T.1 Functional Magnetic Materials: Synergy Between Basic Science And Evolving Technology

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Magnetic materials are used widely in electric motors, loudspeakers, transformers, automobiles, magnetic resonance devices, magnetic memory storage and a diverse range of scientific instruments from large particle-accelerators to tiny multimeters. The continuous evolution in the field of magnetic materials which are important (but somewhat hidden) component of modern technology goes almost unnoticed. Often fresh classes of magnetic materials are discovered with new interesting functionality, which stimulates the growth of newer technology. The research activity pertaining to certain classes of magnetic materials being investigated in the Low Temperature Physics Laboratory (LTPL) at CAT provides an illustration of such emerging science and technology (S&T) involving newer classes of functional magnetic materials.

Magnetic materials with phase transition -driven functionality:

Three classes of magnetic materials have emerged during last two decades with much promise for immediate technological applications. These are (1) giant magnetoresistive and colossal magnetoresistive materials (2) magnetocaloric materials and (3) magnetic shape memory alloys. A brief description of these functional materials is given below.

Magnetoresistance, the change in electrical resistance with an applied magnetic field, is a useful tool in several areas of technology. For example, the computer hard drives use magnetoresistance to read the stored data. Most laptop computers now come fitted with high capacity hard drives which use giant magnetoresistive (GMR) sensors as read head. The basic GMR device consists of a three-layer sandwich of a magnetic metal such as cobalt or iron with a nonmagnetic metal filling such as silver or platinum [1]. It is interesting to note that the research in such artificially engineered magnetic multi-layers actually started in order to shed light on the fundamental question—how magnetic moments interact in magnetic materials? This is a reasonably difficult question in natural solids, which is still fascinating the researchers. The discovery of interesting GMR properties in these artificially engineered materials was rather unanticipated. It came as a bonus out of such curiosity driven basic research and the unforeseen technological development from then on was quite fast. From the first report of the GMR properties in 1988 it took less than ten years for the first product in the form of "read heads" for computer hard disk drives to have major economic impact. This interesting S&T development naturally spurred more research activity in magnetoresistive materials, and in early 1990s a class of rare-earth manganese oxide materials (commonly termed as manganites) were found with colossal magnetoresistive (CMR) properties [2]. Manganites show exotic physical properties in the form of metal-insulator transition and varieties of magnetic, charge and orbital ordering dictated by strong electron-electron interaction and electron-lattice interaction, and provide a challenging area of research. While the phenomenology of the CMR effect in manganites can be explained within the framework of certain microscopic double exchange interaction, this mechanism alone is insufficient to explain the observed effects quantitatively [2]. A prospective picture in this regard is the formation of a percolation path involving the metallic ferromagnetic (FM) and insulating antiferromagnetic (AFM) phases across a FM-AFM transition region, which can be manipulated by an applied magnetic field. This magnetic transition region in manganites has been a subject of many studies in recent times using various experimental techniques including microscopic imaging with electron and magnetic force microscopy. Distinct phase-coexistence in micrometer scale has been reported leading to the actual visualisation of a percolating path [2]. In addition the lattice distortions and long-range strains are known to be important for manganites and the intrinsic complexity of system with strong coupling between electronic and elastic degrees of freedom introduces further interesting features in the phase-coexistence [3].

