MUON SPIN ROTATION¹

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Introduction

 μ SR spectroscopy uses implanted muons to probe properties of matter at the microscopic level. According to one of the earliest definition of μ SR - appeared on the cover of the first issue of the μ SR Newsletter in 1974 -: " μ SR stands for Muon Spin Relaxation, Rotation, Resonance, Research or what have you". The intention of the mnemonic acronym was to draw attention to the analogy with NMR and ESR, the range of whose applications were already well known at that time. More generally speaking, the abbreviation covers any study of the interactions of the magnetic moment of the muon with its surrounding when implanted into any kind of matter.

 μ SR is a relatively new nuclear method. Roughly speaking it can be classified in between NMR and diffraction techniques. The NMR technique which is closest parallel to μ SR is "pulsed NMR", in which one observes time-dependent transverse nuclear polarization or a so-called "free induction decay" of the nuclear polarization. However, a key difference is the fact that in μ SR one uses a specifically implanted spin (the muon's) and does not rely on internal nuclear spins. In addition, and due to the specificity of the muon, the μ SR technique does not require any radio-frequency technique to align the probing spin. On the other hand, a clear distinction between the μ SR technique and those involving neutrons or X-rays is that scattering is not involved. Neutron diffraction techniques, for example, use the change in energy and/or momentum of a scattered neutron to deduce the sample properties. In contrast, the implanted muons are not diffracted but remain in a sample until they decay. Only a careful analysis of the decay product (i.e., a positron) provides information about the interaction between the implanted muon and its environment in the sample.

Similar to many of the other nuclear methods, μ SR relies on discoveries and developments made in the field of particle physics. Following the discovery of the muon by Neddermeyer and Anderson in 1936, pioneer experiments on its properties were performed with cosmic rays. Indeed, with one muon hitting each square centimeter of the earth's surface every minute, the muons constitute the foremost constituent of cosmic rays arriving at ground level. However, μ SR experiments require muon fluxes of the order of 10^4 - 10^5 muons per second and square centimeter. Such fluxes can only be obtained in high-energy accelerators which have been developed during the last 50 years.

Muon Production, Properties and Implantation Muon production

The collision of an accelerated proton beam (typical energy 600 MeV) with the nuclei of a production target produces positive pions (π^+) via the possible reactions

$$p + p \rightarrow p + n + \pi^{-}$$

 $p + n \rightarrow n + n + \pi^{-}$

From the subsequent decay of the pions (τ_{π^+} = 26.03 ns) positive muons (μ^+) are formed via the two-body decay

$$\pi^+ \rightarrow \mu^+ + \nu_\mu$$

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Since the neutrino has a helicity h = -1, both the neutrino and the μ^+ have their spin antiparallel to their momentum in the pion rest frame. According to the value of the pion momentum at the decay-time, two types of μ^+ -beams are available for μ SR measurements.

The first type of muon beam is formed by the π^+ escaping the production target at high energies. They are collected over a certain solid angle by quadrupole magnets, and directed on to a decay section consisting of a long superconducting solenoid with a field of several Tesla. If the pion momentum is not too high, a large fraction of the pions will have decayed before they reach the end of the solenoid. In the laboratory frame, the polarization of a highenergy muon beam is limited to about 80% and its energy is of the order of 40-50 MeV. Although such a high energy beam requires the use of suitable moderators and samples with sufficient thickness, it guarantees an homogeneous implantation of the muons in the sample volume. Such beams are also used to study specimens inside recipients, for example, samples inside pressure cells.

The second type of muon beam is often called the "surface" or "Arizona" beam (recalling the pioneer works of Pfifer *et al.* from the University of Arizona). Here muons are used that arise from π^+ decaying at rest still inside, but near the surface, of the production target. Such muons, which are 100% polarized, ideally monochromatic and have a very low momentum of 29.8 MeV/c, which corresponds to a kinetic energy of 4.1 MeV, have a range width in matter ~ 180mg/cm⁻². Hence, the paramount advantage of this type of beam is the possibility to use relatively thin samples.

Finally, muon beams of even lower energy ("ultra slow muons" with energy down to the eV-keV range) can be obtained by further reducing the energy of an Arizona beam using moderators, as a thin layer of a van der Waals gas frozen on a substrate. The tunable energy range of such muon beams corresponds to implantation depths in solids of less than a nanometer up to several hundred nanometers. Therefore the study of magnetic properties as a function of the distance from the surface of the sample is possible.

