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Magnetic studies in mesoscopic length scale using polarized neutron spectrometer at Dhruva reactor, Trombay

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Abstract. The details of construction and principle of polarized neutron spectrometer at Dhruva reactor, Trombay, for neutron depolarization studies have been described. The feasibility in carrying out neutron depolarization studies in order to know the nature of magnetic ordering in various types of magnetic systems on mesoscopic length scale has been shown.

Keywords. Polarized neutron; neutron depolarization study; mesoscopic probe; domain effect; spin glass-like ordering.

PACS Nos 61·12; 75·60; 75·50

1. Introduction

When a polarized neutron beam traverses through an unsaturated ferro/ferrimagnetic material, a change in the state of polarization P arises from the Larmor precession of P around the local magnetic induction B of magnetic domains. This change of polarization of a polarized neutron beam after transmission through a magnetic material is known as neutron depolarization. The classical approach of depolarization was first initiated theoretically in 1941 by Halpern and Holstein [1], later extended by Rekveldt [2, 3] and the Leningrad group [4]. The quantum mechanical approach [5] is based on small angle neutron scattering (within the analyzer aperture) by magnetic inhomogeneities. However, both approaches are generally equivalent [6]. The first neutron depolarization (one-dimensional) experiment was performed by Burgy *et al* in 1950 [7] in order to determine the mean domain size in iron. Recently, we have set up a neutron polarization analysis spectrometer (PAS) at Dhruva reactor, Trombay for magnetic scattering studies [8]. In our earlier report [8] the description of the spectrometer and its possible application in polarization analysis mode (in scattering geometry) have been discussed. In principle, this type of polarized neutron spectrometer can be used in different modes, such as, diffraction mode (2-axis configuration), polarization analysis mode (3-axis configuration in scattering geometry) and depolarization mode (3-axis configuration in transmission geometry). Here we report the principle of neutron depolarization, the recent upgradation of PAS suitable for neutron depolarization studies, and possible applications of such studies.

2. Principle of neutron depolarization

The neutron is an elementary particle with spin quantum number $s = 1/2$ and only one component along an arbitrary axis can be measured. When the average of the spin component of neutrons from a neutron beam is not equal to zero the neutron beam is said to be polarized. The polarization direction is defined as that direction in which the average spin component measured has an extreme. The polarization vector **P** of a neutron beam is the expectation value of $\hat{\sigma}$, the Pauli spin operator and can be written as

$$\mathbf{P} = \frac{I_+ - I_-}{I_+ + I_-}\mathbf{P}_0, \tag{1}$$

where $\mathbf{P}_0$ is a unit vector in the direction of the polarization and I_+ and I_- are the neutron beam intensities with + (parallel) and − (antiparallel) spin components along the direction $\mathbf{P}_0$, respectively. In an unsaturated ferromagnet or ferrimagnet, the magnetic domains exert a dipolar field on the neutron polarization and depolarize the neutrons owing to the Larmor precession of the neutron spins in the magnetic field of domains. The time dependence of **P** obeys the classical equation of motion and can be given as

$$\frac{\mathrm{d}\mathbf{P}}{\mathrm{d}t} = \gamma(\mathbf{P} \times \mathbf{B}), \tag{2}$$

where $\gamma = 2\mu_n/\hbar = 1{\cdot}83 \times 10^8$ radians $\mathrm{sec}^{-1}\,\mathrm{T}^{-1}$. This means that the polarization vector **P** precesses around the field direction with frequency $\omega_L = \gamma|\mathbf{B}|$, known as the Larmor precession frequency.

Mitsuda and Endoh derived the wavelength dependent transmitted polarization $P(\lambda)$ [9] in a one-dimensional case which can be written in the most general form as

$$\mathrm{P}(\lambda) = \left[\left\langle \frac{B_\parallel^2}{B^2} \right\rangle_B + \left\langle \frac{B_\perp^2}{B^2} \right\rangle_B \langle \cos(cB\delta\lambda) \rangle_\delta \right]^N, \tag{3}$$