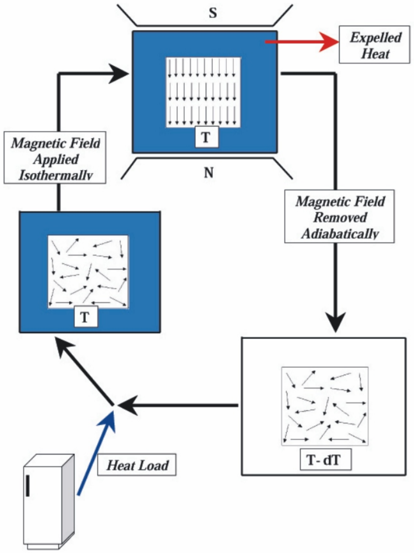

Fig. T.1.1 *Schematic representation of a magnetic-refrigeration cycle*

Originally measured in iron, the magnetic field induced temperature variation in a magnetic solid is known as 'magnetocaloric effect' (MCE). Instead of a working fluid undergoing a liquid-vapour transition in conventional refrigerator, a magnetic refrigerator can be envisioned using a magnetic solid, which heats up when, magnetized and cools down when demagnetized. Such magnetic cooling has a potential to reduce global energy consumption and minimize the need of ozone depleting and greenhouse chemicals. Fig. T.1.1 presents the schematic of a magnetic-refrigeration cycle. Initially randomly oriented magnetic moments in a suitable MCE material are aligned by a magnetic field, resulting in the reduction of magnetic entropy. In turn the material is heated via the increase of its lattice entropy. This heat is removed from the material to its surroundings by a heat-transfer medium. On removing the magnetic field, the magnetic moments become randomised causing an increase in the magnetic entropy. This leads to cooling of the MCE material below the ambient temperature. Using a heat-transfer medium, heat from the system to be cooled can then be extracted. The prospect of magnetic cooling as a viable alternative to vapour-compression technology has increased enormously since the recent discovery of giant MCE in various classes of magnetic materials [4]. The origin of this giant MCE is now traced to an interesting magneto-structural transition [4].

Shape memory alloys (SMA) are metals that have the ability to remember a predetermined shape, and to return back to that shape after being bent, stretched or otherwise mechanically deformed [5]. This shape-memory effect is caused by a 'thermoelastic martensitic transition' — a reversible transition between two different crystal microstructures in the concerned metallic system. SMAs have a wide range of technological applications including those for aeronautics, robotics and biomedical implants. Although the first recorded observation of the shape memory effect dates back to early 1930's, it was not until 1962, when this effect was discovered in equiatomic nickel-titanium (NiTi) alloys, that research into both the science and potential practical uses began in earnest [5]. Within 10 years, a number of commercial products were on the market, and understanding of the effect was much advanced. Study of SMAs has continued at an increasing pace since then, and more products using these materials are coming to the market each year. One of the drawbacks of conventional SMAs is that they are slower to respond because they rely on variations in temperature and the flow of heat. Another class of materials was discovered in late 1990s, which can undergo large reversible deformations in an applied magnetic field. These materials are now known as magnetic shape memory alloys (MSMA)[5]. Compared to the ordinary SMAs, the magnetic control offers faster response in the MSMAs.

Taken together, many common experimental features from these quite distinct types of materials systems - CMR manganites, giant MCE materials and MSMAs, are indications of a common underlying physics at least at the phenomenological level. The researchers at the LTPL, CAT believe that the disorder-influenced first order phase transition (FOPT) provides the basic framework to understand the wide varieties of experimental results in these different classes of functional magnetic materials. This idea of generality is mainly developed on the basis of experimental works performed by these researchers and their collaborators on another class of magnetic materials, namely, doped-$CeFe_2$ alloys. These relatively simple doped $CeFe_2$ alloys [6] with magneto-structural transition [7] have been used as test-bed materials systems to study in some details a first order ferromagnetic (FM) to antiferromagnetic (AFM) transition. Based on detailed study of ac-susceptibility, dc-magnetization and magnetotransport, it is shown that the key features associated with the AFM-FM transition in manganites and MCE materials are clearly observed in this system and that these are a consequence of phase-coexistence and metastability arising out of a disorder-influenced FOPT. A brief summary of this work is narrated below. To extend the same idea to MSMA, researchers at LTPL, CAT have studied a magneto-martensitic transition in a new ternary alloy system NiCoAl showing magnetic shape memory effect [8].

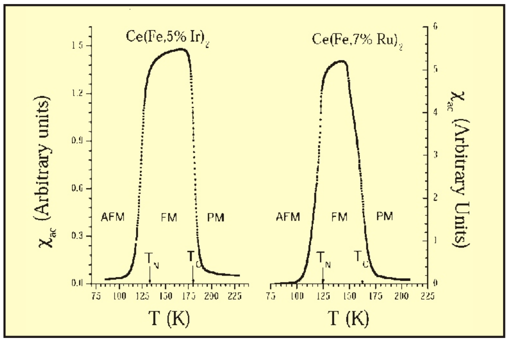

Fig. T.1.2 *ac-Susceptibility (χ) versus temperature (T) plots for Ir and Ru-doped $CeFe_2$ alloys [9]*

Fig. T.1.2 shows the ac-susceptibility (χ) for a couple of Ir and Ru-doped $CeFe_2$ sample as a function of temperature (T) [9]. A sharp increase in χ at a particular temperature T_{Curie} while decreasing T marks the onset of paramagnetic (PM)- to FM transition. Below T_{Curie}, susceptibility more or less flattens out before decreasing sharply at a lower temperature T_N that is indicative of an FM-AFM transition. There is no effect of thermal cycling on the PM-FM transition and this is in accord with the second order nature of this transition [9]. Within the same experimental resolution, however, distinct thermal hysteresis is observed across the FM-AFM transition [9]. This is necessarily a signature of a FOPT. The phase coexistence across this FOPT is studied in detail using minor hysteresis loop (MHL) technique [9,10]. This technique was developed earlier in LTPL, CAT in the context of research in vortex matter phase transition in type-II superconductors [11].

