Defects identification in $FeTiO_3$ using positron annihilation technique

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Abstract. Positron lifetime measurements are conducted in metal oxide material with hexagonal structure, in the temperature range from 30 K to 500 K. The analysis of the positron lifetime spectra is best fitted to two lifetime components. The positron lifetime in a defect-free region ranges from 177 ps to 186 ps in the temperature range. The second lifetime components of localized positron range from 350 ps to 462 ps in the temperature range. The second positron lifetime components are attributed to positron trapping at vacancy complexes. A close analysis of second lifetime components coupled with the fact that annihilation ratios are greater than threshold value of 1.4 suggests a formation of vacancy clusters in the temperature range from about 250 K to 500 K.

1. Introduction

Iron titanium oxide ($FeTiO_3$) has drawn much attention because of titanium which finds its application in the manufacturing of aircrafts parts and in many other applications. Therefore it is extremely crucial that reliability be maintained in order to minimize or control defect density in the material.

Positron annihilation technique (PAT) in the past decade has become a reliable tool in which positrons are used as probes for various forms of open volume defects. This technique has been employed extensively for defect investigations in semiconductors and metals experiments but little research has been conducted in metal oxide perovskite structures.

Positrons in a sample can have several states associated with different kinds of defects each of which gives a characteristic lifetime. The positron lifetime is given by a reciprocal of the annihilation rate given by

$$\lambda = \pi r_o^2 c \int dr \left|\psi_+(r)\right|^2 n(r) \gamma [illegible](r) \quad (1)$$

where r_o is the classical radius of the electron, $\psi_+(r)$ is the positron wavefunction, *n(r)* the electron density and $\gamma[n(r)]$ the enhancement factor [1]

The objective of the present work is to determine the vacancy complexes as a function of temperature using annihilation rates at various temperature points.

2. Experiment

$FeTiO_3$ samples of thickness 2 mm and a diameter of 6 mm were prepared from $FeTiO_3$ powder using a standard pelletizing technique. A $^{22}NaCl$ source of activity of 20 μCi sealed by two electron-welded aluminium foils of thickness 7 μm, was placed between two equal FeTiO3 samples in a standard sandwich arrangement. The sample chamber was kept at 10^{-4} Torr pressure.

The lifetime measurement was carried out using standard coincidence setup by employing two fast scintillator detectors (XP2020) for the start (1275 keV) and stop (511 keV) signals. The time resolution of the positron lifetime coincidence-setup used for the measurements was of the order of 280 ps at FWHM. The measurements were performed between 30 K and 500 K. The sample temperature was monitored through thermocouples and was stabilized to ± 1 K. About 10^6 counts for each spectrum were collected.

Source correction was carried out because 10 – 15 % annihilations take place in the source material and in the encasing foil. Aluminium foil has a positron lifetime component of 210 ps and the intensity was calculated using Bertolucci-Zappa formula given by [2]

$$I_{foil}(\%) = k = 0.324 Z^{0.93} \times \Omega^{\alpha} \qquad (2)$$

where Ω is the surface density of the foil in mg cm^{-3} and $\alpha = 3.45 \times Z^{0.4}$.

3. Results and discussion

Positron annihilation ratios, λ_d / λ_b, shown in figure 1, at various temperature points are certainly above a threshold of 1.5. This is an indicates a reliable positron lifetime values

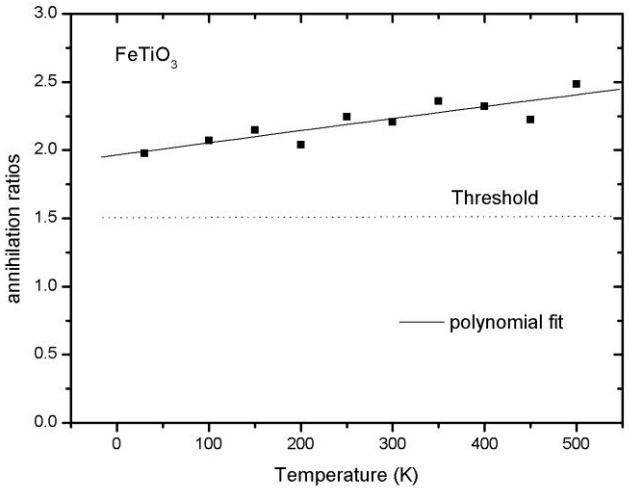

Figure 1. Annihilation ratios indicating a clear separation of two positron lifetime components.

