Chapter 1

Introduction

1.1 General Introduction

With the advent of digital and information technology, there has been a rise in the number of data processing and data storing devices that are being used to handle an influx of data. For this very purpose, electrons are explored with profoundness for their inherent properties. In a traditional semiconductor device, only the charge of the electron is taken advantage of for the processing of information. In terms of storing the information, magnetic recordings like magnetic tapes, hard disks, etc are used that are made up of magnetic materials. This is possible only by exploring the spin property of electron. However, a large amount of energy and space were required to accomplish these processes. As a result, investigations were carried out to develop a device that incorporated the features of both spin and charge of an electron. This gave rise to a new field of study known as Spintronics. With the discovery of giant magnetoresistance (GMR) effects in 1988 by Alfert Fert and Peter Grünberg, it proved to be a boon for spintronic devices [1, 2].

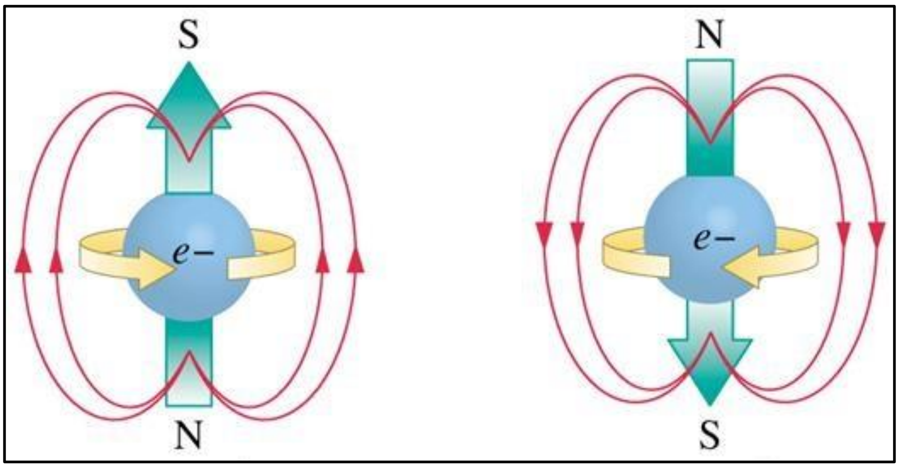

Figure 1.1: *Representation of how electron behaves as a tiny magnet.*

The advantage of using spintronic devices is that it was able to retain the information of the spin state for some time. In addition to this, only low amount of energy is required to alter the spin orientation of the electrons. However, it was challenging to combine both the properties onto a single device and this in turn led to the discovery of dilute magnetic semiconductors (DMS). It possesses ferromagnetic as well as semiconducting properties which results in an interaction between the electrons of semiconductor and the magnetic ions that tend to occupy a vacancy. DMS systems are much more preferable because of their ability to reach high values of Curie temperature desired for practical applications like storage, quantum computation, etc [3].

1.2 Semiconductors

A semiconductor is characterized by its distinct electrical conductivity behaviour, which is intermediate between that of a metal and that of an insulator [4]. In contrast to metals, they are found to have negative temperature coefficient of resistance. The conductivity of a semiconductor can be altered by the process of doping wherein impurities are introduced into the crystal structure. An intrinsic semiconductor is that which is pure and has no doping carried out into it. Electrical conductivity is the result of excitation of electrons from valence band to the conduction band. On increasing the temperature, the electrons transit from its position in valence band making its way to the conduction band, creating an electron deficiency known as hole. In case of an intrinsic semiconductor, number of electrons in conduction band is equal to number of holes in the valence band. In an extrinsic semiconductor, the number of electrons or holes can be increased by doping with impurity atoms. There can be excess electrons in the system if the impurities have more valence electron per atom than the semiconductor atom. As a result, conduction in such a semiconductor is the result of presence of excess electrons also known as donor electrons (n-type). Since these electrons are weakly bound to the impurity atom, they can easily make their way into the conduction band and with a rise in temperature, the number of conduction electrons increases thereby increasing the conductivity. On the other hand, excess holes can be produced in the system by the impurities that consist of fewer valence electron per atom compared to the semiconductor atom. Conduction in this case is prompted by the holes (p-type) that result from incomplete bond between the impurity and semiconductor atom due to lack of electrons. Since they are able to accept electrons in order to complete the bond, their energy level is close to the valence band so that electrons can easily hop from the valence band leaving behind a hole that leads the conduction process. Such impurities are also known as acceptor impurities. Since doping, exposure to electric field and light can bring about a modification in the electrical properties of a semiconductor, this makes them capable of being used in devices that deal with switching and amplification purposes.

