Photoinduced Irreversible Effects on Magnetic Properties and Allied Phenomena in Magnetic Oxides. I*

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Abstract : This is a survey of the photoinduced irreversible effects and related phenomena in magnetic oxides, mainly YIG. Although several pictures on these phenomena and an plausible explanation have been proposed, these have not been established as yet. This survey will concentrate on the authors' experiments and pictures of these subjects, rather than the other researcher's. This is roughly divided into the four separate parts; I. Experimental, II. Photoinduced irreversible permeability effect, III. Allied phenomena, IV. Conclusive discussion.

I. Inttroduction

It has been found that a number of magnetic properties of materials like garnets and ferrites can be changed by illumination. The effect is seen in the magnetic anisotropy [1, 2], the initial permeability [3], the coercive force [4], magnetic switching properties [4], the optical absorption [5], the magnetostriction [6], and magnetic linear dichroism [7, 8, 9]. The materials of which such photomagnetic phenomena or photoinduced magnetic effects (PME) have been observd are magnetic insulators and magnetic semiconductors: yttrium iron garnet with various dopants [10], Ga-doped $CdCr_2Se_4$ [11], $FeBO_3$ [12] and some ferrites [13]. Here, we prefer the term "photoinduced", since the term "photomagnetic" is often confused with "magnetooptic" [8]. The physical mechanism underlying all these phenomena is not fully understood. It seems not determined that an essential condition for PME is the presence of ions which may assume different valence state. If this is the case, howover, the transition of an electron between two such ions effectively changes the ion distribution: for an example, in a crystal with a few Fe^{2+} ions surrounded by many Fe^{3+} ions. Electron motion means an effective ferrous ion displacement. PME are based on changes in the ion distribution caused by light-induced electron transitions. The change in magnetic properties can be caused

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either by a change in local symmetry of the displaced ions or by a change in their ionic surroundings. Depending on temperature, the light-induced distribution changes can be persistent or relax by thermal electron motion. Two classes of observations can be distinguished. The first one concerns the light-induced changes depending on the direction of the magnetization. Teal et al. [1] was first to observe a PME of this type, namely the change of the induced uniaxial anisotropy in Si-doped YIG under the influence of infrared radiation. A related effect which has been intensively studied is the photoinduced dichroism. The second class of observations concerns effects like light induced change of permeability or of absorption constant, which turns out to be independent of magnetization and to be irreversible at temperatures low enough to prevent thermal relaxation. This review deals with mainly the second class of effects which one of the authors (K. H) has studied since 1970 and then the others have become coraborated to study. In advance, almost all of the reports the authors have published prior to 1988 are listed here [14]. All the works on the second type of PME are included the ones on the thermally or dark induced anisotropy. The model of Fe^{2+} ions occupying magnetically inequivalent sites used to explain the thermally induced effects also applies to the PME. Consequently it is convenient to start this review with a discussion of thermally induced effects, after we touch upon the experimental procedure. To clarify the relation of the two classes, we follow an approach in which Enz et al. [3] have first described the underlying mechanism and then the experimental results on irreversible effects. The final discussion treats the properties of the active centers (usually called photomagnetic center) in connection with a simple model the authors proposed, which accounts for phenomenologically; in the process of writing this review, our new insights are included which are mentioned here for the first time.

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II. Fxperimental Procedures

2. 1. Sample preparation

2. 1. 1. Preparation of polycrystalline materials

Polycrystalline ferrites and garnets are usually formed by some types of solid state reaction [1, 2]. For example, the stoichiometric YIG may be written as $Y_3Fe_5O_{12}$, and the reaction forming this from the solid consitituent oxides is simply