In addition to the above mentionned classification based on energy, muon beams are also divided according to the time structure of the particle accelerator, that is, continuous or pulsed.

For continuous muon beams, no dominating time structure is present. By selecting an appropriate muon incoming rate, muons are implanted into the sample one by one. The main advantage is that the time resolution is solely determined by the detector construction and the read-out electronics. There are two main limitations for this type of beam: (1) a non-negligible random background signal and (2) a rather limited muon incoming rate. Only the background problem can be overcome by using electrostatic deflectors to ensure that no muons enter the sample until the decay of the precedent muon has been observed.

For pulsed muon beams, protons hitting the production target are bunched into pulses which is also reflected on the secondary muon beam. The advantages of a pulsed muon beam are that, in principle, one can use the entire incoming muon intensity and there is almost no background due to accidental coincidences between the incoming muons and decay positrons. The absence of background allows the extension of the time window for measurements up to about 10 x τ_{μ} . The reverse of the medal is that the width of the muon pulse limits the time resolution.

µSR – Relevant Muon Properties

The main properties of the muon relevant to µSR are listed below:

• Charge: +e

Since condensed matter physics or chemistry studies are carried out exclusively with positive muons, the following description will be limited to these muons, (studies involving negative muons are primarily of interest for nuclear physicists)

- Magnetic moment: $3.183345118(89) \mu_p = 8.89059698(23) \mu_N$. This large magnetic moment makes the muon an accurate magnetometer which can be implanted directly into a sample.
- Gyromagnetic ratio: $\gamma_{\mu} = 2\pi \times 13.553882 (\pm 0.2 ppm) \text{ kHz/Gauss}$
- Spin: 1/2

The muon is free of quadrupolar interaction. Compared to NMR experiments, this simplifies the analysis of the μ SR spectra, with a reduced set of adjustable parameters

- Mass: $m_{\mu} = 206.7682838(54) m_e = 0.1126095269(29) m_p$ The muon can be considered as a light isotope of the proton.
- The decay of the muon into an electron and two neutrinos occurs via the weak interaction process after an average lifetime of $\tau_{\mu} = 2.19703(4) \ \mu s$:

$$\mu^+ \rightarrow e^+ + \nu_e + \underline{\nu}_{\mu}$$

The parity violation in the weak interaction leads to an anisotropic distribution of the positron emission with respect to the spin direction of the μ^+ at the decay time. The positron emission probability is given by 90

 $W(\theta) d\theta \mu (1 + A\cos\theta) d\theta$

where θ is the angle between the positron trajectory and the μ^+ -spin, A is an asymmetry parameter which depends on the polarization of the muon beam and positron energy. This anisotropic emission constitutes, in fact, the basics for the μ SR technique.

The asymmetry of W is given by $A = aP_{\mu}(0)$, where $P_{\mu}(0) = |P_{\mu}(0)|$ is the beam polarization (~1) and a is an intrinsic asymmetry parameter determined by the weak decay mechanism. Theoretically, an average of $\langle a \rangle = 1/3$ is obtained if all emitted positrons are detected with the same efficiency irrespective to their energy. Practically, values of $A \cong 0.25$ are routinely obtained (Figure 1.).



Figure 1. Polar diagram of the angular distribution $W(\theta)$ of positrons from muon decay: for the maximum positron energy (dotted line) and integrated over all positron energies

Muon implantation

The muons are implanted into the sample of interest where they lose energy very quickly. Fortunately, this deceleration process occurs in such a way that it it does not jeopardize a μ SR measurement. On the one side it is very fast (~ 100 ps), which is much shorted than a typical μ SR time window (up to 20 μ s), and on the other side, all the processes involved during the deceleration are Coulombic (ionization of atoms, electron scattering, electron capture) in origin and do not interact with the muon spin, so that the muon is thermalized without any significant loss of polarization.

The muons invariably adopt interstitial sites of the crystallographic lattice. In metallic samples the muon's positive charge is collectively screened by a cloud of conduction electrons. Thus, in metals, the muon is in a so-called diamagnetic state and behave like a free muon. In insulators or semiconductors a collective screening cannot take place and the muon will usually pick-up one electron and form a so-called muonium ($Mu=\mu^+e^-$), which has similar size (Bohr radius), reduced mass, and ionization potential similar to the hydrogen atom.