The most commonly known semiconductors are silicon and germanium that belong to group IV of the periodic table. Silicon-based devices are found to be much more effective than germanium-based devices as the latter can operate even at high temperatures and are also abundantly available. They are found to have applications in power electronics industry, in components like diodes, transistors, photoelectric cells, MOSFET, etc. Other examples of elemental semiconductors include tin (Sn) also a group IV element, selenium (Se) and tellurium (Te) belonging to group VI of the periodic table. However, with growing demand for semiconducting devices, came the need for improving the performance and other properties that could contribute to a wide range of applications. This motivated the development of compound semiconductors. Compound semiconductors are those that are made up of two or more elements. It is possible to grow them with various lattice constants and band gap energies. Commonly known compound semiconductors contain elements that belong to group III and group V as well as group II and group VI of the periodic table. Some of their examples are gallium arsenide (GaAs), gallium nitride (GaN), indium antimonide (InAs), indium phosphide (InP), etc of III-V group and cadmium telluride (CdTe), zinc selenide (ZnSe), zinc sulphide (ZnS), cadmium sulphide (CdS), etc of II-VI group.

Another class of compound semiconductors are those that consist of elements belonging to group IV and group VI. These classes of semiconductors are categorized as having narrow band gap which makes them suitable as infrared detectors and photovoltaic devices. Germanium telluride (GeTe), lead selenide (PbSe), tin selenide (SnSe), tin telluride (SnTe), lead sulphide (PbS), etc are some of the examples of this group. These semiconductors either have cubic or orthorhombic crystal structure. Lead chalcogenides have found widespread applications due to low cost of production. Presently tin chalcogenides are being explored for similar applications due to their non-toxic nature [5, 6]. Some of these compound semiconductors also exhibit ferroelectric, superconducting and spin-glass nature despite having a basic crystal structure. DMS based on IV-VI group semiconductors display carrier concentration induced ferromagnetic transition, light induced magnetization and high field magnetization [7-9].

1.3 Dilute Magnetic Semiconductors

Even before dilute magnetic semiconductors were first developed, intrinsic magnetic semiconductors in which there is a co-existence of semiconducting and magnetic properties were studied for spintronic device applications [10]. In the late 1960’s to early 1970’s, there were intensive studies on Eu^{2+} doped oxides and spinel-type composites ($ZnCr_2Se_4$) [11, 12]. However, there has been constraints in terms of its fabrication, unsatisfactory transport property as well as low range of Curie temperature T_C (below 50 K) thereby deeming it not beneficial for device applications. With the discovery of EuO as a ferromagnetic semiconductor that could reach T_C of 69 K, it accelerated the research on magnetic semiconductors [13]. Research on magnetic semiconductors took on a new perspective after the discovery of carrier-mediated ferromagnetism in bulk PbSnMnTe [14]. In a bid to improve the value of T_C to go beyond room temperature, dilute magnetic semiconductors were developed. They comprise of a semiconducting host material which is nonmagnetic in nature that is doped with small amount of magnetic ions through transition metals or rare earth metals. Figure 1.2 is a pictorial representation of a non-magnetic semiconductor and a dilute magnetic semiconductor. It is anticipated that the carriers in the host lattice will interact with the atomic spin on these magnetic dopants to produce a ferromagnetic order in the material. In contrast to metals, semiconductors enable the customization of their properties like band gap and carrier concentration to suit the application. Additionally, magnetism in the material can be tuned electrically if it results from carrier concentration. The host material could be compound semiconductors (II-VI group, III-V group, etc.), chalcogenides or even oxide semiconductors. Investigation on II-VI group based DMS system like $Cd_{1-x}Mn_xTe$, $Cd_{1-x}Mn_xSe$, etc showed that large solubility of Mn atoms was possible as the *d* states of Mn atoms were found not to perturb the tetrahedral sp^3 bonds present in the semiconducting system [15-17]. It was also found that the magnetic property of Mn doped II-VI DMS systems were either paramagnetic or spin-glass as the exchange interaction between Mn ions is antiferromagnetic in nature [18]. Despite that, these materials were found to have excellent optical properties finding their application in luminescence devices [19].