where $B_\parallel$ and $B_\perp$ are the components of the local magnetic induction B parallel and perpendicular to the direction of incident neutron polarization $(-z)$, δ is the mean domain size and λ the neutron wavelength. The constant c is related to the neutron gyromagnetic ratio $g[c = gm/2\pi\hbar = 4{\cdot}63 \times 10^{-10}\,\mathrm{Oe}^{-1}\,\mathrm{Å}^{-2}]$ and N is the average number of the domains along the path of neutrons ($N = d/\delta$, d is the sample thickness). The angular brackets $\langle\rangle_B$ and $\langle\rangle_\delta$ represent the ensemble average over the local induction B in each domain and over the domain size, respectively. In each ferromagnetic domain, the spin component parallel to the induction B does not precess whereas the component perpendicular to B does. The precession angle for a neutron of wavelength λ, travelling a distance δ in a field B is $\phi_\delta = (4{\cdot}63 \times 10^{-10}\,\mathrm{Oe}^{-1}\,\mathrm{Å}^{-2})B\delta\lambda$. In antiferromagnets there is no net magnetization and hence no depolarization is expected in antiferromagnets. (In some special situations depolarization may occur in antiferromagnets due to (a) Bragg scattering within the analyzer aperture and (b) uncompensated ferromagnetic planes at the surface of the sample particle [10].) As the neutron depolarization technique probes the magnetic inhomogeneity on a mesoscopic length scale say, from 100 Å to a few thousand ångströms, magnetic inhomogeneity on

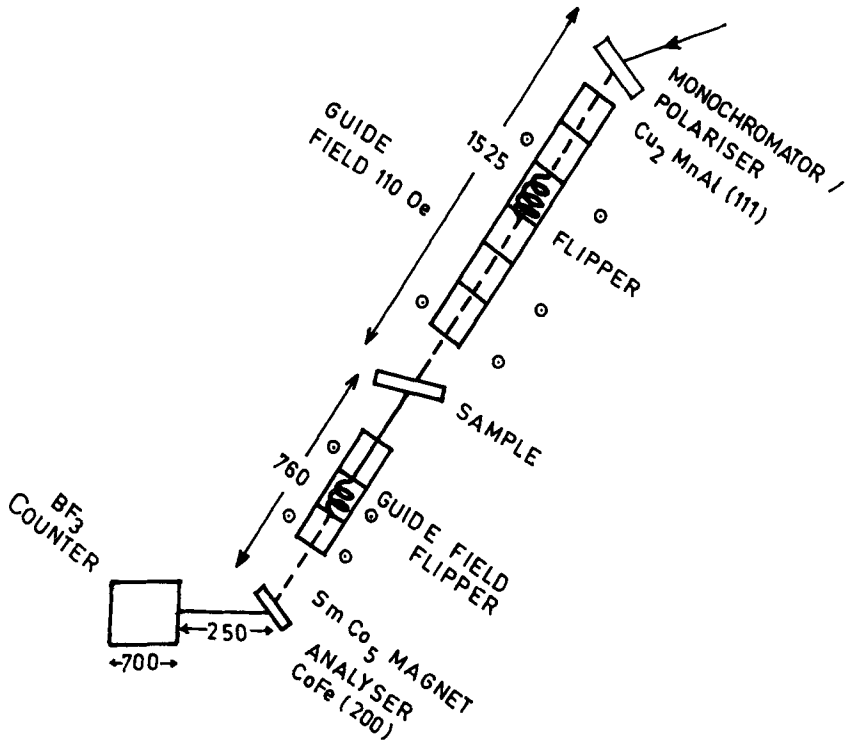

Figure 1. Schematic diagram of the polarized neutron spectrometer for neutron depolarization studies at the Dhruva reactor.

an atomic scale, as in paramagnetic state, has no effect on the neutron polarization, because the temporal spin fluctuation is too fast (10^{-12} s or faster) for the neutron polarization to follow the variation of the magnetic field B acting on the moving thermal neutron. Similarly no depolarization is expected in a true (canonical) spin glass state. In a true spin glass phase the spins are randomly frozen in space on a microscopic length scale and, as a result, the magnetic induction averages out to zero on a mesoscopic length scale. Hence no depolarization is found in true spin-glass systems.

In three-dimensional depolarization study [11, 12] one measures all the nine components of the depolarization matrix: With incident neutron polarization in the direction x, y or z, after transmission, the polarization is measured in all the three directions, namely, x, y and z directions. However, in one-dimensional depolarization study [9, 13] one measures only one component (say z–z component) of the depolarization matrix.

3. Experimental details

The polarized neutron spectrometer is like any unpolarized neutron spectrometer with additional devices such as a polarizing magnet on the 1st axis, an analyzing magnet on the 3rd axis, a magnet at the sample position (2nd axis), magnetic guide modules along the neutron path and two rf flippers. A schematic diagram of neutron polarization

analysis spectrometer (PAS) is shown in figure 1. For details, refer to our earlier report (ref. [8]).