The Technique The fundamentals

A schematic diagram of a μ SR experiment is shown in Figure 2. Incoming muons trigger a clock that defines a time t_0 . The trigger is defined either by the signal produced by the muon crossing a thin plastic detector locate in front of the sample (continuous beam) or by the well defined arrival time of a muon pulse (pulsed beams). If the implanted μ^+ are subject to magnetic interactions, their polarization becomes time dependent, $P_{\mu}(t)$. Since the decay of the muon is asymmetric, the polarization of the muon ensemble can be deduced by measuring the positron distribution as a function of time in different detectors located around the sample. In the time-differential μ SR technique, repeated measurements (~10⁷) are made of the time interval between the μ^+ implantation into the sample and the detection of the emitted positron in a particular direction say, in the direction of the initial polarization $P_{\mu}(0)$; the extension of the following discussion for other arbitrary directions of detection is straightforward.



Figure 2. Schematic illustration of a μ SR experiment. The arrival time t_0 of the muon is either given by a special detector (continuous beam, see figure) or by the arrival of a muon pulse (pulsed beams, not shown). The time between the muon arrival and the subsequent decay recorded by a positron emission at time *t* is used to build a rate vs. time histogram. By Removing the exponential decay due to the muon lifetime and a possible background, one obtains the μ SR signal reflecting the time dependence of the muon polarization.

The time histogram of the collected time intervals has the form

$$N_{e^{*}}(t) = B + N_{0}e^{-t/\tau_{\mu}}\left[1 + A\frac{P_{\mu}(t)}{P_{\mu}(0)}\right]$$

where *B* is a time-independent background, N_0 is a normalization constant and the exponential accounts for the decay of the μ^+ . $P_u(t)$ is defined as the projection of $P_u(t)$ along

the direction of the initial polarization, that is, $P_{\mu}(t) = \mathbf{P}_{\mu}(t) \cdot \mathbf{P}_{\mu}(0) / P_{\mu}(0) = G(t) P_{\mu}(0)$ where G(t) reflects the normalized μ^+ -spin auto-correlation function

$$G(t) = \frac{\left\langle S(t) \times S(0) \right\rangle}{S(0)^2}$$

which depends on the average value, distribution, and time evolution of the internal fields and therefore contains all the physics of the magnetic interactions of the μ^+ inside the sample. Therefor one can write

$$N_{e^{+}}(t) = B + N_0 e^{-t/\tau_{\mu}} \left[1 + AG(t) \right]$$

where AG(t) is often called the μ SR signal and the envelope of G(i) is known as the μ^+ -depolarization function. Since N_0 and B do not contain physical information about the sample investigated, usually only the μ SR signal AG(t) is reported for a μ SR measurement.

Precession and Relaxation

A first example is taken to understand the ability of the muon to study internal magnetic fields. The presence of a local internal field B_{μ} (e.g., created by a magnetic state) at the muon stopping site is to be assumed. If the magnetic field is at an angle θ to the initial muon polarization, the muon will precess around a cone about the magnetic field (see Figure 3). The time evolution of the μ^+ -polarization is therefore given by

refore given by

 $G(t) = \cos \theta + \sin \theta \cos \left(\mu B_{\mu} t \right)$

where $B_{\mu} = |B_{\mu}|$ and the angular frequency $\gamma_{\mu}B_{\mu}$ is known as Larmor precession.

By assuming that the internal field is always perpendicular to the muon polarization (e.g., if an external field is applied), the first term on the right-hand side of the above equation disappears and a simple oscillation of the muon precession is observed (see Figure 2.). On the other hand, if the internal field direction is entirely random (e.g., for spontaneous fields in a magnetic poycrystalline sample), then a simple averaging would yield



Figure 3. Muon-spin precession around an internal field B_{μ} at an angle θ . The wiggling and nonwiggling components on the polarization are schematically represented.

$$G_{z}(t) = \frac{1}{3} + \frac{2}{3}\cos(\gamma_{\mu}B_{\mu}t)$$

Considering the gyromagnetic ratio of the muon and a typical measurement time-window of 10-20 μ s, we see that tiny internal fields as low as 0.1 G can be detected by the μ SR technique.