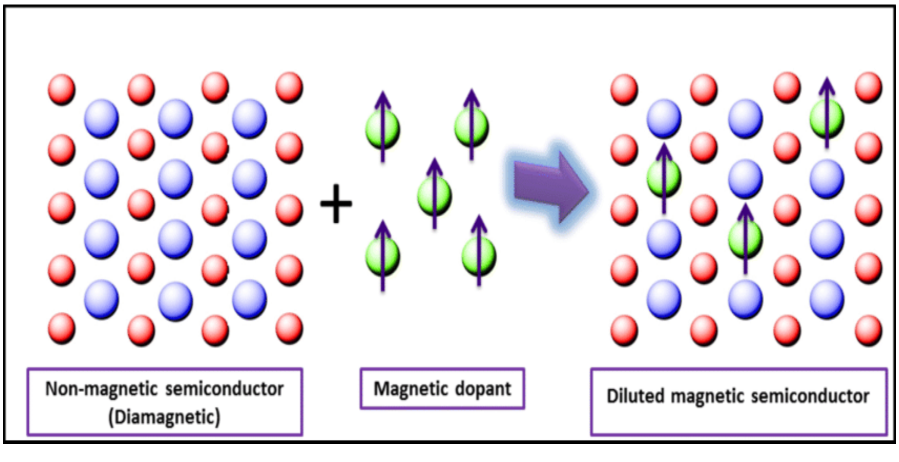

Figure 1.2: *Representation of how dilute magnetic semiconductors are formed.*

III-V semiconductors were found to be substantially less stable than II-VI semiconductors and have a much lower solubility of magnetic ions and thus not many experiments were conducted on them. However, with the development of new techniques, III-V DMS material InMnAs was successfully prepared by Munekata *et al*. using molecular beam epitaxy method and reported ferromagnetism [20]. Using the same technique, Ohno *et al*. prepared manganese doped gallium arsenide (Ga, Mn)As and the curie temperature T_C observed was 110 K [21]. Later, in 2000, it was predicted theoretically by Dietl *et al*. that Mn doped GaN and ZnO could exhibit T_C above room temperature and the magnetic property have been linked with p-type charge carrier concentration [22, 23]. With experimental observation of T_C above room temperature in Co doped TiO_2 as well as in (Ga, Mn)N, it motivated researchers to actively search for new DMS materials which will have a high value of Curie temperature in order to sustain ferromagnetic property even at room temperature [24]. The identification of hole-mediated ferromagnetism in (Ga, Mn)As and the resulting heterostructures opened up a myriad of opportunities for combining magnetic and spin-based phenomena with the widely used microelectronics and optoelectronics [25].

1.4 Magnetic properties of material

The phenomenon of magnetism is what causes a material to exert either an attractive force or repulsive force on one another. Moving electrically charged particles are the fundamental source of magnetic force. The orbital and spin motion of electrons give rise to orbital and spin magnetic moments which in turn provides for the magnetic behaviour in a material. A distinctive behaviour on the application of an external magnetic field is observed in those materials that are magnetic in nature. To determine the type of magnetism, one must have a thorough understanding of magnetic field v/s magnetization (M-H) and magnetic susceptibility χ curves. With regards to the alignment of magnetic moments in the presence of an external field, magnetic materials can be classified into five types: i) Diamagnetic

ii) Paramagnetic

iii) Ferromagnetic

iv) Antiferromagnetic

v) Ferrimagnetic

Figure 1.3 (a) is a schematic diagram that represents above-mentioned magnetic materials whereas (b) represents susceptibility curves for each of them as a function of temperature.