In order to improve the incident neutron flux we have taken the following steps. (a) The $Co_{0.92}Fe_{0.08}(200)$ polarizer-cum-monochromator crystal has been replaced by $Cu_2MnAl(111)$ as the latter has about 3 times higher reflectivity than the former. (b) The incident neutron wavelength is changed from 0·999 Å to 1·201 Å as the peak of the Maxwellian profile of the neutron flux coming out of Dhruva reactor falls around 1·20 Å.

The monochromator-cum-polarizer Cu_2MnAl Heusler alloy is in a single crystal form, rectangular shaped, $50 \times 50 \times 4$ mm (thick) in size. The polarizing plane is the (111) plane in transmission geometry. This crystal is mounted between the pole pieces of a $SmCo_5$ permanent magnet assembly, which provides a vertical uniform field of 2·8 kOe. This assembly is housed on the first axis inside a massive sector-type monochromator drum, 2 m in diameter [14]. The polarization efficiency of the monochromatic ($\lambda = 1{\cdot}201$ Å) neutron beam obtained from this Cu_2MnAl polarizer is $98{\cdot}83 \pm 0{\cdot}01\%$. A similar polarization sensitive analyzer single crystal: either $Cu_2MnAl(111)$ identical to the monochromator/polarizer or $Co_{0.92}Fe_{0.08}(200)$ crystal disc-shaped, 50 mm in diameter and 1·5 mm thick in transmission geometry can be inserted in the pole gap of a $SmCo_5$ magnet assembly mounted on the third axis. The $Co_{0.92}Fe_{0.08}(200)$ and $Cu_2MnAl(111)$ polarizing/analyzing crystals may be considered as devices which have almost 100% reflectivity for up $(+z)$ and down $(-z)$ spin neutrons respectively and almost zero reflectivity for down $(-z)$ and up $(+z)$ spin neutrons, respectively. The purpose of the analyzer crystal is to analyze the spin state of neutrons scattered or transmitted from the sample.

The (222) reflection of Cu_2MnAl has a higher nuclear structure factor than its (111) reflection. As a result, the second order wavelength contamination can, in principle, be problematic. For second order wavelength ($\lambda/2$) contamination measurement, the spectrometer was set at $2\theta_s = 0°$ and a $Cu_2MnAl(111)$ single crystal identical to the polarizer was mounted on the third axis of the spectrometer. After setting the detector at $2\theta_A = 20{\cdot}35°$, corresponding to first order reflection of $\lambda = 1{\cdot}201$ Å and second order reflection of $\lambda/2$, the rocking curve of $Cu_2MnAl(111)$ analyzer crystal was taken. The background corrected integrated intensity obtained from this measurement has contributions both from λ and $\lambda/2$. Then, the detector was set at $2\theta_A = 10{\cdot}18°$ and rocking curve of the analyzer was taken. In this case ($2\theta_A = 10{\cdot}18$) only $\lambda/2$ scattering occurs. From these two independent measurements the $\lambda/2$ contamination was obtained and found to be of the order of 1%, which can be neglected in the data analysis for all practical purposes.

In order to carry out low field neutron depolarization measurements an H-type electromagnet was designed, fabricated and installed at the sample position on the second axis. The schematic diagram of the H-type electromagnet is shown in figure 2. This H-type electromagnet is designed to provide either a horizontal or vertical field. This can carry an APD make closed cycle helium refrigerator for the low temperature measurements in the presence of a magnetic field. The yoke, pole piece and current coil configuration were optimized using a computer program. The magnet consists of four current carrying coils. Each of the two large and two small coils have 1746 and 390 turns of 14 SWG super-enamelled Cu wire (2·05 mm diameter, $\Omega/m = 5{\cdot}209 \times 10^{-3}$)

