1. INTRODUCTION

The present investigations on the characteristics of residence sites taken up by $^{19}$F ions implanted in carbon allotropes were prompted by our finding that in the case of diamond hosts the $^{19}$F implants take up two characteristic sites$^{1,2}$. One site, which we designate the principal site, was taken up by about 50% of the implants and had an electric field gradient with a quadrupole coupling frequency at the $^{19}$F nucleus of $\nu_q = 58$ MHz. This site was of well-defined single crystal character and had a very low $\text{eg}$ distribution parameter ($\delta$) and asymmetry ($\eta$). The other site, with a much lower $\nu_q$, was a diffuse site (large $\delta, \eta$), suggesting a more polycrystalline or amorphous environment in the vicinity of the probe ion, or oscillations of the probe ion within its site. These results were in qualitative agreement with those found for $^{19}$F in Ge and Si hosts by Bonde-Nielsen et al$^{3,4}$. In order to illicit further information on the implant sites, ie whether crystalline or amorphous, and on the nature of the F-C bonding, we have extended our study to $^{19}$F implants in the carbon allotropes, single and polycrystalline graphite and vitreous and colloidal carbon. The $^{19}$F ions were excited in the $^{19}$F(p,p')$^{19}$F* reaction and recoil implanted in the host samples. In-beam Time Differential Perturbed Angular Distribution (TDPAD) measurements on the 197 keV $\gamma$-ray from the $^{19}$F $^{5/2}^+$ state ($Q = .010b, r = 128$ns) were used to investigate the electric field gradients at the sites of the $^{19}$F implants.

2 EXPERIMENTAL DETAILS

The host samples for the implants consisted of thin wafers of the carbon allotropes, with 30$\mu$g cm$^{-2}$ of CaF$_2$ evaporated on their surface. The graphite and vitreous carbon samples were compressed discs, 20 mm in diameter and 3 mm thick. The colloidal aquadag sample was of similar size, formed from a 20 year sediment of colloidal carbon in alcohol. A pulsed proton beam of 4 MeV energy was provided by the University of the Witwatersrand tandem Van de Graaff accelerator. The beam had a repetition period of 500 ns and a pulse width of 3 ns. Standard fast-slow circuitry was used to generate delayed time spectra of the $^{19}$F 197 keV $\gamma$-rays. The start signals were obtained from two 2.5cm $\times$ 4.0 cm NaI(Tl) detectors positioned at 0° and 90° to the beam direction, and the stop signal from a fast plastic "halo" detector positioned 2m upstream from the target. For each host matrix data were collected in a series of runs.
in which the positions of the two NaI detectors were interchanged. To obviate electronic
instabilities and amplifier drift, each run was limited to be of about two hour duration.
The experimental spin rotation spectra were generated from the ratio
\[ R_{\text{exp}}(t) = \frac{2}{N(0^\circ, t) - N(90^\circ, t)} \]
where \( N(0^\circ, t) \) and \( N(90^\circ, t) \) are the normalized count rates at 0° and 90° for a given detector.

3. Analysis

The theoretical spin rotation function is
\[ R_{\text{th}}(t) = \frac{2}{W(\theta, \varphi, 0^\circ, t) - W(\theta, \varphi, 90^\circ, t)} \cdot \sum_{i} f_i \cdot \left[ \sum_{k_1, k_2} A_{k_1 k_2}^{\text{eff}} f(\sigma, \delta, \eta_i \omega_{0i}; t) \right] \] (1)
and for a polycrystalline host
\[ R_{\text{th}}^{\text{pc}}(t) = \sum_{i} f_i A_{k_1 k_2}^{\text{eff}} f(\sigma, \delta, \eta_i \omega_{0i}; t) \] (2)
where \( f_i \) is the fraction of implants at the site \( i \), and \( f(\sigma, \delta, \eta) \) is an amplitude attenuation factor that takes account of the finite time resolution of the system \( \sigma \) and a spread \( \delta \) of the electric field gradient at site \( i \). \( A_{k_1 k_2}^{\text{eff}} \) and \( G_{k_1 k_2}^{\text{eff}} \) are effective anisotropy and perturbation coefficients defined in ref. (1). The frequency \( \omega_{0i} \) is related to the quadrupole coupling constant of the nuclear quadrupole moment \( Q \) with the efg at the implant site,
\[ \nu_{0i} = \frac{eQV_Z}{\hbar}. \]
For a nuclear spin \( I = \frac{3}{2} \), \( \nu_{0i} = \frac{10}{3\pi} \omega_{0i} \). The diagonalized efg tensor has components \( |V_{zz}^i| \geq |V_{yy}^i| \geq |V_{xx}^i| \) and the efg asymmetry parameter is defined by
\[ \eta = (V_{xx} - V_{yy})/V_{zz}. \]