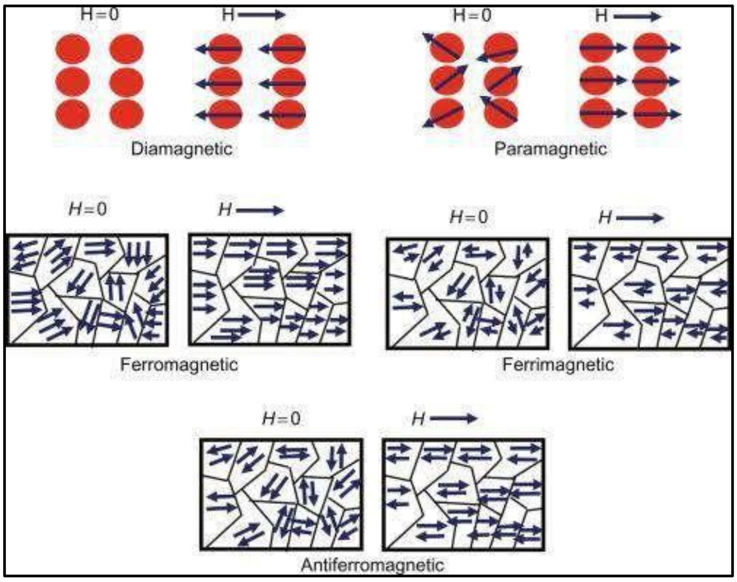

Figure 1.3(a): *Schematic representation of the different types of magnetic materials.*

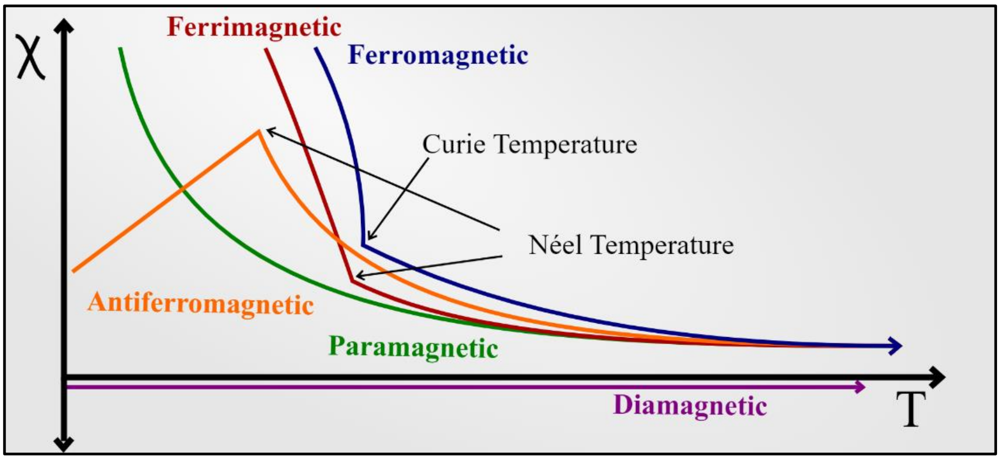

Figure 1.3(b): *Susceptibility curves of magnetic materials with respect to temperature.*

1.4.1 Diamagnetism

This form of magnetism exists only when an external field is applied and is weak in nature. The applied external field brings about a change in the orbital motion of electrons thereby inducing a weak magnetization whose direction is opposite to that of the external field. This gives rise to a negative value of magnetic susceptibility which is in the order of -10^{-6} to -10^{-5}. Diamagnetic property exists in all materials, however this form of magnetism has no importance from the practical point of view.

1.4.2 Paramagnetism

Paramagnetic materials are those that exhibit magnetic susceptibility in the presence of an external magnetic field having a small positive value. In materials with unpaired electrons, each atom has a net magnetic moment caused by the spin of the electrons. However, the orientations of these magnetic moments are random when there is no magnetic field which results in a zero net magnetization. On the application of magnetic field, these dipoles tend to align parallel to the field thereby ensuing in a net positive magnetization. In order to align all of the dipoles, very strong magnetic fields are required as the dipoles lack interaction among themselves. This effect is not permanent as on the removal of the field, the dipoles lose their alignment and at higher values of temperature, the susceptibility undergoes a decrease with an increase in temperature which is given by the equation

$$\chi = \frac{C}{T} \tag{1.1}$$

This equation is known as Curie's law. With increase in temperature, there is randomization of dipoles due to thermal agitation thereby reducing the paramagnetic effect.

