

Review Paper

A Review on Multiferroic in Magnetic Field Sensor Applications

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ARTICLE INFORMATION	ABSTRACT
<p>Corresponding Author: Indu Sharma</p> <p>Article history: Received: 24-06-2022 Revised: 28-06-2022 Accepted: 02-07-2022 Published: 04-07-2022</p> <p>Key words: Multiferroic; magneto electric sensors; Synthesis method; Characterization</p>	<p>Multiferroic magnetoelectric (ME) materials with the capability of coupling magnetization and electric polarization have been providing diverse routes towards functional devices and thus attracting ever-increasing attention. The typical device applications include sensors, energy harvesters, magnetoelectric random access memories, tunable microwave devices and ME antennas etc. Among those application scenarios, ME sensors are specifically focused in this review article. We begin with an introduction of materials development and then recent advances in ME sensors are overviewed. Engineering applications of ME sensors are followed and typical scenarios are presented. Finally, several remaining challenges and future directions from the perspective of sensor designs and real applications are included.</p>

Introduction

Nanocrystalline materials are single- or multi-phase crystalline solids with a grain size of a less nanometers ($1 \text{ nm} = 10^{-9} = 10 \text{ \AA}$), generally less than 100 nm. Since the grain sizes are so small, a significant volume of the microstructure in nanocrystalline materials are composed of intersections, mainly grain boundaries, i.e. large fraction atom locates in grain boundaries. Consequently, nanocrystalline materials show properties that are significantly different from, and frequently improved over, them conventional coarse-grained polycrystalline counterparts [1]. Materials with microstructural features of nanometric dimensions are referred to in the literature as Nanocrystalline materials (a very generic term), nanocrystals, nanostructured materials, nanophase materials, nanometer-sized crystalline solids, or solids with nanometer microstructural features [2]. Nanostructured solids are perhaps the most systematic description, although nanocrystalline materials will be the applicable term if one is proceedings with solids with grains made up of crystals.

The study of nanocrystalline materials has attracted the thought of materials scientists, physicists, chemists, mechanical engineers, electrical engineers, and chemical engineers. This has shown to both in-depth and broad-brush research activities on different expression of nanocrystalline materials. A new journal entitled “Nanostructured Materials” was launched in 1992 and continued till 1999[2]. The nanocrystalline materials pioneered by Gleiter were preceded by studies of nanoparticles by researchers such as Uyeda [3]. Presently the many fields of nanostructured materials are (I) nanoparticles, (ii) nanocrystalline materials, and (iii) nanodevices. The characteristics applications for the many kinds of nanoscale materials include dispersions and coatings, high surface area materials, functional nanostructures (e.g., optoelectronic devices, biosensors, nanomachines) and bulk nanostructured materials for structural or magnetic applications. Nanocrystalline materials can be classified into different categories depending on the number of dimensions in which the material has nanometer modulations. Thus, they can be classified into (a) layered or lamellar structures, (b) filamentary structures, and (c) equiaxed nanostructured materials.

A layered or lamellar structure is a one-dimensional (1D) nanostructure in which the magnitudes of length and width are much greater than the thickness that is only a few nanometers in size [2]. The nanostructured materials may contain crystalline, quasicrystalline, or amorphous phases and can be metals, ceramics, polymers, or composites. If the grains are

made up of crystals, the material is called *nanocrystalline*. On the other hand, if they are made up of quasicrystal line or amorphous (glassy) phases, they are termed Nano quasicrystals and Nano glasses, respectively [4]. Gleiters has further classified the Nanostructured materials just as the composition, morphology, and distribution of the nanocrystalline component.

Now, the many techniques have been developed to prepare nanostructured materials from the vapor, liquid, or solid states. Nanostructured materials have been synthesized in recent years by methods including inert gas condensation, mechanical alloying, electrodeposition, physical vapor deposition, chemical vapor processing, co-precipitation, sol-gel processing, sliding wear, spark, laser ablation, hydrothermal pyrolysis, quenching the melt under high pressure, biological templating, nonchemical synthesis, and devitrification of amorphous phases [2]. In practice, any method able of forming very fine grain-sized materials can be used to synthesize nanocrystalline materials. The grain size, morphology, and thickness can be modified by suitably controlling the process variables in these methods. Both methods have advantages and disadvantages and one should choose the suitable method depending upon the conditions. If a phase transformation is involved, e.g., liquid to solid or vapor to solid, then stages must be taken to increase the nucleation rate and decrease the growth rate at some point in formation of the product phase. In fact, it is this strategy that is used during devitrification of metallic glasses to produce nanocrystalline materials [5]. The selecting of the method depends upon the capability to control the most important characteristic of the nanocrystalline materials, viz., the microstructural characteristics (grain size, layer spacing, etc.). Further aspects of significance are the chemical composition and surface chemistry or cleanliness of the interfaces. Extremely clean interfaces can be produced and retained during processing and subsequent consolidation by conducting the experiments under UHV conditions; but, this adds up to the cost of processing.