$$3Y_2O_3 + 5Fe_2O_3 \longrightarrow 2Y_3Fe_5O_{12}$$
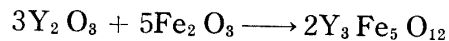
In order to produce such a reaction in a reasonable time, an intimate mixture of the two oxides must be raised to a high temperature, often of the order of 1000°C. For the measurements, the material will be required in solid form, and the substance produced by the reaction will then be powdered again, formed into the desired shape, in this case, troidal ring and again heated to the most high temperature of 1450°C [3] to form the solid by a sintering process. In their experience, the authors believe that both initial and firing conditions play an important part in order to obtain material in reproducible manner. Besides, the final chemical composition may not be unique. In particular, the valence state of the metal ions present and the amount of oxygen taken into the lattice may vary. Some ions are very ready to adopt one of several valencies and there may thus be inhomogeneity of the final product on this account. For example, iron may occur as Fe^{2+} [4, 5], Fe^{3+} and Fe^{4+} [6, 7, 8] in a solid. Moreover, slightly different conditions of preparation may result in the occupation of different crystal lattice sites by a given ion. These variations can affect the physical properties and may give rise to marked changes especially in PME. This might be why the effect is not always reproducible and the data are scattered by different researchers [9]. A sintered polycrystalline sample is not truly solid but contains many pores. The quantity, size, shape and distributions of both crystal grains and pores through the substance will vary with different forming techniques, and with firing temperature, atmosphere and time. Clearly, pore distribution and size will affect PME also. It is not the purpose to review the extensive literature, but merely to give an outline of the preparation techniques themselves. There are basically four steps in the preparation [1]:

(1) preparation of materials to form an intimate mixture with the metal ions in the ratio which they will have in the final product,

(2) heating of this mixture to form the ferrite or garnet (presintering),

(3) powdering of the prepared meterial and pressing or forming into the required shape and

(4) sintering to form the final shape.

The purpose of presintering [10] is

(1) to decompose oxides, thereby reducing the evolution of gas in the final sintering process,

(2) to assist in homogenizing the material,

(3) to reduce the effect of variations in the compositions of the raw materials,

(4) to reduce or control the shrinkage of the material which occurs otherwise during the final firing.

Fig. 2. 1 shows, diagrammatically, the typical flow charts in ferrite and garnet preparation. To produce a sample powder with sufficient reactivity for the sintering process the reacted powder has to be subjected to a second ball-milling treatment. This inevitably leads to considerable contamination by wear of the steel balls commonly used [11, 12]. To produce samples of high purity the authors therefore should prefer wet chemical methods [13, 14]. In Philips research group, powders were prepared by spray-drying of sulphate solutions [14]. In YIG

Fig. 2. 1 Flow chart of stages in polycrystalline ferrites and garnet preparation.

samples, the solutions of $FeSO_4$ were prepared from iron powder and sulphuric acid, $Y_2(SO_4)_3$ solutions from Y_2O_3 and sulphuric acid.

The spray-dried sulphate powder was reacted in an oxygen atmosphere at 1100-1200°C. The resulting garnet powder was directly pressed into pellets under a pressure of 1 kbar. The pellets were sintered for 4 to 6 hours at 1400-1450°C in oxygen.

The absence of a second phase is usually checked by microscopic examination of polished samples. Generally the impurity content of the sintered materials was obtained from a spectroscopic analysis. Recently, the authors adopted ICP (Inductive Coupled Plasma) method to obtain a precise quantity of tetravalent or divalent impurities [13]. Ferrous concentrations ware determined by titration with cerium sulphate [14].

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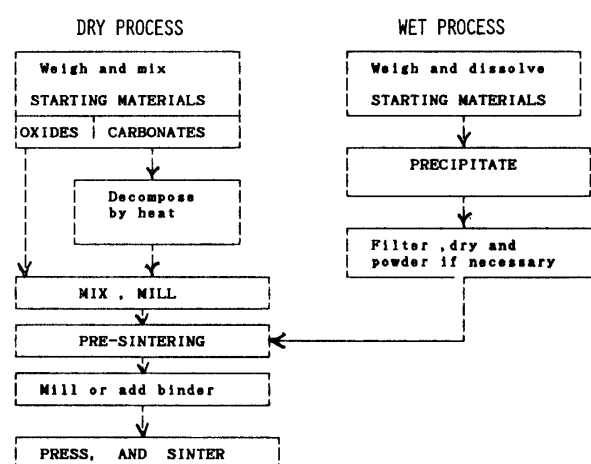14. J. G. M. Delau : Philips Res. Repts Suppl. 6 (1975) p. 41.