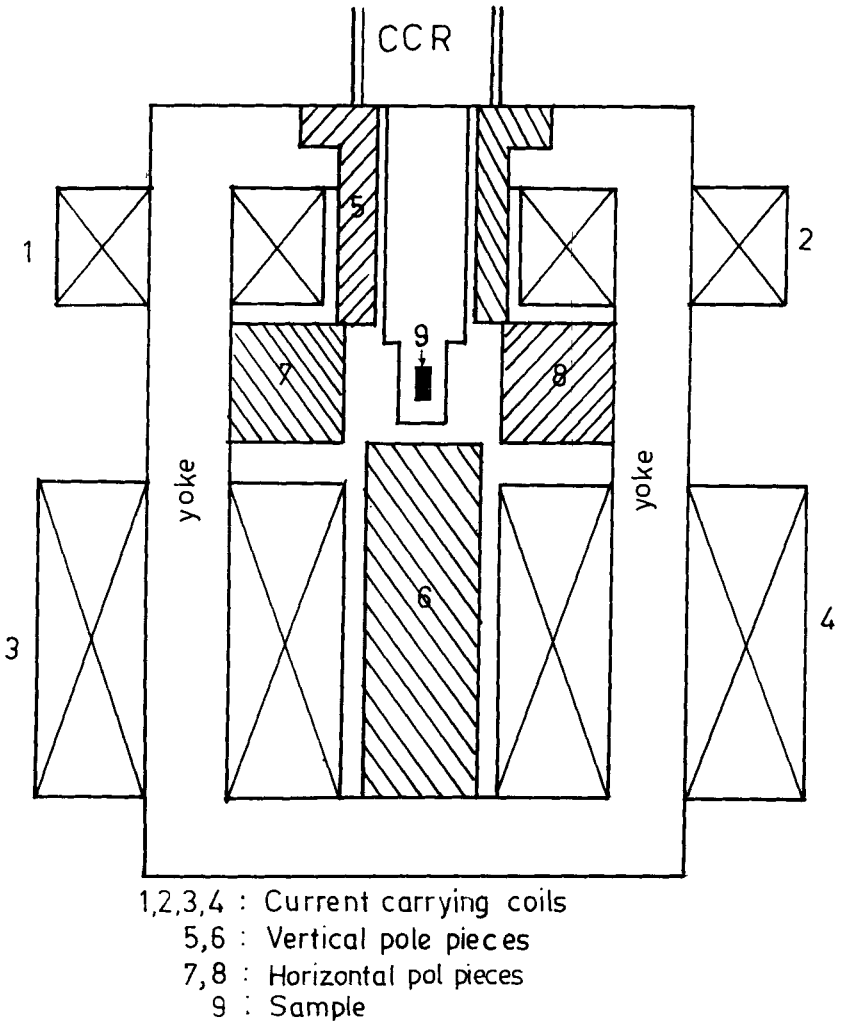

Figure 2. Schematic diagram of the H-type electromagnet along with the closed cycle helium refrigerator used for polarized neutron study. The shaded portions indicate the pole pieces.

respectively. The bobbins are made out of Al with a central gap of 41 × 41 square mm along the axis of the solenoid. The dimensions of the large coils are as follows: internal dimension = 43 × 43 mm, outer dimension = 170 mm diameter and height = 170 mm. The small coils have the following dimensions: internal dimension = 43 × 43 mm, outer dimension = 140 mm diameter and height = 60 mm. The magnet yoke, pole pieces and pole shoes were fabricated out of Tata grade mild steel. The demountable pole shoes are tapered for the purpose of concentrating the magnetic flux. The magnet is air cooled and is operated from electronically controlled constant current DC power supply. The working range of this power supply is 0–100 V DC and 0–10 A current. Several pairs of pole pieces with various pole shoes have been made to have different