1.4.3 Ferromagnetism

When the magnetic moments of paramagnetic ions are bound together to point in the same direction, it gives rise to ferromagnetism in the material. Ferromagnetic materials are those that have large number of unpaired electrons due to energy levels that are left unfilled in the atom. There are strong interactions between the atomic moments in these materials. The application of external field results in a parallel alignment of the atomic moments and because the interaction between the dipoles is quite strong, the material still possesses magnetic moment even when the magnetic field is removed. This is known as spontaneous magnetization (M_S). Rather than magnetizing themselves entirely in the same direction, they divide into small regions having a fixed magnetization also known as domains. In a particular domain, all the spins are aligned in the same direction. The susceptibility of ferromagnetic materials is given by the relation $\chi = \frac{C}{T-T_C}$, in which a ferromagnetic material on crossing Curie temperature T_C starts behaving as a paramagnetic material. Ferromagnetic materials also possess hysteresis behaviour. The maximum value of magnetic moment that gets induced due to applied field is known as saturation magnetization beyond which there is no further increase in magnetization values.

1.4.4 Antiferromagnetism

Similar to paramagnetism is antiferromagnetism (AFM) where the susceptibility is weak as well as positive. In this case, the magnetic moments associated with neighbouring dipoles align in an anti-parallel manner in the absence of an external field. As a result, the net magnetic moment is zero and hence the small values of susceptibility. At a certain critical temperature called Neel temperature (T_N), the susceptibility achieved by AFM materials is maximum. Above this temperature, the material losses its AFM property and gains paramagnetic behaviour.

1.4.5 Ferrimagnetism

Ferrimagnetic property is similar to antiferromagnetism with the fact that the ordering of spins is anti-parallel. But, on the contrary, the net magnetic moment in ferrimagnetic materials is not zero as the spins do not completely cancel out each other. There is a presence of spontaneous magnetization similar to what is observed in ferromagnetic materials. Thus, except for the magnetic ordering, there is a similarity in the properties of both ferrimagnetic and ferromagnetic materials. Another feature of ferrimagnetic material is that below Neel temperature, they assume ferromagnetic property and above which they exhibit paramagnetism.

1.5 Origin of magnetism in DMS

There are different theoretical models and experimental works mentioned in the literature that gives an idea of how magnetism originates in a DMS. The basis of these models depend on exchange interaction between the spins of the electrons and attempts were made to understand if these interactions lead to ferromagnetism, antiferromagnetism or spin-glass like magnetic system in the dilute magnetic semiconductors.

1.5.1 Direct exchange

In magnetic materials, direct exchange refers to an interaction that takes place between the electrons of atoms that are neighbouring with one another. For such an interaction to take place, no mediatory agents are required. Through the overlapping of magnetic orbitals, magnetic ions are directly coupled in this process [26]. When two magnetic atoms are close to each other, the exchange can be negative and the electrons would be aligned anti-parallel giving rise to an antiferromagnetic interaction. If the two atoms are at a little distance from each other, in that case the exchange becomes positive, and the electrons would have parallel alignment showing ferromagnetism. Direct exchange interactions are only valid within a short-range.

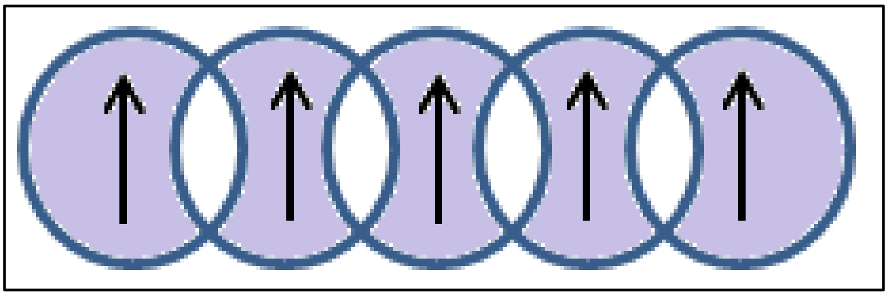

Figure 1.4: *Diagrammatic representation of direct exchange interaction between magnetic ions.*

1.5.2 Indirect exchange

Indirect exchange interaction refers to an interaction that occurs when the atoms of magnetic material interact through the use of intermediaries. They are long range in nature and in metals, which do not have any nearby electrons overlapping each other, these exchange interactions predominate. In general, indirect exchange mechanisms are existent in dilute magnetic semiconductors [27]. The intermediaries decide the different models of indirect exchange interactions that can be classified as RKKY interaction, Super exchange interaction and Double exchange interaction and are discussed below.