2. 1. 2. Preparation of single crystals

Naturally occurring ferrites and garnets include magnetite, magnesium ferrite, zinc ferrite and grossularite. These minerals may be found as single crystals of varying degrees of perfection and presumably occur through natural crystallization from a melt in some favorable condition. In order to produce single crystals of ferrites and other oxides artificially, attempts are not always successful. There are various important factors to consider, such as the temperature of the melt, the type and pressure of the atmosphere above it, the composition and fluidity of the melt and the length of time for crystallization [1]. A melt or solution of appropriate composition must be produced to cause a certain degree of supersaturation and then the solid is allowed to crystallize out slowly. Some of the procedures successfully employed are outlined in the following: a) the Verneuil or frame-fusion method [21,], b) the Bridgman method [1, 3, 4], c) the flux-melt method [1, 5, 6], d) the LPE method [1, 7, 8] and e) floating zone method [9, 10, 11].

a) In this method, very fine powder particles, $1\sim2\times10^{-6}$ in diameter, are dropped into an oxygen-hydrogen flame which maintains a temperature above the melting point of the feed meterial. This material forms molten droplets, devoid of nucleation centers, which fall upon a ceramic rod where they are accumulated and cooled. When a continuous supply of powder is fed into the flame, the solidified droplets build up on the ceramic rod to form a column or boule. If the correct conditions of flow, rate of solidification and so on are maintained, it is possible to build up the boule as a single crystal either by developing the first droplet as a single nucleation center or permitting solidification around a single crystal seed. The ceramic rod upon which the solidified material falls is usually surrounded an electric heater to prevent rapid cooling and the rod is withdrawn slowly as the boule increases in length. Many variations of this type of system have been employed. For an example, a boule of manganese ferrite 4 cm long and 1.5 cm mean diameter was grown in about three hours. However, crystals by this technique often contain strains, cracks and other imperfections. Even if the starting materials of the highest purity are used, then there may be faults due to the dissociation of the ferric oxide component with loss of oxygen at the higher temperatures encountered in the flame. According to the authors'view, PME may be expected in these samples as well as in FZ samples mentioned below.

b) The Bridgman method

This method was used for the first time for the preparation of synthetic magnetite [3, 4]. In this work the raw material was charged in a conical platinum crucible with platinum cap and was heated in an atmosphere of CO and CO_2 above the melting point of the charge, in order to destroy all nucleation centers and to remove all dissolved gases. The crucible with its contents were slowly lowered down the tube, i. e. down the temperature gradient. Solidification thus fiirst commenced at the tip of the cone, producing a very small nucleation center. Ohta [4] applied this method to growth of Mn-Zn ferrite succesfully. Crystals 1 cm in diameter and about 3 cm long were produced, of very high purity and perfection. To YIG, however, it may not be applied since the melting point of YIG is higher than that of platinum crucibles.

c) The flux-melt method

In this method a flux added to the feed materials before melting. Because of the incongruent melting and reduction problems at high temperatures, it was recognized early that molten salt crystallization provided an attractive method for YIG growth. It is essentially a solution-growth technique in which the solvent is a salt which melts at temperature appreciably lower than the ferrite or the garnet. Nielsen et al. [7] succeeded in forming specimens of magnetic garnets, i. e., YIG by this method. In a laboratory where one of authors (K.H) had studied, for example, the starting composition was 53 molar percent PbO, 44 molar percent Fe_2O_3 and 3 molar percent Y_2O_3. This charge was molten below 1350°C and consequently could be contained in platinum crucibles. Afterwards the authors tried it often in a modified way. Surveys of the available techniques have been given in ref. 1. As mentioned in the following, this contains a lot of flux materials and shows often a p-type characteristics [12, 13].