Multiferroic

The development in mainly fields of technique is possible thanks to the search and production of new materials and the investigation of their properties. The investigations of smart materials are particularly important for electronics, informatics, electroacoustic, optoelectronics etc. They can be grouped into several basic categories including piezoelectric, electrostrictive materials, magnetostrictive materials, shape memory alloys, optical fibers etc. Ferroics and multiferroics (biferroics) earn special attention between the smart materials. Ferroic crystals are crystals which involve at least one phase transition which change the directional symmetry of the crystal. In the science of crystals, study of changes of symmetry as a function of temperature, pressure, etc. is a very developed subject [14]. When a crystal changes its symmetry, it is a process of going from one phase to another one. It is called a phase transition entailing a change of symmetry [15]. The term ferroic materials (Ferroics) is a general term covering ferromagnetics, ferrimagnetics/antiferromagnetic, ferroelectrics / ferrielectrics / antiferroelectric and ferroelastics / ferrielastic / antiferroelastics [14]. These ferroics are called primary ferroics. Ferroic materials show a hysteresis.

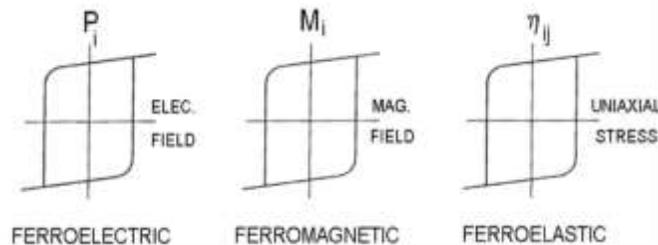


Fig 1: Typical hysteresis loops for ferroelectrics $P(E)$, ferromagnetics $M(H)$ and ferroelastics $\eta_{\parallel}(\sigma)$ [14].

There are number of common features among materials showing the properties of ferromagnetism, ferroelectricity and ferroelasticity, the most significant being the occurrence of a domain structure (importance of spontaneous breaking of symmetry), and the secondary hysteresis behavior. Therefore, created the general term *ferroics materials* (ferroics) for all such materials even if *ferro* has been used for creating the word *ferroic*, it has nothing to do with the presence or absence of iron in a ferroic material. Apart from the domain structure, a second important feature of ferroics is the high value of certain response functions of the material that can acquire in the vicinity of the ferroic phase transition. Ferroics has great importance in the family of smart materials. Between them are the smartest multiferroics and biferroics. This means that they have a spontaneous magnetization (M_s) which can be reoriented by an applied magnetic field (H), a spontaneous polarization (P_s) which can be reoriented by an applied electric field (E), and a spontaneous deformation (ϵ_s) which can be reoriented by an applied stress (σ) [14].

The term multiferroics has been created to describe the materials in which two or all three of ferromagnetism, ferroelectricity and ferroelasticity exist in the same phase. Biferroics are materials in which two ferroic states occur in the same phase [16]. Materials with simultaneous ferroelectric and magnetic ordering are called *ferroelectromagnetics*, those with simultaneous ferroelectric and those with mechanical ordering – *ferroelectroelastics*; when and with simultaneous

ferromagnetic and mechanical ordering occurs simultaneously they are called *ferromagnetoelastics*[17]. This is work concentrated on *ferroelectromagnetics multiferroics*, which are concurrently ferroelectrics and ferromagnetics (FE + FM →FEM).Multiferroic materials are a different category of solid-state compounds, in which minimum two order states such as magnetic, electric or piezo-elastic phases coexist. Therefore, illustrious materials such as ferromagnets showing strong piezo-magnetic properties, as well as piezo ferroelectrics are disputably a special class of magneto-elastic or electro-elastic coupled multiferroics.This complex coupling between electric polarization - electric field - strain, magnetization -magnetic field - strain and stress - strain - electric polarization - magnetization is graphically illustrated in figure 3[18].

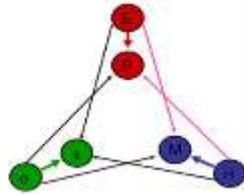


Fig 2: Possible cross-couplings in multiferroics. **E** – electric field; **P** –electric polarization; σ - applied mechanical stress; ϵ - strain; **H** –magnetic field; **M** – magnetization.

Whereas the coexistence of ferro-order and piezo-elastic properties can give rise to exciting piezo-electric and piezo-magnetic couplings, there are two additional multiferroic probable states, each showing cross couplings between the order states. These additional types of multiferroics are the subject of this review article and they are: i) magneto-electric multiferroic, in which the electric and magnetic order states coexist; ii) elasto-magnetolectric multiferroic, in which all three order states are present [18].It is notable to mention that, by electric and magnetic order state it is understood any type of ferroic ordering involving ferromagnetic, antiferromagnetic, ferri-magnetic, paramagnetic, super-paramagnetic and their electrical equivalents, if appropriate. However, what requires multiferroic materials scientifically and technologically desirable is not only their ability to display multiple order states but, most importantly, the cross-coupling results that can occur between the order states. Also, the piezo-ferro couplings, similarly interesting and valuable property of some multiferroics is the so-called “magneto-electric” coupling, which arises in materials where magnetic - electric or magnetic-electric-elastic phases coexist. The magneto-electric effect simplifies the conversion between energies stored in magnetic and electric fields. Hence, magneto-electric multiferroic materials have the unique property that when subjected to an applied external magnetic field, the electric polarization is modified and, conversely, the application of an external electric field alters the net magnetization of the multiferroic solid [18]. The magneto-electric effect in multiferroics can therefore be electrically or magnetically induced and it is mathematically described by the magneto-electric coupling coefficient, [19-23].The magneto-electric coupling are of two types: direct and indirect.