Fig. T.1.3 *M vs T plots for $Ce(Fe_{0.96}Ru_{0.04})_2$ alloy [12]*

Fig. T.1.4 *Resistivity ratio vs T plots for $Ce(Fe_{0.96}Ru_{0.04})_2$ alloy [12]*

Fig. T.1.3 shows magnetization (M) versus temperature (T) plot for a 4%Ru-doped $CeFe_2$ sample in an applied field of 20 kOe [12]. Three different measurement protocols were used: zero-field cooled (ZFC), field-cooled cooling (FCC) and field-cooled warming (FCW). A rapid rise of M with decreasing T below ~210K indicates the onset of PM-FM transition and it is thermally reversible. The FM-AFM transition is marked by the sharp drop in M below 50K and shows substantial thermal hysteresis, which is necessarily a signature of FOPT. It should be noted that the FCC curve does not merge with the ZFC curve down to the lowest measured temperature of 5 K. Similar measurements with applied fields varying between 100 Oe and 30 kOe shows that thermal hysteresis broadens with increasing H, and when H ≥15 kOe the M_{FCC} (T) and M_{ZFC} (T) curves fail to merge. The onset of the FM-AFM transition gives rise to marked increase in electrical resistivity (see inset of Fig. T.1.4). The FM-AFM transition can be suppressed by an applied H and this gives rise to GMR effect, which is clearly visible in Fig. T.1.4. The schematic H-T phase diagram based on the magnetisation measurements for this 4%Ru-doped $CeFe_2$ alloy is shown in Fig. T.1.5 with $T_{NW}(T_{NC})$ as the temperature of the sharp rise (fall) in M in the ZFC (FCC) cycle (see inset of Fig. T.1.5). T* is the low T point where M_{ZFC} and M_{FC} merges and T** is the high T counterpart. T** and T_{NC} appear almost the same in our present magnetisation measurements. Similar H-T phase diagram can be obtained through resistivity measurements and in such measurements (under same experimental protocol) T** and T_{NC} can be distinguished clearly [12]. Note that T_{NW} (H) <T_{NC} (H), i.e. the onset of nucleation of the AFM state on cooling occurs at a higher temperature than does nucleation of the FM phase during warming. This is a signature of a disorder-broadened FOPT. Such influence of disorder is observed in the same sample in the field induced transition also and this is discussed in details in Ref.13. Fig. T.1.6 shows schematic curves of the free energy density expressed in terms of an order parameter S as $f(T,S) = (r/2)S^2 - wS^3 + uS^4$ for a first order transition, where w and u are positive temperature independent constants. At $T=T_N$ the high-T and low-T phases coexist. The standard treatment [14] assumes that r(T)=a[T-T*], where a is positive and temperature independent, and where d^2f/dS^2 at S=0 vanishes at T=T*. The limit of metastability on cooling is reached at $T^*=T_N - w2/(2ua)$ [14], but finite energy fluctuations can destroy the supercooled state in the temperature regime $T^* < T < T_N$. Similarly T** is the limit of metastability while warming. In the low temperature AFM regime a transition from the zero field AFM state to FM state can be induced by application of H [13]. As in the case of temperature variation, this field induced first order AFM-FM transition is also marked by distinct hysteresis and phase-coexistence and, accordingly the limits of metastability H* and H** can be defined [14].