d) The LPE method

In the form of thin film, garnet single crystals find applications in magnetic bubble memory system and magnetooptic devices. For the most part, this layer is grown by liquid phase epitaxy on a garnet substrate [1]. Garnet films have been deposited by RF sputtering [14], hydrothermal growth [15], vapor phase epitaxy (VPE) [16, 17] and liquid phase epitaxy (LPE) [1]. Neither RF sputtering nor hydrothermal growth were developed sufficiently to defect-free films. VPE suffered from lower reproducibility and higher defect densities than LPE. Having not used these LPE layers for a measurement of PME, the authors may expect it successful [18]. LPE takes the two major variations of tipping and dipping. Tipping was first used to grow semiconductor layers for tunnel diodes [19]. The technique was adapted to magnetic garnets [20]; epitaxial YIG films on GGG ($Gd_3Ga_5O_{12}$) substrates. The equipment consists of a platinum boat capable of limited rotation about a horizontal axis through its center. The platinum boat is tilted such that the garnet-flux solution is at the lower end until the growth temperature is reached. It is then tipped such that the solution bathes the substrate at the other end. Growth is terminated by tipping back to the original position. On the other hand, dipping was first introduced by Linareset et. [21] also to grow magnetic films of YIG on GGG. A typical dipping apparatus consists of a crucible in a furnace with access from the top. The substrate is suspended in a vertical or horizontal plane above a garnet-flux solution until it reachss thermal equilibrium with the melt. It is then lowered into the melt for a certain period of time. Both dipping and tipping were both non-isothermal techniques in their original applications, which has detrimental effects on film reproducibility because of the temperature dependence of growth rate and distribution coefficients. Levinstein et al. [22] made a breakthrough when they noted that $PbO-B_2O_3$ fluxed garnet melts could be supercooled considerably with a high degree of stability against spontaneous necleation. They grew magnetic garnet films by dipping into a supersaturated solution under truly isothermal conditions. The stability of the supercooled melt makes dipping more convenient and easier to controle than tipping.

e) The floating zone method

Recently a single crystal of YIG has been produced with completely eliminated Si or other impurities successful in this method and found a PME in this sample. Fig. 2. 2 shows a flow chart of processing of YIG by this method. This furnace is schematically
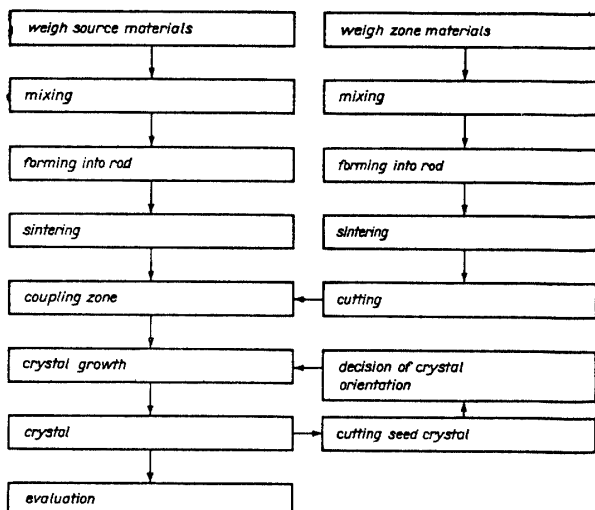
Fig. 2.2 Flow chart of production of YIG single crystal by floating zone method. [10]
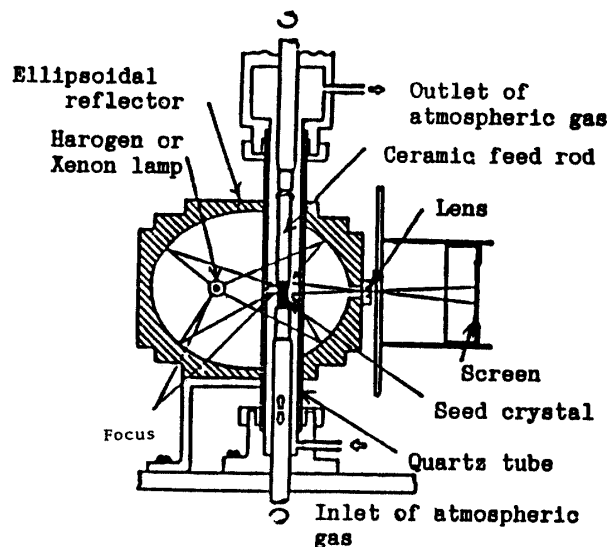

