7 APPENDIX

TDPAC MEASUREMENTS IN TEXTURED HCP SAMPLES

Introduction

The texturing effects, if present predominantly in a hcp sample, could cause ambiguities regarding the hyperfine interaction parameters as experienced by probe nuclei viz., quadrupole frequency and asymmetry parameter. The former could be the case if the intensity of the fundamental component of the quadrupole frequency corresponding to substitutional and defect-free fraction of probe nuclei is almost zero. The latter is possible if the intensities of the second and third harmonic components of the quadrupole frequency are almost zero which in general would give an impression that the probe nuclei are occupying an axially asymmetric charge environment. These effects have to be seriously considered in textured samples in which defects are introduced by any conventional method such as light or heavy ion implantation, quenching or due to the introduction of extrinsic defects such as impurities. For example, the Fourier transform of the time dependent anisotropy spectra R(t) obtained in the helium implanted condition of ZrHf as shown in Figure 5.3a of this thesis, indicates the presence of a predominant peak at the second harmonic of the quadrupole frequency as experienced by the probe atoms. The intensity of the fundamental component of the quadrupole frequency is almost negligible. This implies the texturing of the sample. All TDPAC measurements in such a sample following each step of any heat treatment, in general has to be necessarily carried out in a unique position of the sample with respect to detector geometry. This is required to be done, since the anisotropy spectra and hence its Fourier transform would be varying in terms of the intensities of the frequency components corresponding to probe nuclei occupying sites in oriented microcrystallites.
Considering the case of a hcp sample, if it is well homogenised and in the form of a spherical lump, such a sample, would most likely be perfectly polycrystalline with a random orientation of microcrystallites. Therefore the probe nuclei occupying the substitutional sites with defect free environment will have the ideal intensity ratio of the frequency component corresponding to the concerned matrix. Even such samples, under certain conditions seem to have texturing behaviour. For example, the preferred orientation of microcrystallites has been reported in In metal just below the melting point by \(^{111}\text{Cd TDPAC measurements}\) [1]. Rasera et al [2] have proposed a method of rotation of the sample around an axis perpendicular to the plane of the detectors to get a polycrystalline analogue of the texturised samples.

If the probe nuclei occupy axially symmetric and non-cubic sites in a sample in which microcrystallites are randomly oriented, the values of the amplitudes \(S_{km}\) that appear in equation (36) of Chapter 1 are given as [3],

\[
S_{km} = \sum_{m,m_1} \begin{pmatrix} I & I & 2 \\ m_1 & -m & m-m_1 \end{pmatrix}
\]

where \(2 | m^2 - m_1^2 | = n\) for half-integer \(I\). The values of \(S_{km}\) as given above in terms of wigner 3j coefficients are tabulated for the isomeric state of different spin as given in reference [3]. In the case of a textured sample these \(S_{km}\) have to be taken as free parameters while doing the data analysis.

Here it is aimed at experimenting the ways to get the quadrupole interaction parameters analogous to polycrystalline sample with random microcrystallites and hence resolve the hyperfine interaction parameters of probe nuclei associated with defects, if any, occurring in the sample. Here a method is proposed to obtain the polycrystalline analogue of the textured sample.
Experimental Details

The effect of texturing is illustrated by the results of the present TDPAC measurements on Hf foil. The anisotropy spectra R(t) corresponding to the measurements in the Hf foil sample of area 1 cm x 0.1 cm and 25 µm thickness with two different positions of the sample viz., the flat surface of the sample lies parallel to the axis connecting START and STOP detectors which are collinear, and the flat surface of the sample lying perpendicular to the latter are shown in figure 7.1. In the following text this will be referred to as geometry I and II respectively. The R(t) spectra and their fourier transform (Figure 5.18) are found to be different, showing this dependence on the position of the sample with respect to detector geometry. The Fourier spectra of the above show that the ratio of the components of the frequency is 1:2:3, but their intensity ratio is not in 3:2:1. This would in principle imply the selective orientation of the principal axis of the electric field gradient tensor and hence the orientation of microcrystallites of the sample. The XRD pattern of the Hf foil sample as shown in Figure 7.2 indicates that the microcrystallites of the sample have got textured along (0002) direction as the intensity of this peak is much higher than the peak at (10\bar{1}1). This is due to the reasoning that in a sample with random orientation of microcrystallites the maximum intense peak is expected to occur at (10\bar{1}1) as given by ICDD (International Centre for Diffraction Data). This implies that the microcrystallites in α-Hf get preferentially oriented along (0002) direction due to thermo-mechanical treatments of the samples.