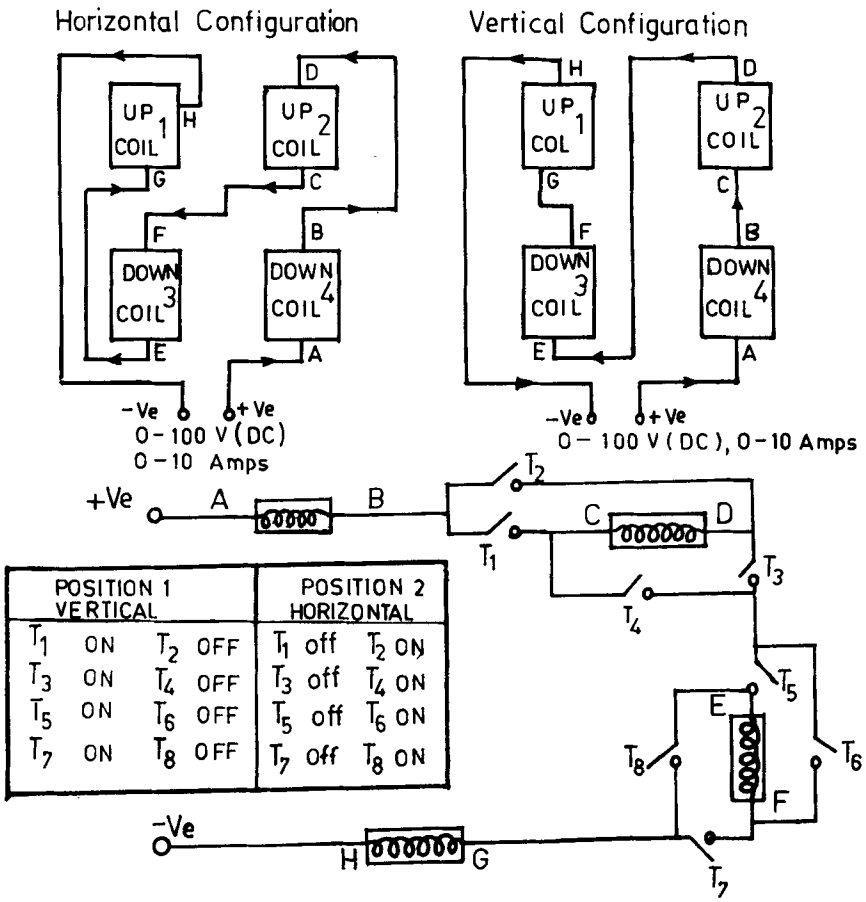

Figure 3. Block diagram of the electrical connections to the current carrying coils for both vertical and horizontal field configurations of the H-type electromagnet.

pole gaps of ~20–60 mm at the sample position with a field homogeneity of about 97·8% in both vertical and horizontal variable field configurations. One such pair is mounted at a time. A proper electrical connection (shown in figure 3) to all four current carrying coils for both vertical and horizontal field configurations have been provided through a rotary switch having 4 poles with 2 way configuration. The value of the measured field with a hollow vertical upper pole piece which can carry a closed cycle refrigerator was found to be 2·2 kOe with a 26 mm pole gap and 5 A current. The measured field values with solid pole pieces of 20 mm pole gap and 5 A current are 3·1 and 2·9 kOe in the vertical and horizontal field configurations, respectively.

In order to carry out low temperature measurements (10–300 K) an APD closed cycle helium refrigerator (CCR) can be easily mounted (using a brass stand) on the H-type electromagnet located on PAS. Figure 4 shows the H-type electromagnet with the CCR mounted on the 2nd axis of the spectrometer. This CCR is a two stage, water-cooled rotary compressor type helium gas cryogenic refrigerator. The sample is

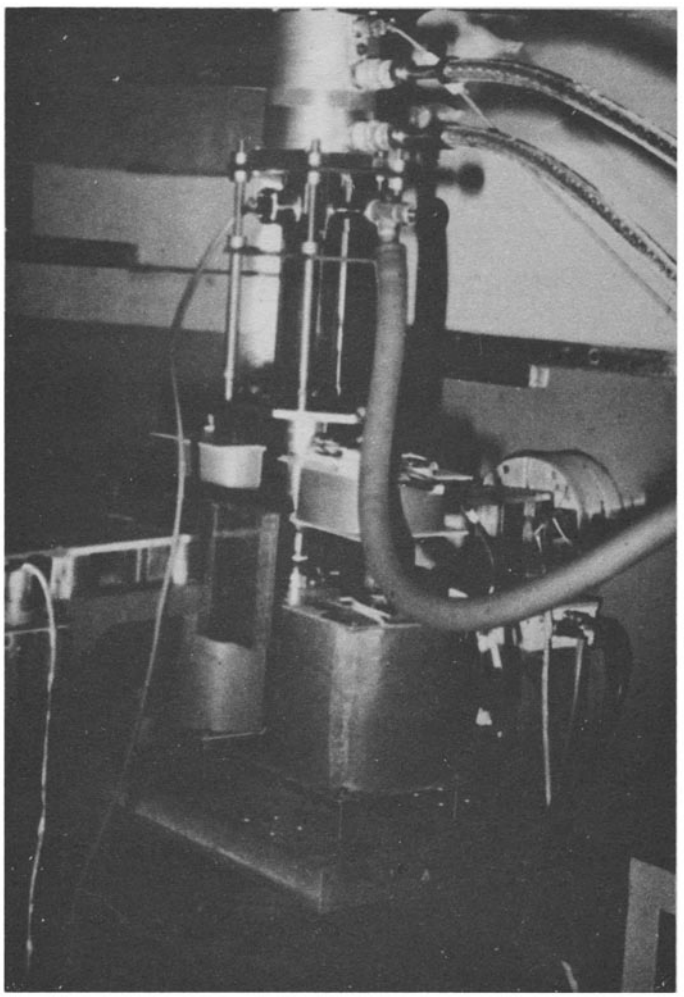

Figure 4. Photograph of the H-type electromagnet with the closed cycle helium refrigerator mounted on the 2nd axis of the polarized neutron spectrometer at the Dhruva reactor.

mounted from the bottom of the cold head. A Scientific Instruments Inc. make of 9650 microprocessor-based digital temperature controller is used for controlling sample temperature with an accuracy better than 0·1 K.

