

Chapter 6

Summary and Future scope

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Summary

This thesis deals with the hyperfine interaction studies in some dilute magnetic alloys and radiation induced damage study in Rhodium metal.

Chapter I gives the general introduction and brief review of work done earlier in DMS system. It also includes theoretical background of magnetic interactions and study of defects in metals. Theoretical consideration for hyperfine interaction studies is also included in this chapter.

Chapter II discusses the experimental methodology and instrumentation used for characterization of the materials studied. Hyperfine interaction technique is the major experimental method used for the study of local interactions in the system. However, other measurements were also done using AC Susceptibility, Magnetoresistance, Hall effect and XRD technique.

Chapter III describes about the new possible DMS materials viz $\text{Sb}_{1-x}\text{Fe}_x\text{Se}_y$ alloy. Fe at 0.008 concentration in Sb showed a quadrupole doublet corresponding to FeSb_2 compound. But a small percentage of Se doping in this system brought magnetic hyperfine interaction. Fe concentration was kept constant at 0.008 and the Se concentration was varied between $0.03 \leq x \leq 0.30$. Mossbauer spectra showed ^{two} magnetic and ^{two} quadrupole sites. The value of quadrupole splitting measured ^{for site C} was 1.28 ± 0.02 mm/sec, which remained constant but fractional area

decreased with increase in Se concentration upto $x=0.10$. This value indicates the FeSb_2 compound phase formation in the sample.

Also two distinct magnetic sites A and B were observed in the spectra as shown in Figure 1. The magnetic field at the site B and the isomer shift value compares well with the formation of Fe_7Se_8 ferrimagnetic phase whose Neel temperature is 473 K. The temperature variation Mossbauer study also indicated the vanishing of B site at 473K. But the Magnetic site A vanished at 573K. The field at Magnetic site A is understood to be due to the sp-d exchange interaction between Se and Fe atoms. This field remained almost constant at all concentrations of Se but the area under the curve of Mossbauer peaks showed increase with increase in Se concentration. However from Hall measurements it was observed that as the Se concentration is increased the charge carrier densities also increases gradually upto $X=0.10$. Hence the introduction of Se brings the conduction electrons into the system and so one can assume that there can be long range Fe-Fe interaction. This type interaction is well known to be RKKY type of interaction. At further higher concentration of Se, Sb_2Se_3 and SbSe compound formation becomes highly probable and Se does not seem to be available for Fe for magnetic sites to form. Hence the field vanishes at $x=0.20$.

On introduction of Se concentration of $X=0.20$ and 0.30 it was observed that there were two quadrupole sites C and D of values 1.45 ± 0.02 mm/sec and 0.51 ± 0.02 mm/sec respectively. Fig 2. shows typical Mossbauer spectra of them. The D sites indicates the formation of Sb_2Se_3 phase from the earlier reported

value. Also the XRD spectra showed clear peaks of Sb_2Se_3 phase with the residual Se and Sb peaks. Hall study revealed that charge carrier density reduced substantially at higher concentration of Se and the value of charge carrier density clearly matched with our earlier study of Sb_2Se_3 phase. Thus one can conclude that at higher concentrations of Se the probability of Sb_2Se_3 compound phase formation increases.

Chapter IV deals with EFG Studies in dilute Fe: Sb_2Se_3 system. Mossbauer study was done at temperatures 297K, 373 K, 473K and 573K. The best fit resulted into quadrupole splitting of 0.51 ± 0.02 mm/sec at room temperature. The XRD spectra of the sample confirmed the formation of Sb_2Se_3 phase in the system. XRD spectra also show residual Se peaks. But the possible nonmagnetic phases like FeSe, FeSe_2 , FeSb_2 , FeSb were absent. Also the reported Quadrupole Splitting (QS) and Isomer Shift (IS) of the above compound phases do not match with our present evaluated parameters of $\text{Fe}_{0.002}(\text{Sb}_2\text{Se}_3)$ sample. Thus it can be concluded that the Sb_2Se_3 is the only compound phase found in the sample.

On increasing the temperature of the sample from 300 K to 573K the quadrupole splitting ~~was~~ ^{was} observed to be constant ^W Whereas the isomer shift was found to be gradually decreasing. The IS values indicated that the Fe is in 3+ charge state. Hence Fe can be expected to substitute for Sb in the Sb_2Se_3 . But as the quadrupole splitting is not dependent on temperature it is possible that Fe may be sitting in the interstitial position. The small ionic radius of Fe^{3+} (0.60 Å) also supports this view. The experimental data for the isomer shift of ^{57}Fe in Sb_2Se_3 system yields a

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slope of -5.73×10^{-4} mm/sec/K for the temperature range of 298K to 597K, which is different from that of the ^{57}Fe SOD value. Since the thermal expansion of the lattice can only decrease the S-electron density at the probe nucleus, the observed strong dependence must be understood in terms of change with temperature of the character of the occupied valency states.