The data analysis was carried out by first generating the experimental \( R(t) \) spectra
for each gamma detector from the appropriate normalized delayed time spectra. The
\( R_{\text{exp}}(t) \) is fitted with the theoretical function (1) or (2) with the aid of the CERN code
MINUIT6 to yield for each implant site \( i \) the parameters \( f_i, \omega_{0i}, \eta_i \) and \( \delta_i \).
RESULTS

The experimental $R(t)$ spectra and the theoretical fits for the four carbon allotrope hosts are displayed in Figs. 1(a)-(d). Table 1 lists the efg parameters determined from the analysis together with the type of model function used. For comparison the efg parameters at $^{19}$F implant sites in diamond IIb$^{1,2}$ are also listed.

Fig 1.(a)-(d) Experimental $R(t)$ spectra for single and poly-crystalline graphite and vitreous and colloidal carbon. The solid lines are a fit to the data using equations (1) or (2) above.
Table 1. Efg data for $^{19}\text{F}$ in various carbon allotropes

<table>
<thead>
<tr>
<th>Host sample</th>
<th>efg</th>
<th>Site</th>
<th>f</th>
<th>$\nu_Q$</th>
<th>$\delta$</th>
<th>$\eta$</th>
<th>Theory type</th>
<th>$\chi^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single crystal</td>
<td>fixed but measured</td>
<td>1</td>
<td>36(2)</td>
<td>57(2)</td>
<td>3(1)</td>
<td>1(1)</td>
<td>single</td>
<td>.70</td>
</tr>
<tr>
<td>Graphite</td>
<td></td>
<td></td>
<td>2</td>
<td>8(1)</td>
<td>6(1)</td>
<td>30(3)</td>
<td>crystal</td>
<td></td>
</tr>
<tr>
<td>Polycrystalline</td>
<td>-</td>
<td>1</td>
<td>75(3)</td>
<td>57(2)</td>
<td>5(1)</td>
<td>10(1)</td>
<td>poly-</td>
<td>.70</td>
</tr>
<tr>
<td>Graphite</td>
<td>-</td>
<td>2</td>
<td>25(2)</td>
<td>14(1)</td>
<td>10(1)</td>
<td>16(2)</td>
<td>crystalline</td>
<td></td>
</tr>
<tr>
<td>Vitreous</td>
<td>-</td>
<td>1</td>
<td>66(3)</td>
<td>56(2)</td>
<td>5(1)</td>
<td>4(1)</td>
<td>poly-</td>
<td>.89</td>
</tr>
<tr>
<td>Carbon</td>
<td>-</td>
<td>2</td>
<td>34(2)</td>
<td>17(2)</td>
<td>38(3)</td>
<td>2(1)</td>
<td>crystalline</td>
<td></td>
</tr>
<tr>
<td>Colloidal carbon (Aquadag)</td>
<td>fixed but measured</td>
<td>1</td>
<td>70(3)</td>
<td>57(2)</td>
<td>5(1)</td>
<td>4(1)</td>
<td>single</td>
<td>.91</td>
</tr>
<tr>
<td>Diamond IIb</td>
<td>&lt; 111 &gt;</td>
<td>2</td>
<td>32(2)</td>
<td>18(1)</td>
<td>31(3)</td>
<td>0</td>
<td>crystal</td>
<td>.70</td>
</tr>
</tbody>
</table>

A comparison with theoretically simulated $R(t)$ spectra for various host lattice-detector geometries and efg principal axis directions shows that the experimental $R(t)$ spectrum for single crystalline graphite and that for the colloidal carbon (aquadag) closely resembles that due to a cubic single crystal host in which the principal axis of the efg is along a $< 110 >$ direction. The experimental spectra were therefore analysed in terms of such a model function. However graphite crystallizes in a hexagonal, layer-type lattice of ABAB stacking. Table 1 reflects the efg principal axis as “fixed but not determined”. Attempts to fit the spectra with a polycrystalline model function resulted in a $\chi^2$ at least 40% larger, but with efg parameters close to the listed values.