For depolarization measurements it is necessary to reverse the direction of polarization before or after transmission. The principle of resonance inversion is used (as described in ref. [8]) to achieve neutron spin reversal or flipping.

The one-dimensional depolarization measurements are performed with $Cu_2MnAl(111)$ as polarizer and $Co_{0\cdot92}Fe_{0\cdot08}(200)$ as analyzer i.e. with the incident neutron beam polarized along the $-z$ direction (vertically down) with a beam polarization of 98·83 $\pm$ 0·01% and the transmitted neutron beam (transmitted through the sample) polarization

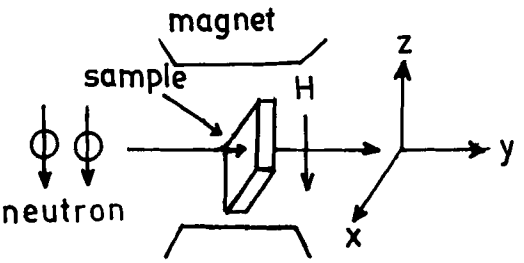

Figure 5. Schematic representation of a neutron depolarization measurement.

is measured along the $+z$ direction [15]. The first rf flipper is used before the sample for flipping the incident neutron spin.

In neutron depolarization study one measures the polarization ratio (R) [16] (ratio of transmitted intensities for $+z$ to that of $-z$ states of incident neutron spin) which is a measure of z–z transmitted polarization. The expression for polarization ratio (R) can be given in the following form

$$R = \frac{1 - P_i D P_A}{1 + (2f - 1) P_i D P_A}, \tag{4}$$

where P_i is the incident beam polarization, P_A is the efficiency of the analyzer crystal, f is the efficiency of the rf flipper and D is the depolarization coefficient (due to the sample under investigation). $P_i D (\equiv P)$ therefore corresponds to the transmitted beam polarization. In the absence of any depolarization in the sample the depolarization coefficient $D = 1$. When there is total depolarization $D = 0$ (which corresponds to $R = 1$). Hence the transmitted neutron polarization $P_i D$ decreases as depolarization increases. The measurements of the instrumental parameters P_i, P_A and f are discussed in ref. [8].

Flat rectangular aluminium sample holders with different effective thicknesses of 1 to 10 mm are used for depolarization studies of powder samples as a function of sample thickness. The powder samples are used in the form of compressed pellets. If the sample is in thin plate form (as in the case of amorphous metallic ribbons), the sample is kept between two thin aluminium plates. The sample is placed in the neutron beam in such a way that its plane surface (xz) remains perpendicular to the propagation direction (y) of the polarized neutron beam (see figure 5). The beam size is restricted with a cadmium slit which is within the size of the sample. An external magnetic field can be applied on the sample along the $-z$ or x direction using the electromagnet mounted on the spectrometer. The temperature of the sample can be varied from 300 to 12 K using the closed-cycle helium refrigerator with a temperature accuracy of better than 0·1 K.

Data acquisition programs for recording data in depolarization mode of operation have been written in Fortran and are stored and accessed by the control system.

4. Experimental results

Using this spectrometer, one-dimensional depolarization measurements have been carried out recently on various types of magnetic systems, such as anisotropic UCu_2Ge_2

intermetallic compound [17], superparamagnetic Ce_2Fe_{17} alloy [18], disordered ferrites $Zn_{0.5}Co_{0.5}Fe_{2-x}Cr_xO_4$ ($x \sim 0.9$, 1.0) [15] and frustrated $KMnFeF_6$ system [19] in order to understand the magnetic nature of these systems in mesoscopic length scales and hence to resolve the discrepancies between the commonly available results on macroscopic and microscopic length scales. We have also carried out similar studies on $Co_{1.4-x}Zn_xGe_{0.4}Fe_{1.2}O_4$ disordered ferrites [20] and $Fe_{90-x}Ru_xZr_{10}$ amorphous system [21]. In this paper we present illustrative examples of just one composition from each of these two systems.

(a) *$Fe_{90-x}Ru_xZr_{10}$ amorphous system*

A number of amorphous and crystalline materials show sequential paramagnetic–ferromagnetic-spin glass like transition i.e. so called reentrant spin glass transition (RSG), as the sample temperature is lowered. The RSG systems have drawn considerable attention as these investigations have raised a lot of controversy about their exact nature of ordering. For example, recent low field ac susceptibility, dc magnetization (in both low and high field region) and Mössbauer studies of $Fe_{90-x}Ru_xZr_{10}$ amorphous alloys [22, 23] show that Ru substitution destroys ferromagnetism very rapidly and the system goes directly from para to a spin glass-like state for more than 3 at. % of Ru. In order to study the nature of magnetic ordering in the spin glass like phase in $Fe_{85}Ru_5Zr_{10}$ we have carried out zero-field cooled (ZFC) and field-cooled (FC) neutron depolarization studies as a function of temperature and magnetic field.