The direct magneto-electric coupling indicates the multiferroics in which the direct application of electric or magnetic field results in changes of magnetization or electric polarization of the system, respectively. This effect is characteristic to a special class of multiferroic materials called “single-phase” multiferroics [24]. A second mechanism of activating the magneto-electric effect occurs primarily in elasto- magneto- electric multiferroics, also known as composite multiferroics [25-26]. This is called the indirect magneto-electric effect. In the indirect magneto-electric effect, the application of magnetic or electric field does not involve directly the electric polarization or magnetization, respectively. Instead, they are modified indirectly via a strain mediated magneto-electric coupling [27].

Yttrium manganese oxide

Yttrium is a chemical element with the symbol *Y* and atomic number 39. It is a transition metal chemically like the lanthanides and has been classified as a rare-earth element. Nanocrystalline multiferroic oxides have accepted remarkable research interest in the historical decade due to the coexistence of magnetically and ferroelectrically well-ordered states. The magnetoelectric phenomena originate from the coupling between the pairing of magnetic properties by application of an electric field and vice versa. The study of multiferroic materials has become an interesting subject because of their possible applications in electric devices such as electric field induced magnetic memory effects and electro-optic transducers controlled by magnetic fields [6-8]. They have also been used in the fields of radio, television, satellite communications, sensors, memory devices and digital recording media.Among them, $YMnO_3$ has shown a wide range of applications in capacitors, transducers, actuators, nonvolatile information storages, spintronic devices and magnetoelectric sensors. Several chemical routes viz. reverse micelles, solvothermal, sonochemical and polymeric citrate precursor methods are available in literature for the fabrication of various nanostructures [9].Among them, Pechini type polymeric precursor method is highly efficient to prepare homogeneous nanostructures without careful control of gel processing time [9]. Nanocrystalline $YMnO_3$ has been prepared by hydrothermal and sol–Gel methods. However, there is no report in literature for the synthesis of $YMnO_3$ nanoparticles using polymeric citrate precursor method. However, there is no report in literature for the synthesis of $YMnO_3$ nanoparticles using polymeric citrate precursor method. The crystal structure of nanoscale hexagonal $YMnO_3$ deviates from the bulk and the ferroelectric displacement of Y^{3+} cations decays progressively with the decreasing size as well as different

magnetic interactions with different grain sizes were also reported [10]. Such different properties have not been studied to large extent and the origin of these properties of $YMnO_3$ is still uncertain. The synthesis of Nano-sized $YMnO_3$ at low temperature is found to be difficult, because the maximum meta stable phases are mostly stabilized at high temperature and pressure. The effect of Nano size on magnetic properties like curie temperature of $MnFe_2O_4$ system as compared to bulk was due to finite size scaling [9]. A projected description for the irregular behavior of $MnFe_2O_4$ prepared formulated by wet and standard ceramic methods was due to non-equilibrium cation size distribution over tetrahedral and octahedral sub lattices. This procedure is not being described for $YMnO_3$. The $YMnO_3$ nanoparticles are prepared by metal organic precursor method using citric acid and ethylene glycol as complexing agents for the first time. As nanoparticles are prepared, then examined by means of X-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM), UV-visible, putrescence and BET surface area studies. The electrical and magnetic properties have been studied in detail for the multiferroic characterization.

Crystal structure of $YMnO_3$

Materials presenting magneto-electric coupling exhibit magnetic properties coupled to the electric properties, such as polarization or dielectric constant [11]. These materials have attracted a lot of attention over the last years then the magneto-electric coupling concedes a possible control of the magnetic properties by an electric field and over electric properties using a magnetic field. Unfortunately, the microscopic origin of the coupling between the magnetic and electric order parameters is still ill known. The knowledge of phonons spectra can however bring help on understanding this coupling. Indeed, not only the phonons modes are strongly related to the existence and amplitude of a spontaneous polarization, but in addition strong spin-phonons coupling occur in multiferroic material. This coupling can even be strong enough in order to result in hybrid excitations built from the mixing between phonons and spin-waves[11]. Further, newly such excitations were also found by inelastic neutrons scattering in the hexagonal manganite $YMnO_3$.

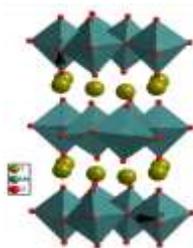


Fig 3: Representation of the $YMnO_3$ crystal structure.

Hexagonal $YMnO_3$ is a layered compound where the manganese ions are in triangle-based bipyramids. These bipyramids are arranged in planes parallel to the direction so that the manganese ions form a distorted triangular lattice (see in figure 1) and they share an oxygen atom in the (a,b) planes. The yttrium atom is localized in between the bipyramids layers. $YMnO_3$ is paraelectric at high temperature with space group $P6_3/mmc$ [12-13]. Below the critical temperature, it is ferroelectric, with space group $P6_3cm$ and the polarization is aligned along the c direction. The temperature of this paraelectric (PE) to ferroelectric (FE) phase transition is however still under dispute. Indeed, usually assumed to be around ≈ 920 K, it was recently proposed to occur at a higher temperature, $\approx 1050 - 1100$ K [11]. A very recent neutrons diffraction investigation suggests the existence of an iso-symmetric phase transition at ≈ 920 K, resulting in a strong lowering of the polarization amplitude, while the non-centrosymmetric to centrosymmetric transition occurs at 1258 ± 4 K only [11]. At much lower temperature ($T_N \approx 75$ K) $YMnO_3$ undergoes a paramagnetic (PM) to antiferromagnetic (AFM) transition. The ferroelectric and antiferromagnetic order parameters are coupled and a giant atomic motion was revealed at the Neel temperature [11].