Fig. T.1.5 *H-T phase diagram for* $Ce(Fe_{0.96}\,Ru_{0.04})_2$ *alloy*

Fig. T.1.6 *Schematic free energy curves for* $T^*<T<T_N$. *The high temperature state remains in a local minimum and is stable against infinitesimal energy fluctuations in this temperature regime. The barrier height between the minima corresponding to the high and low temperature phases at TN (curve a) decreases with the decrease in T and goes to zero at* T^* *(curve d)*

H-T phase diagram in Fig. T.1.5 shows that it is possible to retain residual FM state in this 4%Ru-doped $CeFe_2$ alloy down to the lowest T of measurements by following the FCC path with applied H≥15 kOe. This is exactly what is known as field annealing of FM state in manganites systems [15]. It has been shown that this residual FM obtained by the FCC path is very much metastable in nature and can be erased by field cycling [12]. All these observations can be rationalized in terms of supercooling of the FM state. While cooling across the first order FM-AFM transition, some amount of the FM state will supercool into the T regime well below the transition line. It is clear from fig. T.1.5 that with applied H<15 kOe the supercooled FM state will cease to exist below a finite T and one can reach the stable AFM state. This is indicated by the merger of the FC and ZFC magnetization. With H≥15 kOe some amount of supercooled FM state remains down to the lowest T of measurement. The region between $T_{NC}(H)$ and T*(H) lines in Fig. T.1.5 marks the phase-coexistence region formed during the cooling path. This region consists of mixtures of AFM and FM clusters and it is metastable in nature. A new concept, namely, 'lack of end point memory effect' [16] has been used to study in details the metastable nature of this phase-coexistence regime [12,13]. Any field cycling in this metastable phase-coexistence region (obtained via FCC path) introduces energy fluctuations, which drive the clusters of metastable FM state to the stable AFM state. Further support of metastable nature of the phase-coexistence state has come from large relaxation in both magnetization and resistivity data [13].

The actual composition in any alloy or doped compound varies around some average composition simply due to the disorder that is frozen in as the solid crystallises from the melt. It was proposed earlier [17] that such static, quenched in, purely statistical compositional disorder could under certain circumstances introduce a landscape of transition temperatures in a system undergoing FOPT. Effect of quenched disorder in a FOPT was probably known to the metallurgists for ages; this can also be seen in day to day life by throwing some salt in boiling water which gives rise to inhomogeneous boiling. However a systematic study of the effect of disorder on a FOPT process started only in mid 1970s [17]. Detailed computational studies [2] confirm the applicability of disorder-influenced FOPT in CMR manganites and further emphasise that phase-coexistence can occur in any system in the presence of quenched disorder whenever two states are in competition through a FOPT [2]. Such intrinsic disorder induced landscape of transition temperature/field has actually been observed across the vortex solid melting transition in a high temperature superconductor BSCCO [18]. The applicability of such a picture in the AFM-FM transition of the doped-$CeFe_2$ alloys has now been pointed out through an imaging study AFM-FM transition using a micro Hall probe [19]. LTPL, CAT has collaborated with the Experimental Solid State Physics (EXSS) group of Imperial College (IC), London in this study of phase-coexistence in $CeFe_2$-alloys [19] and this collaboration has now been extended to other functional magnetic materials. Two state-of-the art experimental techniques namely micro-Hall probe scanning and microcalriomtry have been specifically employed for such study. It was observed both in T and H variation measurements in doped-$CeFe_2$ alloys that the FM clusters of various sizes appear in random positions of the sample at the onset of the AFM-FM transition. As the temperature or field is increased newer FM clusters appear until the whole sample is converted in to the FM state. This is clearly indicative of the local variation of the AFM-FM transition temperature (T_N) or field (H_M) leading to a rough T_N/ H_M landscape. This distribution of T_N or H_M gives rise to the impression of a global rounding of the transition in bulk measurements. This imaging study also provided a visual proof of supercooling of the FM State across the AFM-FM transition and that such supercooled state can easily be destabilized with a small energy fluctuation [19]. In the less disordered samples, the growth process of the clusters is relatively fast with smaller number of nucleating clusters, which suggests that different disorder landscapes can control nucleation and growth with the key point that if the growth is slow enough, percolation will occur over an observable T or H interval before phase-coexistence collapses. Such percolative behaviour can be controlled by subtle changes in sample doping, and the ramification to tuning the functionality of the CMR-manganites systems, for example, is obvious [19].

