of domain structure and the characterization of physical properties. A brief review of the recent research work on these ferrites is present here.

The classical method of preparation of permanent magnet type ferrite powders is by mixing the compounds as powders in a ball mill and reacting them by means of heating. The detailed preparation of these materials has been described by stuijts⁶ and by stuijts et al.,⁷ It could be remembered that the permanent magnet characteristics of hexagonal ferrites depend largely upon the sintering procedure and the control of compositions. Considerable improvement in properties has been achieved in this manner by several workers⁸⁻¹³ variety of similar ferrimagnetic oxides with closely related hexagonal structure can be prepared by the appropriate selection of the constituent oxides and the replacement of divalent metal ions^{5,14}. The attempts for preparing permanent magnet type ferrite powders with improved properties by co-precipitation method have been made by Sutarno and Bowman¹⁵, Basel and Hempel¹⁶

and Von Basel¹⁷. Ba-ferrite in the form of thin films have been prepared by DC diode sputtering by M.NaOe¹⁸.

As mentioned earlier the present interest in hexagonal ferrites stems from their possible use as permanent magnet material and other electronic devices. Many of the μ wave and mm wave frequency devices operate in the ferromagnetic resonance state. The frequency of this resonance is the function of the nature and the magnitude of anisotropic magnetic field present in the particular hexagonal ferrite. The efficiency of a resonance device depends in part, on the resonance line width; the narrower the line width the more efficient is the device. Narrow line width, as well as the best measure of intrinsic properties, are attainable from single crystal.

A number of investigations concerned with the growth of single crystal of ferroxdure ($\text{Ba Fe}_{12}\text{O}_{19}$) have been reported.¹⁹⁻²⁰

Growth and characterization of single crystals of hexagonal ferrites with M,Y,W,Z,X,V structure has reported by savage and Tanber (1964)²¹ and the recrystallization of ba-ferrite and other substituted composition under high pressure oxygen recently by Menashi (1973)²².

Magnetic and electric properties of ferrite single crystals with Y structure and described by J. Verweel²³. He has also reported on anisotropic electrical resistivity anomaly in these single crystal similar to Zaveta²⁴.

The fact that the anisotropy of the resistivity is a structural effect and is not due to sample preparation, has been established from these measurements.

Magnetization of a single crystal of hexagonal ferrite has been measured by Fonton and Zaleski (1965)²⁵ and recently by Licce²⁶.

The property of low anisotropy in the basal plane and large anisotropy out of plane is common to all compounds with 'Y' structure. The Zn_2 Y compounds have been particularly studied and discussed by Verweel (1967) in relation to μ wave applications²³. Theretical calculations on anisotropy were carried out later on by Xu et al²⁷.

Measurement of complex dielectric constant and field dependent conductivity were carried out on polycrystalline Ba ferrite. It has been observed that it shows two distinct relaxations in the variation of complex dielectric constant and frequency²⁸.

Low temperature heat capacity for Ba-hexaferrite along with other ferrites has been measured in the temperature range 90 K to 303 K and it is found that it decrease with decreasing temperature²⁹. Hall effect measurements on Ba-ferrite have been successfully used by Napijalo and Zizic^{30,31} for determining the type carries and also the mechanism of electrical condition.

They have proposed Zane mechanism as the conduction mechanism. Lot of research is diverted towards the studies of substituted hexaferrites³²⁻⁴¹. The important results these measurements can be concluded by noting that

- 1) The substitution of Ce or La for Ba in BaW improves partially the chemical stability of W type ferrite³⁴.
- 2) (i) Nickel and Cu occupy the same position as Zinc in Zm_2W ³⁵
 (ii) Cu-substitution improves the permanent magnet properties of Zm_2W ³⁵.
- 3) Intrinsic magnetic properties of Ba and Sr ferrites do not change appreciably with increasing Ca-content.
- 4) Co-substitution in hexaferrites with M,W,Y structure exerts a big effect on the magnetic ordering and magnetic anisotropy³⁹.
- 5) In, Sc prefer positions belonging to R block^{40, 41}

Mechanical properties of hexaferrites are also as interesting as their electrical and magnetic properties. Investigations on fracture of isotropic and textured Ba-hexaferrite, deformation and grain orientation of polycrystalline hexaferrite under uniaxial compressive load, mechanical properties, microstructural fracture anisotropy have been reported from time to time⁴²⁻⁴⁵.

Low temperature thermal expansion has been reported by Clerk et al.,⁴⁶ . The advanced techniques like FMR⁴⁷⁻⁵² and Mossbauer^{32,40,41,53} , have been advantageously used from the beginning in characterizing these ferrites.

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