Literature Review

Metallurgists and materials scientists have been conducting research investigations for several centuries to develop materials which are 'stronger, stiffer, and lighter' than the existing materials and capable of use at elevated ('hotter') temperatures [28]. The technological industries in the developed countries have given an added spur to these works. Several novel and non-equilibrium processing methods have been developed during the past few decades to improve the performance of the existing materials; these include rapid solidification from the liquid state, mechanical alloying, plasma processing, vapour deposition, etc [28].

A central underlying theme in all these methods is to energize the material to bring it into a highly non-equilibrium (metastable) state (also including a possible change of state from the solid to liquid or gas) through melting, evaporation, irradiation, application of pressure, storing of mechanical energy, etc. [29]. The material is then taken to another lower-energy metastable state by quenching or connected to this processes when it can exist as a supersaturated solid solution, metastable crystalline or quasicrystal line phase, or even in a glassy state, providing abundant opportunities to modulate the crystal structures or micro structures. These processes have proceeded to large development in the properties of many alloy

systems and therefore some industrial applications; these have been explained and fully documented in the references. The different way of converting a material to a metastable state is to reduce its grain size to very small values of a few nanometers ($1 \text{ nm} = 10^{-9} \text{ m}$) when the proportion of atoms in the grain boundaries is equivalent to or higher than those inside the grains. This type of metastability can be classified as morphological meta (or 'in') stability in the scheme of Turnbull (1981) [28]. The small grain sizes materials are known as nanocrystalline materials (and as nanocrystals, nanostructures, nanophase materials, or nanometer-sized crystalline solids). They have been shown to have properties massively greater to those shown by conventional grain-sized ($> 10 \mu\text{m}$) polycrystalline materials. It is a combination of unique compositions and new microstructures that leads to the extraordinary potential of the nanocrystalline materials.

There has been a continued improvement in the research studies in recent years on the synthesis/processing, characterization, and applications of these novel materials. A new journal entitled 'Nanostructured Materials', published by Pergamos Press, was started in 1992 [28]. In addition to many national conferences and as a part of other seminars, a series of international conferences is organized entirely to discuss the developments in nanostructured materials. The first international conference was held in Cancun, Mexico, in September 1992 and the second is listed to be held in Stuttgart, Germany, in October 1994. The idea of the present article is to present a very general overview of the structure and properties of nanocrystalline materials. Potential applications of these new materials will also be underlined. This field has been reviewed prior with highlighting on some certain topics [28]. However, there is no inclusive review related to the materials facets; but this review fulfills that gap. The current article reviews the repute of research and development on the structure and properties of nanocrystalline materials. Nanocrystalline materials are the polycrystalline materials with grain sizes less than 100 nm. Because of the very small sizes, a large fraction of the atoms in these materials is situated at the grain boundaries, and this consults special aspects. Nanocrystalline materials can be prepared by inert gas-condensation, mechanical alloying, plasma deposition, and many other methods. These have been briefly reviewed.

Multiferroics: Historical and Present Developments

It is believed that Schmid first introduced the term “multiferroic” in 1994 [30]. Although the term multiferroic is rather recent, multiferroic materials and the possibility of magnetoelectric coupling in solids was first predicted by Curie in 1894 based on crystal symmetry considerations [31], while the term magneto-electric coupling was first coined by Debye in 1926 [32]. Pioneering work on magneto-electric effect in multiferroic materials goes back to more than half a century [30]. In 1959, Dzyaloshinskii was the first to report a magneto-electric effect in anti-ferromagnetic Cr_2O_3 dielectrics [33], followed by similar experimental and theoretical studies published by Astrov, Folen, Stader and Rado [34-36]. O'Dell summarized most of the theoretical and experimental achievements in the field of magneto-electric media up to 1970 in an article published in 1962 [37] and a book published in 1970 [38]. Following these exiting developments, the interest in magneto-electric multiferroics grew substantially because of the realization of their potential for technological applications [30]. This culminated with the publication by Wood and Austin in 1973 of the only article reviewing possible applications of multiferroics at that time [39], followed by a recent short review published by Scott in 2012 and briefly covering applications published in the 2009 - 2012 timeframe [40]. These topographies complete multiferroics nothing more than an interesting subject for academic research. Since most useful applications contain room temperature operation and large magneto-electric coupling. Moreover, this was complemented by a limited understanding of the microscopic nature of multiferroicity and magneto-electric coupling, which led unsurprisingly to a decline in multiferroics research activities for approximately 20 years. For example, per the Web of Science (WoS), since 1970 until 2000 there have been typically, fewer than 30 articles per year published on this topic [41]. Since 2000 however, the number of publications on multiferroic and magnetoelectric materials grew year on year and today there are thousands of articles published per year [18].

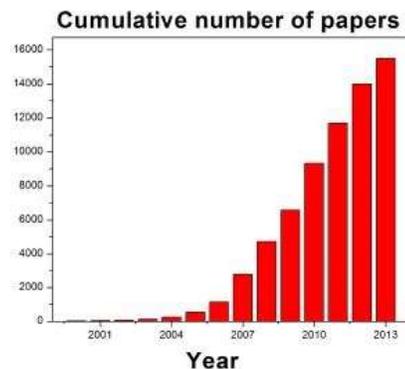


Fig 4: Cumulative number of published articles searched using the keyword “multiferroic” in Google Scholar. The data covers the period 2000 to 2013 and excludes patents [18].