The annihilations in the bulk, i.e. the defect-free region, and in vacancies are shown in figure 2. The average positron lifetime, τ_{av}, which represents a statistically accurate parameter is shown in figure 3. The short lifetime of 185 ps is typically attributed to the free annihilation of positrons. The observed first positron lifetime component agrees very well with a theoretically calculated free positron lifetime of 188.05 ps in the bulk. The second positron lifetime component arises from

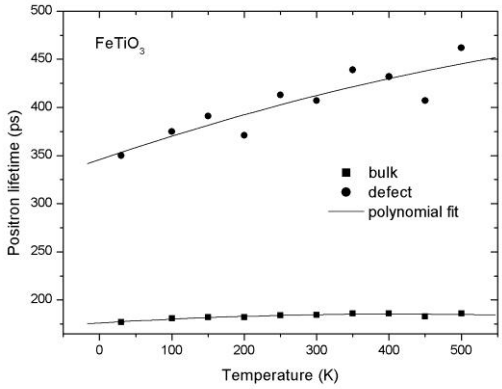

Figure 2. First and second positron lifetime components

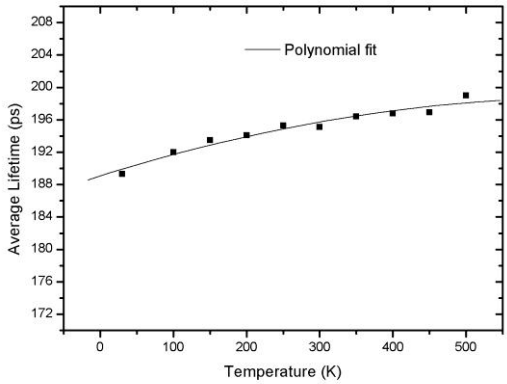

Figure 3. Average positron lifetime as a function of temperature

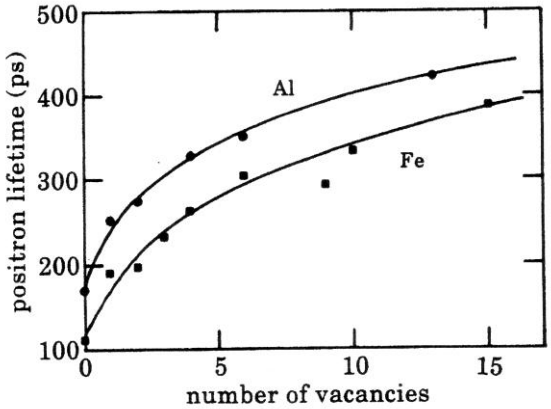

Figure 4. Number of vacancies as a function of positron lifetime in Fe [5]

annihilation of positrons at defect sites. In this case it is believed to be coming from annihilation of positrons at vacancy complexes possibly formed as clusters. Average positron lifetime of 196.18 ps compares very well with a theoretically value of 197.17 ps in which a deviation is within 0.5 %.

Fe free-atom vacancy has annihilation rate of 4.8260 /ns which was calculated using Local Density Approximation (LDA) in which the enhancement is calculated from Boronski – Nieminen [3]. Ti free-atom vacancy calculated by Mizuno [4] has an annihilation rate of 4.975 /ns which is higher than that for Fe. This is an indication of uneven distribution of positron wavefunctions at various metal vacancies. The observed average positron lifetime value of 196.18 ps in figure 3 suggest a divacancy type of defect at Fe-ion sites. This is confirmed by a theoretically calculated [5] value as shown in figure 4.

4. Conclusion

Positron annihilation technique clearly reveals the temperature dependent of vacancy complexes. The variation of free-atom annihilation rates gives a contribution of individual metal ion in terms of the distribution of positron wavefunctions in the vicinity of individual metal ions.

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