Chapter V deals with the radiation induced damage study in Rhodium (Rh) metal. This study is made to see the defects produced in the cubic metal matrix of the Rh by heavy ion irradiation. The ~~experiment~~^{irradiation} was done at Liq N₂ temperature. The defects were studied by using the highly sensitive Time Differential Perturbed Angular Correlation technique (TDPAC). Electric Field Gradient (EFG) studies in metals using PAC technique is extensively done earlier to determine the defects in metals, the lattice parameters and effect of conduction electrons in the system. In this chapter, we discuss the radiation damage produced by implantation technique and isochronal annealing study done using TDPAC technique. A high purity Rh metal of 1cm X 1cm and thickness of 120 micron was chosen as target material and as a sample to study. A beam of ^{12}C with an energy of 69MeV was used to irradiate the target material to produce a nuclear reaction $^{103}\text{Rh} (^{12}\text{C}, p 3n)^{111}\text{Sn}$. ^{111}Sn ($\tau_{1/2} = 35\text{min}$) so produced decayed via electron capture to ^{111}In which was used as TDPAC probe for the study. Nuclear reaction cross section was simulated first using cascade programme available. The Experiment was performed at the Nuclear Science Centre, New Delhi. The Nuclear reaction and implantation was carried out at Liq N₂ temperature and a few

PAC studies were also done at Liq N₂ temperatures. Isochronal annealing was done on the irradiated sample. The target material was then annealed at room temperature for 15 minutes and studied again at 77 K. Similar procedure of annealing was followed at 673 K, 1073K and 1473K temperatures also. The least square fitted spectra revealed two quadrupole interaction frequencies, one low frequency and another very high value of quadrupole frequency. The higher frequency remained constant for all temperatures and this was assigned to In probe ions getting trapped at Rh-C complexes. The lower frequency disappeared at RT annealing and it was assigned to In trapped at vacancies.

Future Scope

Introduction

Sb-Se system doped with Fe in dilute quantity seems to be a very promising DMS material as discussed in Chapter 3. We have proposed the sp-d exchange interaction and RKKY conduction electron mechanism for the magnetic behaviour in the system. To get a better handle on this system we wish to characterize and get detailed knowledge of the physical mechanisms that are taking place in this system. For this purpose, it is valuable to study the transferred magnetic hyperfine fields at the nuclei of Sb and Se ions. The best way to do this is to implant suitable radioactive ions at these sites and study the magnetic interactions at the nuclei of these ions through TDPAC/ online Mössbauer Spectroscopy. TDPAC and Mössbauer Spectroscopy are excellent local probe techniques and give information about the immediate neighbourhood of the probe atoms. Hence they are the most suitable techniques for the such studies. In order to supplement these studies and get information on the lattice location of the magnetic ions the Emission Channeling (EC) technique can also be employed .



Experiment

In order to observe the hyperfine interaction at the Se site, we intend to implant 57hour $^{77}\text{Br} \rightarrow ^{77}\text{Se}$. It has been shown recently, that Br substitutes Se in ternary semiconductors and that the local magnetic hyperfine field can be probed at the 250 keV state of ^{77}Se by TDPAC. In order to have a case where the probe substitutes Se without any doubt, 7.1hour $^{73}\text{Se} \rightarrow ^{73}\text{As}$ shall be exploited for a TDPAC study. This isotope with good yield has proven to be suitable for TDPAC studies at ISOLDE, CERN, Geneva, Switzerland. For such short lived isotopes the online radioactive ion beam is needed and is available at CERN, Geneva.

As a probe for the Sb-site, 38hour $^{119}\text{Sb} \rightarrow ^{119}\text{Sn}$ can be used for online Mossbauer Spectroscopy and $^{116\text{m}}\text{Sb} \rightarrow ^{116}\text{Sn}$ (5-, 370ns), 60.4m for TDPAC studies. Mossbauer spectroscopy has the advantage, that magnetic splitting can be ^{easily} disentangled from quadrupolar interaction caused by electric field gradients that can be present at non cubic lattice sites. Hence we would also like to do the in beam conversion electron Mossbauer spectroscopy by implanting ^{57}Mn into the Fe site of the sample

The experiment was proposed for doing at ISOLDE, CERN, Geneva and our proposal has been accepted. We are waiting for the beam time allotment for our experiment.