This is best accentuated by data in figure 4, showing the cumulative number of articles published in the period 2000 to 2013. The figures are extracted from Google Scholar using the keyword “multiferroic”. This method of measuring the worldwide interest in a each research topic is much more correct than *WoS* because it totals all written contributions Not only peer review scientific articles. It is believed that the recent renewal of research in the magneto-electric effect and multiferroics is due to the industrial needs for novel multifunctional materials and devices for advanced applications. Latest advances in the fabrication and discovery of new materials and thin films, including the ability to deposit nano and sub nano-scale thin film hetero-structures, also supported this revival of interest in multiferroic materials. A simple search using the keyword “multiferroic” in the Google Patent search engine yielded around 2000 results.

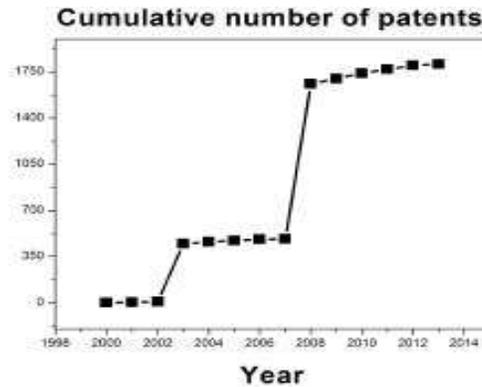


Fig 5. Cumulative number of patents by filing date searched using the keyword “multiferroic” in Google Patents. The data covers the period 2000 to 2013[18].

The search refers to patent filing date (not granted) over the period 2000 – 2013 [18]. Multiferroic patent search data is summarized as a cumulative plot in figure 3. Interestingly, according to figure 5, there are two major jumps in which most multiferroic patents were submitted, followed by relatively slow development periods. The first jump occurred in 2002 - 2003 and the second jump was in 2007 - 2008. Since 2008 the number of patents filed every year continued to grow, although at a slower pace. Today multiferroic materials are recognized as a true emergent field of research and technologies. The fascinating properties of multiferroic materials make them ideal candidates for a new wave of technological applications with potential to produce billions of US dollars in revenue.

ynthesis and Methods

Increased activity on the synthesis of nanocrystalline materials in recent years dates back to the pioneering investigations of Herbert Gleiter of the University of Saarlandes in Germany in 1981 [42]. He synthesized ultrafine nanometer-sized metallic particles using an inert gas condensation technique and strengthened them in situ into small disks under ultra-high vacuum conditions.

Starting Phase	Techniques
Vapour	<ul style="list-style-type: none"> • Inert gas condensation • Sputtering • Plasma processing • Vapour deposition (physical and chemical)
Liquid	<ul style="list-style-type: none"> • Electrodeposition • Rapid solidification
Solid	<ul style="list-style-type: none"> • Mechanical alloying/milling • Sliding wear • Spark erosion

Table 1. Methods to synthesize nanocrystalline materials [42].

Generally synthesis of nanomaterial’s can be classified two types, 1. Bottom-up approach 2. Top-down approach. In bottom-up approaches include the miniaturization of materials components (up to atomic level) with further self-assembly process leading to the formation of nanostructures [43]. In self-assembly the physical forces working at Nano scale are used to combine units into larger stable structures. Ex: quantum dot formation during of nanoparticles from colloidal dispersion. In top-down approaches use larger initial structures, which can be externally controlled in the processing of nanostructures. Ex: ball milling and plastic deformation [44].

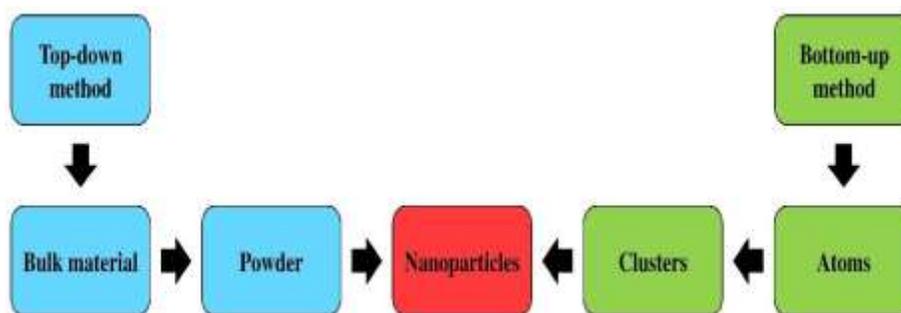


Fig 6: A simplified representation of the Top-Down and Bottom-Up process is presented.

Bottom-Up Method

Bottom-up or constructive method is the build-up of material from atom to clusters to nanoparticles. Sol-gel, spinning, chemical vapour deposition (CVD) is the most commonly used bottom-up methods for nanoparticle manufacture.

Sol-gel

The sol – a colloidal solution of solids suspended in a liquid phase. The gel – a solid macromolecule immersed in a solvent. Sol-gel is the most selected bottom-up method due to its modesty and as most of the nanoparticles can be synthesized from this method. Sol gel is a wet-chemical process containing a chemical solution working as a precursor for an integrated system of separate particles. Metal oxides and chlorides are the typically used precursors in sol-gel process [47]. The precursor is then dissolved in a host liquid either by shaking, stirring or sonication and the resulting system has a liquid and a solid phase. A phase separation is carried out to recover the nanoparticles by various methods such as sedimentation, filtration and centrifugation and the moisture is further removed by drying [48].