So far, a well defined value of the internal field was assumed. But nature is of course more complicate and usually a distribution has to be considered. Such a distribution can arise from disorder, dynamics or additional sources of internal field (as nuclear moments). In the case of static local magnetic fields, and assuming a magnetic field distribution function $f(B_{\mu})$,

$$G(t)$$
 is given by $G(t) = \int f(B_{\mu}) \left[\cos\theta + \sin\theta\cos(\psi_{\mu}B_{\mu}t)\right] B_{\mu}$

In other words, the value of the Larmor precession observed in the μ SR signal provides the value of the internal field, whereas the envelope of the signal provides information on the field distribution itself.

Hence, even field distributions centered around zero can be detected through a depolarization of the μ SR signal. As an example, if the strength of the local magnetic field is taken from a Gaussian distribution with zero average, then one observes a signal

$$G(t) = \frac{1}{3} + \frac{2}{3} \left(1 - \Delta^2 t^2\right) \times \exp\left(-\frac{\Delta^2 t^2}{2}\right)$$

where Δ^2/γ_{μ}^2 represents the second moment of the field distribution along one Cartesian axis perpendicular to the initial μ^+ -polarization, that is, $\Delta^2/\gamma_{\mu}^2 = \langle B_x^2 \rangle = \langle B_y^2 \rangle = \langle B_z^2 \rangle$.

This relaxation function is often observed experimentally and reflects the field due to the neighboring nuclear dipoles, which are randomly oriented and considered static within the μ SR time window.

Transverse – Field (TF) Technique

For this technique, an external field H_{ext} is applied perpendicular to the initial muon polarization $P_{\mu}(0)$. $P_{\mu}(t)$ precesses around the total field B_{μ} at the muon site, the value of which can be extracted from the oscillatory component of G(t), recalling that the μ^+ frequency $v_{\mu} = \omega_{\mu}/2\pi = \gamma_{\mu}B_{\mu}/2\pi$.

For a system which is not magnetically ordered, the total field at the muon site is given by $B_{\mu} = H_{\text{ext}} + B_{\text{hf}} + (B_{\text{DF}} + B_{\text{LF}}).$

The last two terms are the demagnetization and Lorentz fields which can be easily calculated from the bulk magnetization and demagnetization tensor. $B_{\rm hf}$ are the internal fields induced by $H_{\rm ext}$.

The μ^+ -frequency shift $K^*_{\mu} = \frac{|B_{\mu}| - |H_{ext}|}{|H_{ext}|} = \frac{\langle \omega_{\mu} \rangle}{\omega_{ext}} - 1$ can be corrected for the contributions

of B_{DF} and B_{LF} to furnish the μ^+ -Knight shift K_{μ} which, as in NMR, contains the relevant information about the hyperfine fields, and therefore about the local susceptibility. In addition, the knowledge of the μ^+ -Knight shift provides valuable indication concerning the type of interstitial site occupied by the muon in the crystallographic lattice.

In addition, the TF technique allows one to gain more insight into the inhomogeneous field distribution at the muon site caused by static fields and into the presence of $1/T_2$ processes responsible for homogeneous line broadening.

Zero - Field (ZF) and Longitudinal - Field (LF) Techniques

With the ZF technique one monitors the time evolution of the muon ensemble under the action of internal magnetic fields. This technique is widely utilized to measure the spontaneous μ^+ -Larmor frequencies in magnetically ordered phases, providing information about the values of the static moment and the type of magnetic structures. The observation of spontaneous μ^+ -frequencies is, however, limited to systems where the presence of static electronic moments produces well defined local fields at the μ^+ -stopping site. Hence, the μ^+ -spin autocorrelation function G(t) depends sensitively on details of the magnetic structure and of course on the μ^+ -stopping site.

In a ZF experiment, a depolarization can occur due to the presence either of a static distribution of internal fields arising from static nuclear or electronic dipole fields, or of a time dependence of the internal fields at the muon site. This time dependence may arise from a fluctuation of the magnetic moments or from muon diffusion. For fast fluctuation (diffusion) the depolarization function will no more assume a Gaussian character at early times, but will be given by

$$G(t) = \exp(-\lambda t)$$
, $\lambda = 2\Delta^2 \tau$

where τ represents the characteristic fluctuation (diffusion) time.

Muon Spin Rotation

In a LF configuration (i.e. $H_{ext} || P_{\mu}(0)$), by choosing H_{ext} to be stronger than the internal fields, the muon's states "up" and "down" are eigenstates of the Zeeman Hamiltonian and any inhomogeneous (static) distribution of the internal fields will not affect the time evolution of the μ^+ -polarization, which will remain constant. This behavior reflects the decoupling of the μ^+ -spin from the static internal fields. Hence, to pinpoint the origin of the depolarization, decoupling experiments in a longitudinal fields are routinely performed.