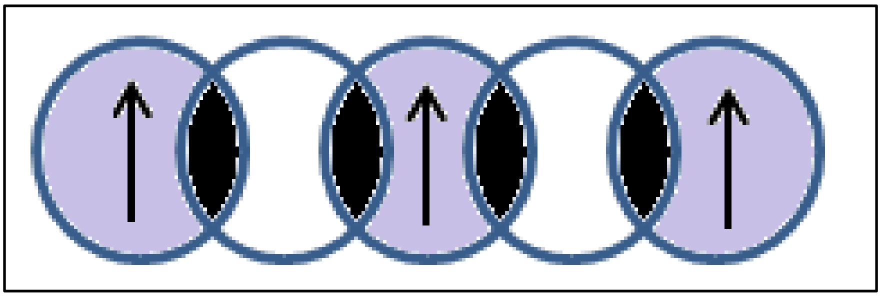

Figure 1.5: *Diagrammatic representation of indirect exchange interaction between magnetic ions.*

1.5.2.1 RKKY interaction

The Rudderman-Kittel-Kasuya-Yoshida (RKKY) interaction being a long range interaction, is one of the models that evaluates indirect exchange interaction between the magnetic ions via band electrons [28-30]. This interaction takes place in a degenerate DMS due to Coulomb exchange that is explained by the s-d Kondo Hamiltonian. The conduction electrons surrounding the magnetic ion are polarized by it. With increasing distance from the magnetic ion, the degree of polarization diminishes in an oscillatory manner. The resulting interaction due to such an indirect exchange will either be ferromagnetic or antiferromagnetic depending on how far the ions are located from each other.

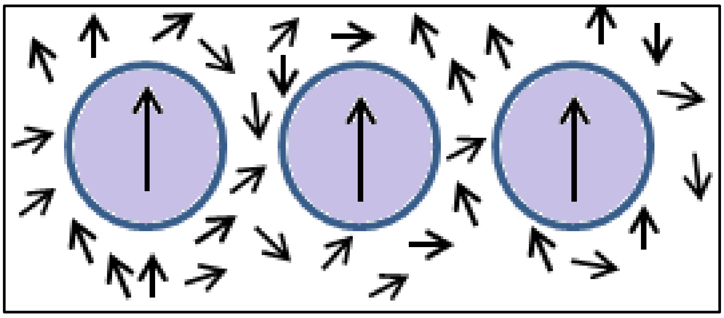

Figure 1.6: *Diagrammatic representation of RKKY exchange interaction where coupling is mediated by band electrons.*

1.5.2.2 Super exchange interaction

Here, a non-magnetic ion (usually oxygen ligand) is positioned between the magnetic ions that are not next to each other, to act as a mediator in this indirect exchange interaction. The p orbital of oxygen mediates the hopping of magnetic ions. Such an exchange interaction can explain antiferromagnetism in transition metal elements as hopping can take place between the d orbitals of transition metal cations through the p orbital of large oxygen anions. The way oxygen interacts with the two-transition metal ion, super exchange mechanism is further divided into 180° and 90° super exchanges based on geometry. The 180° super exchange interaction has both the d orbital of transition metal coupled to the same p orbital whereas d orbital of transition metal tends to overlap the orthogonal p orbital in case of 90° super exchange without any hopping mechanism between p orbital [31]. The 90° and 180° interaction are ferromagnetic and antiferromagnetic in nature respectively. Hoping between p-orbitals is plausible when the angle of the TM-O-TM is more than 90° and less than 180°. The sign of exchange interaction can be deduced from the bond angle between metal-oxygen-metal and the configuration of d-electron on the transition element. The semi-empirical Goodenough-Kanamori-Anderson rules provide an overview of these dependencies. [32].