Spinning

The synthesis of nanoparticles by spinning is held by a spinning disc reactor (SDR). It has a rotating disc inside a chamber/reactor where the physical parameters such as temperature can be controlled. The reactor is generally filled with nitrogen or other inert gases to remove oxygen inside and avoid chemical reactions [45]. The disc has is rotated at different speed where the liquid i.e. precursor and water is filled in. The spinning causes the atoms or molecules to fuse together and is precipitated, collected and dried [49]. The several functioning parameters such as the liquid flow rate, disc rotation speed, liquid/precursor ratio, location of feed, disc surface, etc. concludes the characteristics nanoparticles synthesized from spinning disc reactor.

Chemical Vapour Deposition (CVD)

Chemical vapour deposition is the deposition of a thin film of gaseous reactants onto a substrate. The deposition is carried out in a reaction chamber at ambient temperature by combining gas molecules. A chemical reaction occurs when a heated substrate comes in contact with the combined gas [46]. This reaction produces a thin film of product on the substrate surface that is recovered and used. Substrate temperature is the influencing factor in CVD. The advantages of CVD are highly pure, uniform, hard and strong nanoparticles. The disadvantages of CVD are the requirement of special equipment and the gaseous by-products are highly toxic [50].

Top-Down Method

Top-down or destructive method is the decrease of a bulk material to nanometric scale particles. Mechanical milling, laser ablation, sputtering and thermal decomposition are some of the maximum commonly used nanoparticle synthesis methods.

Mechanical Milling

Including the various top-down methods, mechanical milling is the very expansively used to produce various nanoparticles. The mechanical milling is used for milling and post annealing of nanoparticles during synthesis where different elements are milled in an inert atmosphere [51]. The influencing factors in mechanical milling is plastic deformation that leads to particle shape, fracture leads to decrease in particle size and cold-welding leads to increase in particle size [52].

Laser Ablation

Laser Ablation Synthesis in Solution is a common method for nanoparticle production from various solvents. The irradiation of a metal submerged in a liquid solution by a laser beam. As Laser ablation synthesis in solution provides a stable synthesis of nanoparticles in organic solvents and water that does not require any stabilizing agent or chemicals it is a 'green' process [52].

Sputtering

Sputtering is the deposition of nanoparticles on a surface by ejecting particles from it by colliding with ions [53]. Sputtering is usually a deposition of thin layer of nanoparticles followed by annealing. The thickness of the layer, temperature and duration of annealing, substrate type, etc. determines the shape and size of the nanoparticles [54].

Thermal Decomposition

Thermal decomposition is an endothermic chemical decomposition produced by heat that breaks the chemical bonds in the compound [55]. The specific temperature at which an element chemically decomposes is the decomposition temperature. The nanoparticles are produced by decomposing the metal at specific temperatures undergoing a chemical reaction producing secondary products. Table 1 lists some of the nanoparticles synthesized from these methods.

Characterization Technique

Nanoparticles are generally characterized by their size, morphology and surface charge, using such advanced microscopic techniques as scanning electron microscopy (SEM), transmission electron microscopy (TEM) and atomic force microscopy (AFM). The average particle diameter, their size distribution and charge affect the physical stability and the in vivo distribution of the nanoparticles. Electron microscopy techniques are very useful in ascertaining the overall shape of polymeric nanoparticles, which may determine their toxicity. The surface charge of the nanoparticles affects the physical stability and responsibility of the polymer dispersion as well as their in vivo performance.

Scanning Electron Microscopy

Scanning electron microscopy (SEM) is giving morphological examination with direct visualization. The techniques based on electron microscopy offer several advantages in morphological and sizing analysis. However, they provide limited information about the size distribution and true population average. For SEM characterization, nanoparticles solution should be first converted into a dry powder, which is then mounted on a sample holder followed by coating with a conductive metal, such as gold, using a sputter coater. The sample is then scanned with a focused fine beam of electrons [56]. The surface characteristics of the sample are obtained from the secondary electrons emitted from the sample surface. The nanoparticles must be able to withstand vacuum, and the electron beam can damage the polymer. The mean size obtained by SEM is comparable with results obtained by dynamic light scattering. Moreover, these techniques are time consuming, costly and frequently need complementary information about sizing distribution [57].

Transmission Electron Microscope

TEM operates on different principle than SEM, yet it often brings same type of data. The sample preparation for TEM is complex and time consuming because of its requirement to be ultra-thin for the electron transmittance. The nanoparticles dispersion is deposited onto support grids or films. To make nanoparticles withstand the instrument vacuum and facilitate handling, they are fixed using either a negative staining material, such as phosphotungstic acid or derivatives, uranyl acetate, etc., or by plastic embedding. Alternate method is to expose the sample to liquid nitrogen temperatures after embedding in vitreous ice. The surface characteristics of the sample are obtained when a beam of electrons is transmitted through an ultra-thin sample, interacting with the sample as it passes through [58].

Atomic Force Microscopy

Atomic force microscopy (AFM) offers ultra-high resolution in particle size measurement and is based on a physical scanning of samples at sub-micron level using a probe tip of atomic scale [59]. Instrument provides a topographical map of sample based on forces between the tip and the sample surface. Samples are usually scanned in contact or noncontact mode depending on their properties. In contact mode, the topographical map is generated by tapping the probe on to the surface across the sample and probe hovers over the conducting surface in non-contact mode. The prime advantage of AFM is its ability to image non-conducting samples without any specific treatment, thus allowing imaging of delicate biological and polymeric nano and microstructures [60]. AFM provides the most accurate description of size and size distribution and requires no mathematical treatment. Moreover, particle size obtained by AFM technique provides real picture which helps understand the effect of various biological conditions [61].