As in the case with diamond hosts, the analysis yielded two residence sites for the $^{19}\text{F}$ implants, with unique electric field gradients. However in the single crystal graphite, only 36% of the incident $^{19}\text{F}$ ions was found to take up the principal site which was of low ($\eta, \delta$) and an efg quadrupole coupling frequency of $\nu_Q = 57(2)$ MHz. A much lower fraction (8%) took up a very diffuse ($\delta = 30\%$) low frequency site (6 MHz). The Laue X-ray pattern of the polycrystalline graphite sample showed the ring...
pattern expected of such a matrix, while that for vitreous carbon showed no pattern at all, which is expected of amorphous material. The TDPAD spectra for these two hosts were analysed in terms of polycrystalline fit functions which yielded two sites for the $^{19}$F implants in both samples. The characteristics of the principal component ($\nu_q = 57$ MHz) were found to be in very good agreement with those found for the single crystal graphite (and diamond); the second component has a slightly higher $\nu_q$ ($\approx 15$ MHz) and a distribution parameter $\delta$ of 10% for graphite and 38% for vitreous carbon.

Lastly we come to the data for aquadag, which we hesitate to label "single crystal" or "polycrystalline". The aquadag target was formed from a 20 year sediment of fine graphite particles in a colloidal suspension in alcohol. The sediment was hard enough to be just machinable. With a polycrystalline theoretical function, the TDPAD spectrum analyses to three fractions. However, the quadrupole coupling frequency of one fraction is close to that expected for the second harmonic of the principal fraction ($\nu_q = 57$ MHz) and so we do not interpret this as due to implants at a unique site. The best fit to the data was obtained with a single crystal model function, with the principal axis of the efg parallel to the <110> crystal direction, and two sites for the implants. If crystalline, we are unable at this stage to determine the symmetry, and hence, for this host also, we list the efg axis as "fixed but not determined".

5. CONCLUSIONS

Our results show that in the carbon allotropes investigated, the $^{19}$F implants take up two distinct residence sites. The principal site has a quadrupole coupling frequency of $\nu_q = 57$ MHz, and is taken up by a larger fraction of the implants. The single crystal graphite has a secondary site of extremely low mean quadrupole coupling frequency (6 MHz), which is taken up by only 8% of the implants. In the other samples the second site has $\nu_q = 15$ MHz, and is taken up by 20% - 30% of the implants. Our results for the carbon allotropes are consistent with those for diamonds$^2$ where we found two resident sites for the $^{19}$F implants with efg parameters almost identical to those determined in the present work. These results are in qualitative agreement with those found for Ge and Si hosts$^{3,4}$. The $^{19}$F implants also take up two unique sites in these samples. One is well-defined, and the other more diffuse, and the efg parameters at the sites in crystalline and amorphous Si are very similar.
The Laue pattern for the graphite was the ring pattern expected for polycrystalline material, and the TDPAD analysis was consistent with this. For the vitreous carbon and aquadag samples neither dot nor ring Laue patterns were observed, suggesting that these were amorphous materials. The \( R(t) \) spectrum for the \( ^{19}\text{F} \) implants in vitreous carbon was similar to that for polycrystalline graphite. The principal residence site for the implants appears clearly polycrystalline, which is not surprising as these materials, like the metallic glasses, have short range order in their structure, and the TDPAD technique is sensing this. The larger distribution in the efg's (\( \delta = 38\% \)) corresponding to the second implant site indicates that this site is more amorphous in glassy carbon than in graphite. The aquadag spectrum was best fitted with a single crystal model function, although a function for a polycrystalline host also gave a reasonable fit. We tentatively reason that in aquadag we have a structure that is sufficiently better ordered than a random aggregation of microcrystals so that it is starting to acquire single crystal features.