Fig. 2.3 Schematic representation of floating zone furnace. [10]

shown in fig. 2, 3. First, powders of Y_2O_3 and Fe_2O_3 were mixed mechanically in the desired ratio, formed like a bar and the sintered. Second, a raw rod of sintered YIG was set for the material feeding on the shaft in the so called FZ-furnace of the infrared radiation convergence type of which details have been reported elsewhere, while the seed of YIG crystal was holded on the other end. Between the rod and the seed, the sintered YIG was placed in order to provide the melt. When melting started, the raw rod and the seed were connected via liquid and they were counter-rotated. On the seed, the melted material was solidified as the focus of the radiation source passed the position. From the single crystal, several toroidals were cut in the crystalline plane (110), size of which was almost the same each other; 3.6 mm in the outer-diameter, 1.8 mm in the inner-diameter and 1.1 mm in the hight. The result of chemical analysis, using a high sensitive Xray microanalyzer and ICP, shows that Si or Ca impurity has not been detected within the accuracy of 0.01 molar percent in FZ-YIG (hereafter termed the sample grown by this method), while the Flux-YIG (the samle grown by the flux method) was found to contain the impurity of Pb (9 mol%) considerably. Before a measurement of the sample, an annealing at 700°C for 6 hours in pure oxygen was carried out in order to remove mechanical stresses.

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2. 2. Experimental arrangement