X-Ray Diffraction

X-ray diffraction (XRD) patterns of the pure *YMO* powders sintered at many temperatures.

Powders annealed 900 °C displays a poor crystalline behavior as temperature is not enough to complete the reaction. By further increase in the sintering temperature (1000 °C), crystallization of *YMO* phase was found to be introduced and well crystallized *YMO* powders have been found by annealing at a temperature of 1100 °C with reside time of 6 h. The diffraction peaks for the *YMO* samples annealed at 1100 °C can be indexed on the basis of a hexagonal unit cell of space group *P63cm* and no extra impurity or intermediary phases have been detected.

Applications

ac / dc magnetic field sensors

Ferromagnetic-ferroelectric composites have attracted interests in recent years for studies on the nature of magneto-electric interactions (ME) and for use as sensors, memory devices, and for signal processing [62]. The coupling between the two subsystems is mediated by mechanical strain. An applied AC field H produces a magnetostrictive strain in the ferromagnetic layer, leading to a voltage response V in the ferroelectric layer. The ME voltage coefficient (MEVC) = $V/(t \cdot H)$ is a measure of the strength of ME coupling, where t is the thickness of the ferroelectric layer. Multiferroic composites studied so far include ferrites, manganite, or transition metals/alloys for the ferromagnetic phase and barium titanate, poled lead zirconate titanate (PZT), or PMN-PT for the ferroelectric phase [63]. A giant low-frequency DME effect was observed in several layered composites [62]. A related ME phenomenon of fundamental interest's is the coupling at bending resonance or electromechanical resonance (EMR) modes in the composite [63]. When the AC field is tuned to these modes, MEVC increases by orders of magnitude. A new generation of magnetic field sensors based on layered composites of ferromagnetic and ferroelectric/piezoelectric phases has been reported in recent years [62]. In composites of poled lead zirconate titanate (and magnetization (M) graded ferromagnetic layer consisting of Ni (with $4 \mu\text{M} = 6 \text{ kG}$) and Metglas ($4 \mu\text{M} = 21 \text{ kG}$) a strong ME coupling was measured at zero-bias ($H_b = 0$) [62]. The coupling is due to the interaction of out-of-plane internal magnetic field arising from grading in M and the AC magnetic field H . Magnetic sensors are devices that can sense, without physical contact, the formation or deviation in magnetic fields of the surrounding environment and from these changes they derive information on the properties of the magnetic field itself, as well as indirect properties of other individuals, such as: direction, presence, rotation, angle, speed, electrical currents, temperature, and so on. The output signal of magnetic sensors involves some signal processing for translation into the desired parameter. Magnetic field sensors are widely applied in a range of applications with huge effect on our society. Examples of such applications are: magnetic field sensors for scientific measurements and metrologies, microscopy imaging, magnetic field mapping of Earth's field, other cosmological entities and geo-positioning, detection of metals and weapons, security systems, magnetic recording readers for magnetic data storage, biomedical applications, medical imaging, positioning and speed measurement, automotive applications, to name a few. According to a new market research report (www.marketsandmarkets.com), the demand for magnetic field sensors in the year 2010 was 3.67 billion units with a total market cost of 1.1 billion US dollars. The magnetic field sensors market is expected to reach 7.14 billion units in 2016, with a total value of 2.0 billion US dollars. The main driver of growth in the magnetic sensors market is the increased demand from the automotive, industrial and consumer electronics markets. The magnetic field sensors market is expected to reach 7.14 billion units in 2016, with a total value of 2.0 billion US dollars. The main driver of growth in the magnetic sensors market is the increased demand from the automotive, industrial and consumer electronics markets. In the case of automotive industry, magnetic sensors are utilized in at least 10 applications [http://memblog.wordpress.com/2010/04/05/isupplisilicon- magnetic-sensors head-for-big-time/]. The most common types of magnetic field sensors are summarized in Table 2, together with their physical operating principle magneto-electric voltage coupling coefficient (α_H^V), which gives a relationship between the voltage induced on the electrodes of a multiferroic device and the amplitude of an externally applied. Multiferroic materials and devices can be used to fabricate magnetic field sensors by utilizing the magnetically induced magneto-electric effect. This is mathematically described by the magnetic field – i.e. the field to be sensed. Integrating gives:

$$V = \alpha_H^V H \cdot t$$

where t is the thickness of the ferroelectric layer in the case of laminates, or the thickness of the sample in the case of a single-phase composite, H is the amplitude of the magnetic field under measurement and V is the voltage response of the multiferroic sensor. Relation indicates that the voltage response of a multiferroic sensor varies linearly with the amplitude of the applied magnetic field. A linear response is a critical requirement in order to operate as a sensor. However, the measured field H in this case is in fact an ac magnetic field.

Table 2: Different types of magnetic field sensors listed by their operating principle and best estimates of their approximate detection limits [64].