The sample does not show any depolarization over the entire temperature range either in ZFC or in the FC case, when a field of 7 Oe is applied on the sample. But the same sample when cooled in 1200 Oe, shows field induced time dependent depolarization which led us to do a relaxation study. The relaxation measurement were carried out in the heating cycle (in field cooled case) with a cooling field of 1200 Oe and a measuring field of 7 Oe at 12 K and 80 K by looking at the depolarization caused by the sample. In these measurements, the transmitted neutron counts were measured in crossed polarizer-analyzer mode (i.e. Cu_2MnAl (111) as polarizer and $Co_{0.92}Fe_{0.08}$ (200) as analyzer and with rf flipper off) as a function of time. In this configuration, a higher count rate corresponds to higher depolarization and lower count rate corresponds to lower depolarization. The counting was started after reducing the field value from 1200 Oe to 7 Oe. At each time step the data were collected for about 5 min (normalized to a fixed monitor count).

At 12 K a strong depolarization was observed, which decreases as a function of time (figure 6). The observed decay of depolarization was found to be reasonably well described by a stretched exponential function of the form $N(t) = N_0 \exp[-(t/\tau)^{1-n}]$ with $\tau = 18.4$ h and $n = 0.88$. Present study confirms that at higher Ru concentration, ($x \sim 5$) the ferromagnetic correlation totally disintegrates. At this higher Ru concentration the system shows a true spin glass behaviour when the external field applied on the sample is low, but at relatively higher cooling field the formation of field induced spin-glass cluster is observed. From this field induced observed depolarization, it is apparent that the cluster growth mechanism is involved at a higher cooling field. When the sample ($x = 5$) is cooled in the presence of a higher external field it helps the clusters to grow in the framework of a cluster spin glass phase and cause depolarization. After removal of the field from 1200 Oe to 7 Oe, the clusters start decaying with time, causing

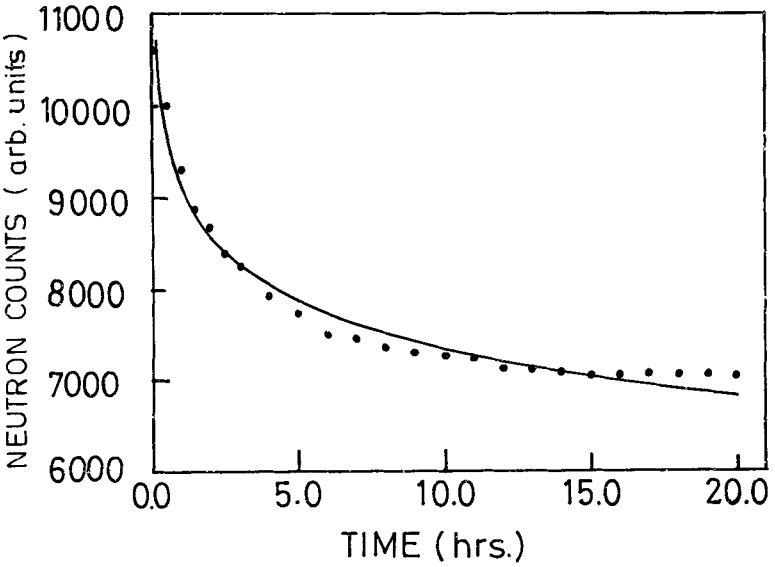

Figure 6. Time variation of depolarization for the amorphous $Fe_{85}Ru_5Zr_{10}$ sample at 12 K, solid line: stretched exponential fitted curve (see text).

the relaxation behaviour as shown in figure 6. The depolarization relaxation essentially scales to the relaxation of magnetization. Hence, figure 6 corresponds to the relaxation of magnetization of field induced spin glass clusters. This kind of slow, stretched exponential relaxation of magnetization are known to be exhibited by spin glass systems [24]. We would like to remark here, that as far as we know, the study of magnetization relaxation through neutron depolarization measurements has not been reported so far in literature.