Fig. T.1.7 *M vs H plots of* $Ce(Fe_{0.96}Al_{0.04})_2$ *obtained after cooling in zero field at T=5K. Note that the virgin M-H curve lies outside the envelope M-H curve[20]*

The magneto-structural coupling in doped-$CeFe_2$ alloys [7] probably plays an important role in certain anomalous low temperature properties, which are particularly visible in Al-doped $CeFe_2$ alloys. In these alloys below 20K the field induced FM state does not revert back completely to the AFM state on withdrawal of the applied field [20,21]. This gives rise to the striking feature of the ZFC virgin M-H curve lying out of the envelope M-H curve obtained by subsequent field cycling between $\pm H_{max,}$ where $H_{max} >> H_M$ (see Fig.T.1.7). This anomalous feature is clearly reflected in the field dependence of resistivity as well [21]. A very similar behaviour has now been observed in CMR Mn-oxides and magnetocaloric materials [22]. As a possible explanation of the observed anomalous features, the researchers at LTPL, CAT have introduced the idea that the kinetics of the FM to AFM transition gets arrested at low T and in high H like in a quenched metallic glass [20]. Metastable state thus obtained will have qualitatively different features from the usual phase-coexistence regime expected across the disorder-influenced FOPT.

In a nutshell, a first order magneto-structural transition is the common feature in CMR-managanites [2], systems with giant MCE [4] and ferromagnetic shape memory alloys [8]. Phase-coexistence and metastability are the essential features of this transition process and influences the functionality of these materials. Understanding these phenomena in details will help in tuning the functionality of the existing materials as well as finding new materials with better functionality.

The developments of the Hall-imaging system and the microcalorimeter mentioned above are typical examples of unforeseen technological spin-off, which often arises during a curiosity driven science research. Commercial Hall-probes available for magnetic field imaging have dimension in millimetre length scale. The FM-AFM phase-coexistence of the present interest takes place in micrometer scales and below. That demands the development of state-of-the- art calibrated high field-resolution Hall-probes of size 50 microns and less. Such micro-Hall probe scanning system is quite robust, non-intrusive and cheaper than magnetic force microscope, and can be really useful in studying the magnetic profiles of various systems including the recording media. The micro-calorimeter, which uses micro-gram sample, is developed mainly to obtain thermodynamic evidence of the phase-coexistence in the micrometer scale. Such micro-calorimeter will definitely benefit the scientific community as standard calorimetric measurements require fairly large amounts of samples (50 mg and above). Imperial College like many western academic institutions and national laboratories have technology transfer/support cell, which can consider the feasibility of transferring the technology developed (albeit to satisfy the scientific questions raised during LTPL, CAT—IC, EXSS collaboration) to the appropriate industries.

Spintronics materials:

Information processing technology has so far relied on purely charge-based semiconductor devices. These conventional electronic devices work on the principle of controlling the flow of current by an applied voltage. This was the same basic principle of working in the first transistors used more than 50 years ago. However, as rapid progress in the miniaturization of semiconductor electronic devices leads towards the scale smaller than 100 nm, one will soon reach the mysterious quantum world with many uncertainties. Efforts are underway to avoid this bottleneck, and new breakthrough is welcome by the industries if existing material processing infrastructure can be used. A new idea has emerged during the last decade to realise electronic devices which use electron spin instead of the charge and this has given rise to an entirely new subject of "Spintronics"[23]. The realization of spintronic devices requires the ability to control injection, transport and detection of spin-polarized charge carriers in a semiconductor channel, and to manipulate their density by an applied field.

The "Spintronics" related functionality promises devices such as light-emitting and laser diode with polarized output, transistors with low power use, integrated logic and memory chips (leading to computers that would turn on immediately without having to boot-up the operating system that is currently stored in magnetic memory in the hard drive), and powerful remote sensor systems that incorporate magnetic detection functions with on-chip signal processing and off-chip optical communication. Manipulating spins takes much less energy and is much faster than the conventional field effect transistor process, and thus the spintronics devices are going to be energy efficient too.

The crucial element in a proposed spintronics device is the spin-injector source, which will inject spin-polarised charge carriers in semiconducting channel. One source of spin-injector materials is a magnetic semiconductor. However, there is no suitable magnetic semiconductor to date, working at room temperature and the search is on world wide to find such a material. The other possibility is to find a ferromagnet with high spin-polarization, which can be coupled to an appropriate semiconductor to give rise to a suitable metal-semiconductor hybrid structure. There is a family of intermetallic compounds known as Heusler alloys, which can be a source of such spin-injector materials [24]. One member of this family, namely NiMnSb is a subject of much scrutiny in recent years in this regard. Although NiMnSb was quite promising initially, detailed investigations by various groups including LTPL, CAT have now cast some doubt on the functionality of this material at room temperature. Some unforeseen but related S&T development, however, took place during such exercise. NiMnSb/Si hybrid structures revealed GMR effect at room temperature [25]. This GMR effect is quite unusual and distinctly different from the usual GMR effect described earlier. In conjunction with some other related recent developments, it is indicative of an emerging area of interesting physics namely 'disordered ferromagnetic metals' with promising technological implications.