Cluster calculations performed by our collaborators [Lo 88, Ve 88] showed total energy minima for \( ^{19}\text{F} \) in diamond at off-centre positions at the tetrahedral interstitial site and the substitutional site [Lo 88]. The TDPAD frequencies calculated from the charge distributions in the cluster for the two sites strongly suggested the tetrahedral interstitial site for the first (principal) fraction and the substitutional site for the second (diffuse) fraction. In addition the shape of the potential well that the \( ^{19}\text{F} \) finds itself in was sharp for the first site and very broad for the second. Finally, the depth of the potential well that \( ^{19}\text{F} \) in the tetrahedral site finds itself in correlates well with the C-F bond energy of 5.08 eV. Also the temperature dependence of the fractional population of the \( ^{19}\text{F} \) at the substitutional site has a thermal activation energy of .14 eV. This corresponds closely with the cluster calculation of the depth of the potential well for \( ^{19}\text{F} \) at this site. These strong correlations between experiment and theoretical cluster calculations argue for the correctness of these site assignments in the case of diamond. The cluster calculations also showed that at both sites the \( ^{19}\text{F} \) became slightly charged, indicating the existence of chemical effects in the host-impurity interactions.

In addition, the principal efg component at the \( ^{19}\text{F} \) site has \( \nu_e \approx 59 \) MHz for all carbon allotropes, which is in very good agreement with the frequency determined for \( ^{19}\text{F} \) sites in \( \text{CF}_4 \), \( (\text{C}_2\text{F}_4)_n \) and various halo methanes. Together with the
results from the cluster calculations, this strongly suggests that the hyperfine interaction resulting in this quadrupole coupling is due to a fairly undisturbed C-F bond.

In summary, the present measurements taken with our previous ones on diamond show that the residence sites of $^{19}$F ions in diamonds and the carbon allotropes are locally very similar. This argues that chemical effects are playing a major role in determining the charge distributions surrounding the probe ions. It is reasoned that the efg for semiconductors and insulators is substantially more determined by the electronic charge distributions than is the efg in metals. It seems that in these cases the formation of strong chemical bonds may override first order information on the residence sites, rendering the efg for $^{19}$F in all allotropic forms of carbon locally very similar.

ACKNOWLEDGEMENTS

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REFERENCES


Fig. 5.3 (a)-(d) For archival purposes - Fourier transforms of the experimental $R(t)$ spectra for $^{19}$F implanted in single and poly-crystalline graphite and vitreous and colloidal carbon.
CHAPTER 6.  
MSR WORK IN DIAMOND

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Introduction

This chapter reports on a Muon Spin Rotation (MSR) experiment performed on diamond [Od 86a, Od 86, Od 88]. This experiment measured the absolute signs of the Mu* hyperfine parameters and the parameters describing the transition from Mu to Mu* in a single Arrhenius law. Conclusions concerning the site assignment and the dynamical behaviour of Mu* in diamond are made from our results. (Two of the papers reporting on these experiments, but written by the first author thereon, are included in the Appendix for completeness.)

Muonium and Hydrogen

When a positive muon, which is regarded as a light isotope of the proton, stops in matter, it may pick up an electron to form the $(\mu^+-e^-)$ paramagnetic bound state, muonium. Muonium turns out to have very similar chemical properties to the hydrogen atom, since the reduced masses of both species are nearly equal (Table 6.1). Consequently, the behaviour of the muonium impurity in matter is regarded as analogous to that of a light hydrogen impurity.

Table 6.1 Comparison of properties of Muonium and Hydrogen

<table>
<thead>
<tr>
<th></th>
<th>Rest Mass</th>
<th>Reduced Mass</th>
<th>Bohr Radius</th>
<th>Ionization Energy</th>
<th>Hyperfine Frequency</th>
</tr>
</thead>
<tbody>
<tr>
<td>Muonium</td>
<td>.1131 m_H</td>
<td>.9952 $\mu_H$</td>
<td>1.0044 $a_0$H</td>
<td>.9956 $E_H$</td>
<td>3.1423 $\omega_H$</td>
</tr>
</tbody>
</table>