Magnetic Field Sensor	Operating Principle	Special Requirements	Magnetic Field Detection Limit
Inductive coil	Faraday effect	ac magnetic fields only	$\sim 10^{-4} \text{ T}$
Flux gate	Magnetic induction	Large size	$\sim 10^{-8}$
Magneto – optic	Kerr/ Faraday polarization rotation	Optical alignment	$\sim 10^{-12}$
SQUID	Quantum interference/ Josephson effect	Cryogenic cooling	$\sim 10^{-15}$
Hall sensor	Hall effect/ Lorentz force		$\sim 10^{-5}$
AMR	Anisotropic magnetoresistance	dc biasing	$\sim 10^{-3}$
GMR	Spin dependent interface Scattering	–	$\sim 10^{-3}$
TMR	Spin dependent quantum	–	$\sim 10^{-3}$

	Tunneling		
CMR	Double exchange and semiconductor-to-metallic transition	Highly temperature dependence	$\sim 10^{-2}$
Rubidium	Spin-exchange relaxation	Cryogenic cooling dc biasing	$\sim 10^{-14}$
Multiferroic	Magneto-electric coupling	dc biasing	$\sim 10^{-12}$

Almost all magnetic field sensing applications based on multiferroics are related to the detection of ac signals [64]. Ac detection using multiferroics requires the application of a dc magnetic field bias. The dc bias field couples with the ac magnetic field to produce a pseudo-piezomagnetic linear response, which in turn modulates the electrical response of the multiferroic sensor. Relation is applicable to single-phase as well as composite multiferroics. However, as seen in relation, the voltage output also depends on the strength of the magneto-electric coupling coefficient. Since single-phase multiferroics show very small magneto-electric coupling effects and usually require cryogenic temperatures, most of the magnetic sensor applications are based on composite multiferroic structures, in which large strain mediated magneto-electric effects occur at room temperature. These are usually bi-layer or tri-layer composite multiferroic structures with various geometries. Obviously, for magnetic sensing applications, the strain mediated magnetoelectric coupling is magnetically induced and intimately related to the magneto-strictive properties of the magnetic phase.

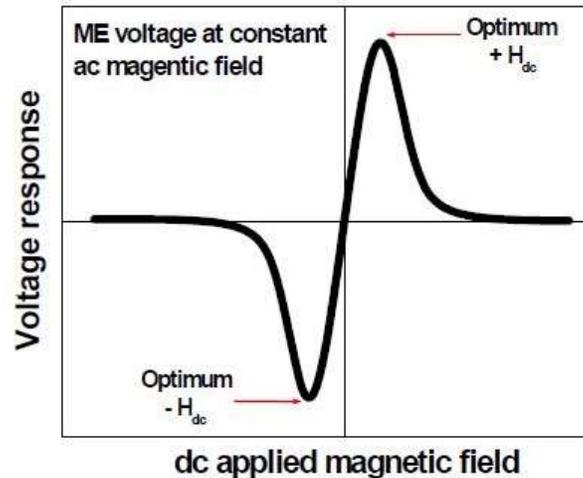


Fig 6: Typical response of a composite multiferroic to a dc applied magnetic field at constant ac magnetic field amplitude and frequency [64].

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Conclusion

Therefore, not surprisingly most applications of multiferroic composites are related to detection of ac magnetic fields. Experimental and theoretical results indicate promising qualities of multiferroic sensors with magnetic field detection sensitivities varying from $10^{-13} < H_{ac} < 10^{-3}$ T [64], especially when driven at electromechanical resonance frequency

[79,82,83]. For more details on multiferroic composite sensors, the following excellent review articles are strongly recommended to the reader [64]. It is important to mention that the angle between the dc bias magnetic field vector and an excitation field is critical for the operation of the multiferroic sensor. Studies of the magnetoelectric coupling coefficient as a function of this angle have indicated that the maximum coupling effect is achieved when the fields are parallel to each other, while the effect is zero for the transversal case [65]. In fact, coupling measured at intermediate angles between 0 and 180 shows almost a linear variation of the sensor's response [64]. This indicates the possibility of utilizing multiferroic sensors for direction detection / compass applications. Indeed, Lage et al. have recently reported the realization of a vector field magnetometer based on two orthogonal self-biased multiferroic composite sensors that can be used for the detection of 2D magnetic field vectors and, in principle, it can be expanded to 3D magnetic field vector mapping [64]. In terms of dc magnetic field detection, the linearity condition in dc-magnetic field must be fulfilled. Examining figure 6, there is a range of dc-magnetic fields where the response is indeed linear. This will depend on the geometry and constituent materials of the sensor, but essentially would allow detection of small dc magnetic fields with sensitivity limit of about, 10^{-7} T as already reported in [66]. Other mechanism in which a linear dc magnetic field response is possible, is when a composite multiferroic displays linear behavior in $M(H)$ curves (i.e. no hysteresis loop) or a linear response in strain – field curves. Such conditions can be fulfilled by a multiferroic containing a paramagnetic phase or a ferromagnetic phase with strong perpendicular magnetic anisotropy. The concept of multiferroic composite magnetic field sensor for dual detection of dc / ac magnetic fields has been indeed demonstrated experimentally [67]. In this paper, it has been shown experimentally that a linear magneto-electric response can be achieved for both ac and dc applied magnetic fields by using multiferroic $SrAl_xFe_{12-x}O_{19}$ (hexaferrite) / $PbZrTiO_3$ (PZT) laminates with strong perpendicular magnetic anisotropy. These kinds of structures can therefore operate in dual mode as both ac and dc sensors, with linear dc response over the $-2 kOe < H_{dc} < +2 kOe$ field range.

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