(b) *Disordered ferrites*

In disordered spinel ferrites very often [25–27] one finds a broad hump in the ac susceptibility curve and a deviation between zero field cooled (ZFC) and field cooled (FC) magnetization (in low field values) around the same temperature where a broad peak occurs in the ac susceptibility curve. These features are normally interpreted as signatures of a spin glass like freezing. Our recent neutron depolarization study on $Zn_{0.5}Co_{0.5}Fe_{2-x}Cr_xO_4$ (with $x = 0.9$ and 1.0) clearly indicated that these macroscopic features (in low field ac susceptibility and magnetization measurements) which mimic a spin glass are not due to any spin glass like freezing but, a signature of kinetic freezing of magnetic domains. Here we will discuss similar results obtained on another disordered ferrite $Co_{1.4-x}Zn_xGe_{0.4}Fe_{1.2}O_4$ with $x = 0.6$. The real part of ac susceptibility curves of $x = 0.4$, 0.5 and 0.6 samples of this series shows a broad maximum around 260, 200 and 120 K, respectively [28]. The low field ZFC and FC data show deviation around the same temperature. These features (for $x = 0.6$) were taken as singnature of spin glass like freezing. We have carried out neutron depolarization measurements (both ZFC and FC) on $x = 0.6$ sample (figure 7). The procedure of measurement is same as described in ref. [15]. The presence of a ferrimagnetic domain

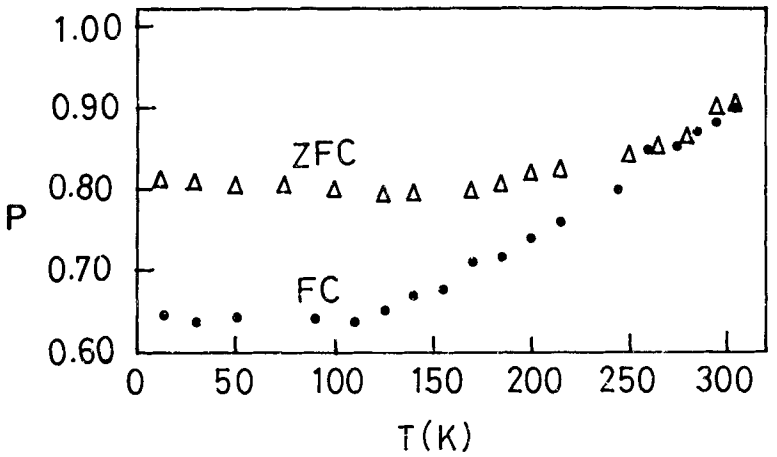

Figure 7. ZFC and FC polarization P versus temperature in a 30 Oe field for $Co_{0.8}Zn_{0.6}Ge_{0.4}Fe_{1.2}O_4$ ferrite.

structure is confirmed by the presence of significant depolarization up to 12 K, the lowest measured temperature. A continuous drop in both the ZFC and FC transmitted polarization right from room temperature (the highest measured temperature) up to about 160 K and 110 K for ZFC and FC cases, respectively, clearly indicates that the ferrimagnetic order parameter increases with decrease in temperature. The difference between the ZFC and the FC depolarization increases with decrease in temperature until about 110 K and it remains constant below 110 K. There is no indication of breakdown of ferrimagnetic domain structure at any temperature. The observed branching between ZFC and FC depolarization clearly tells us that the domain mobility strongly depends upon the cooling process of the sample. The observed higher depolarization in the FC case than in the ZFC case indicates that, when the sample is cooled in the presence of an external field, it helps the domains to grow. At this low field (the measured field value is 30 Oe) favourably oriented domains grow in size [29]. So a higher depolarization is expected from larger domains. It should be pointed out that our sample contains Co^{2+} ions, and this introduces uniaxial random anisotropy [30]. In the presence of such anisotropy field, the cooling in field cooled condition can induce a large unidirectional anisotropy so that very large domains can be maintained. From the above discussion, it can be inferred that the features in susceptibility and low-field magnetization (which mimic a spin glass like freezing) do not result from transition to any spin glass-like phase or a breakdown of ferrimagnetic correlation, but can be related to magnetic domain effects.

5. Conclusion

In order to know the nature of magnetic ordering on a mesoscopic length scale, 1-dimensional neutron depolarization study can be carried out using polarized neutron spectrometer at Dhruva reactor, Trombay. Using this spectrometer, zero-field cooled depolarization, field cooled depolarization and relaxation study through neutron depolarization can be carried out over a temperature range of 12 to 300 K and field range of about

$-3{\cdot}0$ to $+3{\cdot}0$ kOe. From our studies it is evident that interpretation of macroscopic observations alone can be quite misleading. Macroscopic properties of many systems, especially, in systems where frustrations are present, might have some common features but the mesoscopic nature of the system and the underlying physics can be quite different.

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