Experimental

The present experiment was performed at the Swiss Institute of Nuclear research (SIN) near Zürich in Switzerland. A 590 MeV beam of protons at a current of $\approx 100 \mu$A is directed onto a rotating beryllium target. The resulting proton-proton collisions (above the pion production threshold) lead to the copious production of pions via the reaction

\[ p + p \rightarrow p + n + \pi^+. \]
If the pion ($r = 26$ ns) stops in the beryllium target near the surface and decays, both the lab and centre of mass energies of the muon decay product are equal to 4.1 MeV. This is the lowest energy muon produced; it has a momentum of 28 MeV/c and is termed a “surface” muon. Its range in diamond is approximately 400 µm. The non-conservation of parity in the pion decay results in a backward spin polarization for the muon. Polarized muons are magnetic hyperfine probes due to the fact that their spin orientation can be detected experimentally. This is possible since the decay positron is emitted after the mean lifetime of the muon of 2.2 µs in a direction correlated to the muon spin axis [Fr 74]. Detection of the muon spin axis is achieved by detection of the decay positron. In time differential MSR one follows the time dependence of the spins of an ensemble of polarized muons, implanted one at a time into the sample. Fig. 6.1 shows schematically the apparatus used for accumulating a time histogram describing the decay of the muon and the direction of its spin axis.

![Fig. 6.1 Time Differential MSR apparatus.](image)

Though it is possible to use a pulsed muon beam, in this case it was not necessary as there were no experimental limitations due to count rate. Instead the muon beam current was adjusted by use of a lead collimator so that the average muon arrival rate
was about one muon per four muon lifetimes. Plastic scintillator detectors generated the start and stop signals, from the muon arrival and positron detection respectively. These signals were processed by a Time to Amplitude Converter (TAC) to generate the time histograms, in a similar way to that described for the TDPAD case in Chapter 4. An electronic "data gate" and muon pile-up rejection ensured that only one muon was present in the diamond target per event. The muon thus represents an infinitely dilute probe, and there is almost no chance of impurity-impurity interactions, which is also the case for $^{19}$F TDPAD.

The angular distribution of the decay positron from a muon stopped in a solid is described by

$$W(\theta, t) \propto 1 + A \cos(\omega t + \theta)$$

where $\theta$ is the angle between the initial spin polarization of the muon and the decay positron, and $\omega$ describes the Larmor precession about an external field. The maximum asymmetry is given by

$$A = P/3$$

after averaging over the $\beta^+$ spectrum energy. Since in practice the muon polarization $P$ is not quite 100%, the total asymmetry in our case was only $A = 20\%$. Note that the dipole symmetry of the muon decay requires the two positron detectors to be 180° apart, whereas in the TDPAD case the quadrupole symmetry of the $^{19}$F* decay requires the two $\gamma$ detectors to be 90° apart.

Ion-solid Considerations

Typical implanted doses are $2 \times 10^6$ muons/cm$^2$, which is about the same as the $^{19}$F recoil implanted dose. The thermalization time of picoseconds is too rapid to allow the muon to become depolarized. During the experimental time window of about 8 $\mu$s however, the muon is highly mobile, in contrast to the $^{19}$F implantation. Since the damage created by the muon is very small (only 15 vacancies per implant, as compared to 490 for $^{19}$F), and since its ballistic energy transfer is limited by its small mass, it stops quite far (19 $\mu$m for $\mu^+$ in diamond [Pa 88]) from its own defects. As a result [Pa 88], the damage due to the muon itself plays no role in a MSR experiment (again similarly to the $^{19}$F case). With regard to the effects of its own ionization cloud produced in the stopping, there are $\approx 7$ e$^-$-h pairs per $\AA$ (broken or weakened bonds) in the final
part of the track [Pa 88]. This gives a conduction electron density of 6 times that in copper in the vicinity of the muon. This diffuses away within nanoseconds [Pa 88], much quicker than the electron-hole recombination time, and so there are also no persistent interactions between the muon and its ionization cloud.