For the measurements cores were produced from the sintered pellets or single-crystalline plates, with dimensions 10 mm O. D., 3 mm I. D. and 0.3-1.0 mm height in the former case and several variations in the latter case. A few windings were put around the core for permeability measurements. The induced voltage was determined with a lock-in-amplifier, allowing measurements of μ' and μ'' in the frequency range 10 Hz-20 kHz as shown in fig. 2.4 schematicaly. Unless otherwise indicated, all permeability values given in this review measured at 10 kHz with a 10 mOe drive field. For the measurement of PME of permeability, the authors generally used the following procedure. After cooling in the dark by immersion of the sample in liquid nitrogen, the aample is a. c, demagnetized and the permeability value obtained about 1 min after demagnetization is defined as μ_d. Previously automatic demagnetion was carried out by Enz for a measurement of DA with the aid of a capacitor discharge in a resonant circuit consisting of the inductance L, a capacitance C and a ring sample [1]. One of the authors (K. H) followed Enz's experimental arrangements specially of automatic demagnetization. As far his experience was limited, no success was obtained to get reproducible result. To breakthrough this difficulty, he separated them independently and got a very good reproducible result. Based on the idea of multiplying output of oscillator and linearly changing discharge voltage with time, an automatic a. c. demagnetizer has been made in the laboratory, which may be controled in the points of intensity, duration time, time between demagnetization and frequency of demagnetizing current as shown in fig. 2. 4 [2, 3, 4]. Recently, Y. H. Lee has developed a new method for a measurement of disaccommodation (DA) [5] and in a near future will be applied to the purpose of a future measurement of PME. The sample is illuminated with the unfiltered light of an incandescent lamp, although in an early stage of measurements a set of filter was aided to check a sensitivity of wavelength. When a saturation value has been reached, the lamp is switched off, and the permeability value obtained in this way is denoted by μ_{irr}. Fig. 2.5 shows the arrangement for the measurement of PME and μ''.
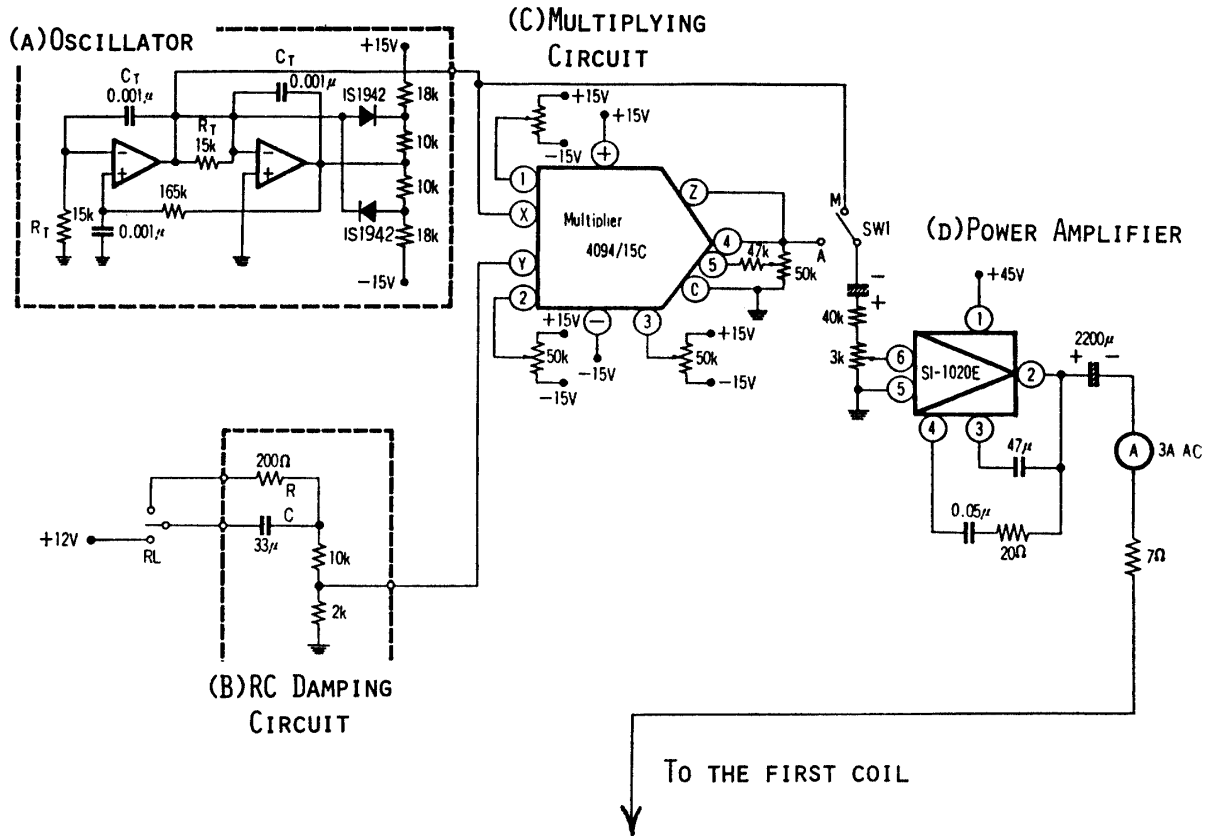
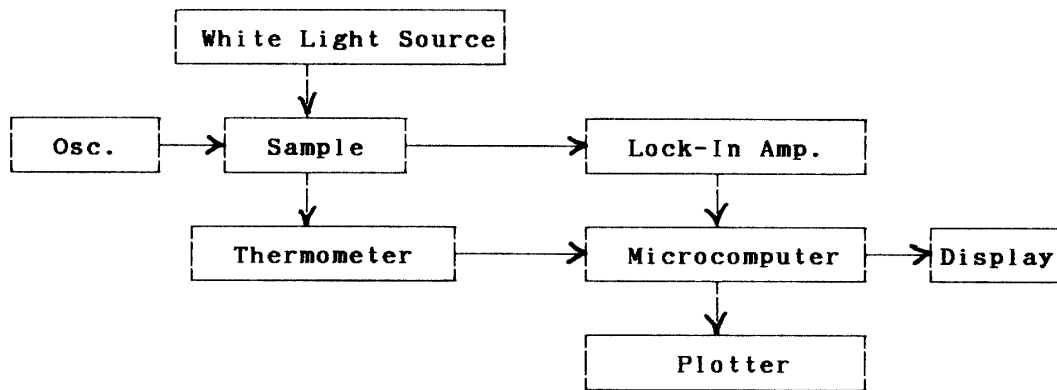Fig. 2.4 Demagnetizing circuit [4]

Fig. 2.5 Arrangement for the measurement of PME and μ".

References

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