Results and Discussion

The measurements are done in the following way in a Hf foil to average out the effects of texturing and hence get the analogue of the perfectly polycrystalline sample.

(1) In this new method proposed for getting the polycrystalline analogue of the textured sample, it is proceeded as follows. The experimental anisotropy spectra obtained in the above mentioned two geometries, I & II of the sample, were averaged and the resultant
Fig. 7.1. Time dependent anisotropy spectra $R(t)$ for $^{181}$Ta and the corresponding Fourier transform $P(\omega)$ in Hf foil located at two geometries w.r.t detectors (a) Geometry-I and (b) Geometry-II as mentioned in the text.
anisotropy spectra $R(t)$ was analysed, to derive the hyperfine interaction parameters of probe nuclei in the polycrystalline analogue of the sample. The data analysed $R(t)$ spectra are shown in Figure 7.3. The Fourier transform of the spectra indicates the occurrence of peaks at 290, 580 and 870 MHz with their intensities roughly in the ratio 3:2:1, as indicated in Figure 5.20. The importance of such measurements is that it leads to a predominant intensity of the fundamental component of the quadrupole frequency. The averaged spectrum as compared to the individual spectrum obtained in the each geometry of the sample, as shown in Figure 7.3, shows that the former leads to results analogous to the perfect polycrystalline sample.

(2) Secondly, measurements were done by rotating the sample along an axis perpendicular to the plane of the detectors. The time dependent anisotropy spectra $R(t)$ and its Fourier transform for $^{181}$Ta in Hf sample with and without rotation are shown in Figure 7.4. It has been found from the Fourier transform of the $R(t)$ spectra obtained in the above cases that the ratio of the intensity of the quadrupole frequency components tends to approach that of the polycrystalline sample. The probe nuclei are found to experience a quadrupole frequency $< \nu_Q > = 290 \pm 4$ MHz and an asymmetry parameter $\eta_0 = 0.08$. By rotating the sample about an axis perpendicular to the plane of the detector, we could average the selective orientations of microcrystallites of the sample.

Conclusions

The measurements, conducted either by averaging the $R(t)$ obtained at different geometries of the sample or by rotating the sample at a constant speed, are found to result in the polycrystalline analogue of the textured samples. These measurements would help to find out the hyperfine interaction parameters of the defect(s) related fraction(s) occurring in such hcp samples without any ambiguity as it is possible delineate the fraction of probe nuclei that are defect free in the matrix.
Fig. 7.3. Time dependent anisotropy spectra $R(t)$ for $^{181}$Ta and its Fourier transform $P(\omega)$ in Hf foil. The $R(t)$ is the average of the experimental spectra obtained at two geometries of the Hf sample as explained in the text.
Fig. 7.4. Time dependent anisotropy spectra $R(t)$ for $^{181}$Ta and its Fourier transform $P(\omega)$ in Hf foil (a) with out sample rotation and (b) with a constant speed of rotation $\approx 2\text{rps}$. 
References


LIST OF PUBLICATIONS

Journal

1. TDPAC studies of Hf doped YBCO

   R. Govindaraj, M. Premila, Padma Gopalan and K. P. Gopinathan

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4. TDPAC measurements in textured hcp smaples

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Conferences

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