Theoretical

The measured parameters in a MSR experiment are analogous to a PAC experiment using magnetic perturbations. The muon "precesses" around the local field with a frequency \( \nu \), amplitude \( A \), depolarization rate \( \lambda \) and phase \( \phi \), all of which are extracted from the time histograms by computer fits in a similar procedure to that described for TDPAD. The model function describing a time histogram is

\[
N(\theta, t) = N \cdot W_\theta(t) \cdot \exp(-t/\tau) + B.
\]

\[
W_\theta(t) = \sum_i A_i \cos(\omega_i t + \phi_i) \cdot e^{-\lambda_i t}.
\]

Apart from the replacement of an electric quadrupole perturbation factor by a magnetic perturbation factor, this expression is identical to that of equation (3.38) in Chapter 3. The index \( i \) runs over the different forms (or sites) of the muon in the diamond target. There were three such sites observed in our experiment, with \( f_i = A_i/A \) giving the fraction of the muon ensemble which populated the site \( i \). The first site (\( f_1 = 20\% \)), arose from the diamagnetic bare muon stopped in the target holder. This was of no interest to us apart from its convenience in calibrating the frequency spectrum. Two other "sites" were also observed. These two paramagnetic sites are the two different types of muonium which had previously been observed in diamond [Ho 82], denoted isotropic muonium (Mu) and anisotropic muonium (Mu*) respectively. Note that the anisotropic form of muonium has a perturbation function \( W_\theta \) that depends on the crystallographic orientation of the diamond. In \(^{19}F\) TDPAD measurements, this is always the case due to the quadrupole interaction.

The spin Hamiltonian describing Mu and Mu* is

\[
H = \hbar A_\perp \vec{I} \cdot \vec{S} + \hbar (A_{||} - A_\perp)(\vec{I} \cdot \hat{n})(\vec{S} \cdot \hat{n}) - g_\mu \mu_B \vec{I} \cdot \vec{B} - g_e \mu_B \vec{S} \cdot \vec{B}.
\]

The \( A \) here are hyperfine constants proportional to the electron spin density at the muon. Their values thus relate directly to specific site models for Mu and Mu*. \( \vec{I} \) and \( \vec{S} \) are the muon and electron spin operators and \( \vec{B} \) is the external field.

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For the isotropic Mu, \( A_\parallel = A_\perp = A = 3771 \text{ MHz} \), which is 83% of the vacuum value. Matching of this experimental value with that of the energetically favoured sites given by cluster calculations has led to the general acceptance of the tetrahedral interstitial site for isotropic Mu [Sa 83]. This site is in fact one of the most open positions in the diamond lattice, and it is not really surprising to find it as the first site at which the \( \mu \) chooses to stop. \(^{10}\text{F}\) readily occupies this site, though it binds chemically to a C atom and so is not at an isotropic position.

For Mu*, the isotropic part of the Hamiltonian's hyperfine coupling constant, \( A_* = (A_\parallel + 2A_\perp)/3 \), has a magnitude of 205.7 MHz. The anisotropic part, \( A_p = (A_\parallel - A_\perp)/3 \), has a magnitude of 186.6 MHz, showing that the anisotropy is extremely marked. The unit vector \( \mathbf{n} \) is found by experiment to be along a \(<111>\) crystallographic axis of diamond.

The Zeeman behaviour of the hydrogen-like energy levels obtained from solving the spin Hamiltonian is shown in Fig. 6.2(a) for isotropic Mu and in Fig. 6.2(b) for anisotropic Mu*. Notice that in the case of the anisotropic muonium, the plotted lines refer to a particular geometry, that is the magnetic field is considered perpendicular to a particular \(<111>\) axis. There are four such equivalent axes, resulting in many more levels than those drawn. Muon polarization "precession" frequencies in magnetic fields are observed for those transitions where the frequency given by the energy level difference is within the time resolution of the detection equipment. Fig. 6.3 shows these "precession" frequencies for Mu and Mu*(90°) as a function of magnetic field.

**Results and Conclusions**

**Previous Experiments**

As already mentioned, both Mu and Mu* had previously been observed in diamond [Ho 82]. The theoretical description of the two forms of Muonium via an isotropic and an anisotropic spin Hamiltonian was satisfactory. The magnitudes of all the hyperfine parameters were known. However, only the relative sign \( A_\parallel/A_\perp < 0 \) was known before the experiment to be described. In addition, although the tetrahedral interstitial site was well established for the isotropic Mu, and although cluster calculations based on
Fig. 6.2(a) Zeeman behaviour of \( \text{Mu} \).

Fig. 6.2(b) Zeeman behaviour of \( \text{Mu}^* \).

this site reproduced the coupling constant fairly well, the site for the anisotropic \( \text{Mu}^* \)