1.5.2.3 Double exchange interaction

In order to explain ferromagnetism in perovskite samples where coupling between magnetic ions have two different ionic states at the same crystallographic sites, double exchange interaction was introduced [33]. The coupling is the result of virtual hopping of an extra electron from either ion. As in case of Mn^{2+}-Mn^{3+} system, d-electrons belonging to both these ions are able to hop through the mediation of p-orbitals of the adjacent anions. However, due to electrons being localized at the magnetic ion prevent them from being a part of charge transport, this mechanism cannot clearly help in the understanding of long-range order in DMS. In case of parallel orientation of spins of neighbouring magnetic ion, it allows the extra electron in Mn^{3+} delocalize to Mn^{4+}. This process of hybridization would result in the d band getting widened and thereby resulting in the reduction of band energy. If the spin states are antiparallel, the electron stays localized with Mn^{3+} and is unable to pair with Mn^{4+}.

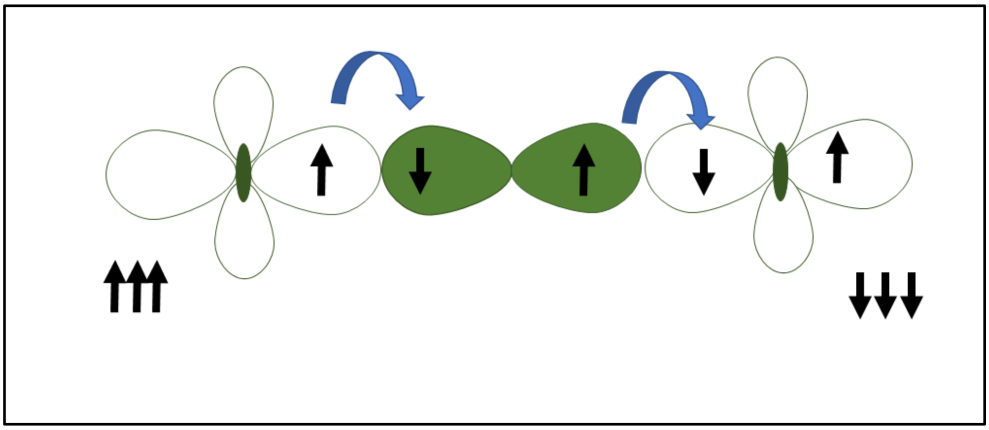

Figure 1.7: *Diagrammatic representation of superexchange interaction.*

1.5.3 Bound Magnetic Polaron Model (BMP)

BMP model was initially proposed in order to comprehend how dc conductivity of Europium chalcogenides are affected by temperature and magnetic field [34, 35]. When the carrier spin interacts with the localized spins of the magnetic ions in DMS materials, magnetic polarons are created. According to this model, the localized spins of transition metal ions interact via a substantially smaller number of weakly localized carriers to create bound magnetic polarons. With the overlap of neighbouring polarons comes ferromagnetic order having a long range and as the size of polaron becomes comparable to that of sample size, there is a ferromagnetic transition that takes place. In case of n-type dilute magnetic oxides, as suggested by Coey *et al.* presence of oxygen vacancies help in trapping the shallow donor electrons which helps in mediating ferromagnetism in the material [36]. Thereafter, their hydrogenic orbitals are where bound magnetic polarons are formed. Defects play an important role in this model as an increase in the number of defects increases the number of overlapping BMPs. Oxygen defects can bring in stability of the ferromagnetic state of the system. Further, a few Bohr radii separation between two BMPs can bring about ferromagnetic exchange between them [37]. The existence of an interaction that is not strong between the neighbouring polarons will give rise to an insulating or paramagnetic state.