In the area of magnetic semiconductors, effort is being made to induce ferromagnetism in a semiconductor at practical operating temperatures by introducing appropriate magnetic dopants such as Mn, Cr or Fe. The highest magnetic transition temperature or Curie temperature achieved (reproducibly) today is 150K in Mn-doped GaAs. Although the Curie temperature in magnetically doped GaAs and GaN is steadily being pushed towards the room temperature, these materials can only be fabricated in thin-film form, are heavily defective, and are not obviously compatible with Si, the most favoured materials in the industry. So there is an incentive to look in to newer classes of semiconductor materials involving Si. There exists such a semiconductor material namely FeSi which has been already drawing attention of the condensed matter physics community for its enigmatic physical properties. LTPL, CAT intuitively embarked upon some activity with the Co-doped FeSi alloys, and such effort paid some dividend in the form of finding interesting magneto-transport properties in these alloys [26]. These Co-doped FeSi alloys are now being considered as promising spin-injector materials [27].

Interdisciplinary research gains:

Such S&T activity in these technologically important functional magnetic materials is also helping in the deeper understanding of the FOPT process. Study of a FOPT process involving the water-ice transition has remained a very active area of research since it has tremendous implications for the biological as well as ecological systems [28]. Although many aspects of a FOPT, namely phase-coexistence and metastability (supercooling/superheating), nucleation and growth are actually known intuitively to the metallurgists and crystal grower for the ages, a systematic and universal understanding of a FOPT process is still lacking. This is now more than of academic interest especially when it is becoming more and more apparent that a first order transition process is involved in many of the practical issues in our surroundings [28]. Some of these magnetic materials of current interest can actually be used as test bed materials to study a FOPT process in a two-parameter magnetic field (H)– temperature (T) phase space. Such exploration in conventional pressure (P) – temperature (T) phase space is relatively difficult. Knowledge gathered in exploring H-T phase space in magnetic materials can also be applied in other areas of technological interest namely vortex matter phases of type-II superconductors and ferroelectric materials. While implications in ferroelectric materials are well known, the understanding of vortex matter phase space is absolutely necessary for tuning the dissipation less current carrying capacity of a type-II superconductor.

Many of the potential sources for spin-injector materials show physical properties which are of fundamental interest in the modern day condensed matter physics namely, metal-insulator transition, Kondo lattice and non-fermi liquid behaviour, quantum criticality etc. These are basically manifestation of strong electron-electron correlation. These families of materials are also hunting grounds for magnetically mediated unconventional superconductivity and materials with large thermoelectric response. It is a fair expectation that newer interesting properties leading to new kind of functionality can also emerge as a by-product of the ongoing research and some such indications are already there.

Future Outlook:

While GMR using multilayer structure is already a matured technology, CMR materials in spite of their initial promise is yet to reach that stage. However, the recent identification of the phase-coexistence as a crucial element to understand the CMR properties [2] have raised the prospect of tuning their properties for technological applications. The progress from basic research to technological applications in magnetocaloric materials and magnetic shape memory alloys is also taking place very rapidly. It is clear that MCE is one of the most critical parameters defining the performance of a magnetic refrigerator. The stronger the MCE the higher the efficiency of the device, hence the hunt is on to find better MCE materials. It is interesting to note here that some of the CMR mangnaites and magnetic shape memory alloys are now found to be showing large MCE around room temperature. With the improvements in magnetic materials, magnet systems and understanding of the active magnetic regenerator cycle, there is promise that a mature magnetic refrigeration technology will soon produce thermodynamic efficiencies comparable to or higher than the best available gas compression devices. It provides an example of closely intertwined science and technology reinforcing each other on the way from laboratory based basic research to useful futuristic technology. Research on the spintronics materials, on the other hand, is a typical example of technology motivated basic research. To make a spin-transistor work, various problems need to be solved. To find a suitable 'spin-injector' material is an example of such problems. In the process of finding technologically suitable spin-injector material, newer basic phenomenon like half-metallic ferromagnetism [24] is being discovered which in turn broadens the horizon of research in magnetism.

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