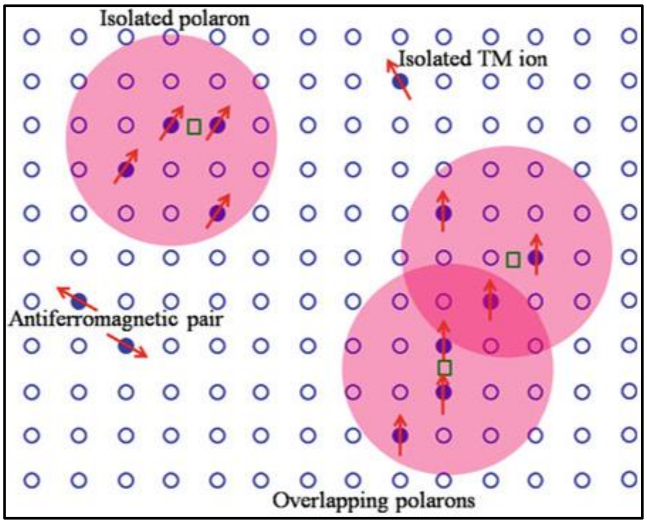

Figure 1.8: *Diagrammatic representation of formation of BMP.*

1.6 Motivation and Objective of the present work

The research work on dilute magnetic semiconductors is carried out with the intent of achieving high Curie temperature and to be able to bring about ferromagnetic properties in non-magnetic semiconductors. In order to achieve ferromagnetic ordering above room temperature, there is a need to have a cognizance of how magnetic interactions work between the magnetic ions that are doped and the semiconducting host material. A thorough investigation also requires adopting various characterization techniques and there is a constant effort to make use of sophisticated instruments that would help in better probing. On reviewing the literature, most of the studies were focused on developing II-VI, III-V group semiconductors as well as oxide-based semiconductors to function as dilute magnetic semiconductor. On the other hand, IV-VI group semiconductors are widely studied for their thermoelectric and optical properties due to their narrow band gap whereas magnetic studies are quite limited.

The main motivation behind the present work is to find out whether IV-VI group semiconductor, with suitable doping of magnetic ions, can exhibit ferromagnetism at room temperature. In that respect, Tin Telluride (SnTe) and Tin Selenide (SnSe) are chosen as host semiconductors for the present work. Additionally, a work that predicted room temperature ferromagnetism in elemental tellurium doped with silicon gave a push to also explore possibilities of developing Te as a potential DMS candidate [38]. In the past, there were many studies on germanium-based alloy systems that looked into their magnetic properties [39-41]. The doping in all three materials is carried out using transition metal Iron (Fe) in a fixed concentration. Alongside doping of magnetic ions, a group V element Antimony (Sb) is also substituted into these materials in varying concentrations thereby introducing charge carriers into the system. Antimony increases electron concentration when substituted in SnTe and SnSe whereas it increases hole concentration in Te. Despite the materials being narrow band gap semiconductors, appropriate dopant and its concentration in the material could present ferromagnetic features as established by past studies on IV-VI group semiconductors like $Ge_{0.92}Mn_{0.08}Te$, $Sn_{1-x}Cr_xTe$ alloys, $Ge_{1-x}Cr_xTe$ films, etc [42-44]. We can also investigate the potential of the materials for practical applications if we have an understanding of their structural, electrical and magnetic properties with various doping concentrations. The objectives of the present work can be summarized as follows:

1. Synthesis of $Fe_{0.05}(Te)_{1-x}Sb_x$ (x = 0, 0.01, 0.03 and 0.05) and characterizing the samples for their structural, optical, electrical and magnetic properties.
2. Synthesis of $Fe_{0.05}(SnSe)_{1-x}Sb_x$ (x = 0, 0.03 and 0.05) and characterizing the samples for their structural, optical, electrical and magnetic properties.
3. Synthesis of $Fe_{0.05}(SnTe)_{1-x}Sb_x$ (x = 0, 0.01, 0.03 and 0.05) and characterizing the samples for their structural, optical, electrical and magnetic properties.
4. To keep the concentration of Fe fixed at 0.05 in order to avoid formation of Fe clusters or any magnetic compounds of Fe.
5. To check for substitutional effect of non-magnetic impurity Sb that introduces charge carriers into the system on the magnetic ordering of the samples.
6. To address the origin of magnetic ordering in the system on the basis of any of the above-mentioned exchange interactions.
7. To check whether a particular sample can remember its memory information through magnetic-memory measurement.

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