µSR and Condensed Matter

The aim of the present Section is to shortly present some aspects of the application of μSR in condensed matter. The interested reader is referred to more complete review articles.

Magnetism

There are several advantages in using the µSR technique to study magnetic systems.

- As said, due to the large muon magnetic moment, the technique is sensitive to extremely small internal fields and therefore can probe local magnetic fields which can be nuclear or electronic in nature.
- The local probe character of the muon makes μ SR very sensitive to spatially inhomogeneous magnetic properties. Hence the occurrence of different phases in the sample will be reflected by different components in the μ SR signal, that is, $AG(t) = \sum A_i G_i(t)$

A careful analysis of the the amplitude A_i of these components furnishes a direct measure of the fraction of the sample involved in a particular phase (with the condition $\sum_i A_i = A$). Similarly, μ SR is a powerful tool when the magnetic order is of

short range and/or random nature where neutron experiments will fail. In addition, the μ SR technique can be utilized to check the coexistence of different types of ground states at the microscopic level.

In magnetic phases, the possibility • given bv uSR to extract independently the value of the ordered static moment and the sample volume involved in the magnetic phase allows one to overcome a shortcoming of other techniques (such as the neutron scattering technique) for which these two parameters are strongly coupled therefore hampering their absolute determination.

Organic magnets constitute a typical example of the application of the μ SR technique to study magnetic phenomena. It is well known that neutron scattering can provide extensive information on magnets, in particular, on the magnetic structure and on the excitations. But in compounds with small magnetic moments such as the organic magnets, the μ SR technique is a sensitive and useful probe: it easily yields the value of the critical temperature and information on the



Figure 4. Zero-field muon-spin rotation frequency in the organic ferromagnet *p*-NPNN (from Blundell SJ (1999) Spin-polarized muons in condensed matter physics. *Contemporary Physics* 40: 175.) and temperature dependence of the local field at the muon site deduced from the observed frequency.

thermal behaviour of the order parameter. Hence, the first direct observation of spontaneous magnetic order in the system para-nitrophenyl nitronyl nitroxide (*p*-NPNN) was made using the μ SR technique, and has been subsequently confirmed by neutron diffraction.

Figure 4 exhibits the zero-field muon spin rotation frequency in the organic ferromagnet p-NPNN, which orders only at very low temperatures $T_c = 0.67$ K. The temperature dependence of the frequency of oscillations is a direct measure of the temperature dependence of the internal magnetization, that is, of the ferromagnetic order parameter.

Superconductors

An important research area of recent μ SR studies is the characterization of the superconducting state.

 $B(z) = B \exp(-z/\lambda).$

Although generally accepted, such law was only very recently confirmed on a high-T_c system $(YBa_2Cu_3O_{7-\delta})$ by using low energy muons. In this experiment muons were implanted at several different depths in the superconducting by film, adjusting their kinetic energy (Figure 5). Therefore, the internal field B(z) could be measured directly as a function of depth, and the validity of the above equation could be verified experimentally for the first time. In contrast. with bulk experiments, such as magnetization, or even uSR using usual muons, the functional form of the above equation is assumed when and the appropriate magnetic penetration depth is



Figure 5. Values of the field vs. Depth measured in a high- T_c sample (YBa₂Cu₃O_{7.8}) for various values of temperature. The muon implantation depth is changed by tuning the kinetic energy of the muons (T.J. Jackson et al., Phys. Rev. Lett **84** (2000) 4958)

extracted. This direct determination illustrates the power of low energy muons for near surface studies in superconductivity and magnetism.

Vortex State: It is well known that for a type II superconductor in the mixed (or vortex) phase an applied magnetic field $H_{c1} < H < H_{c2}$ enters the superconductor in the form of quantized flux lines or vortices arranged into a lattice, usually hexagonal. Each vortex contains one quantum of flux $\Phi_0=h/2e$ and the distance *d* between vortices is, except at very high magnetic field, much larger than the unit-cell dimensions (given for a hexagonal vortex-lattice by the relation $d^2=2\Phi_0/s^3 B$). Outside the core region of a vortex, the magnetic field falls off exponentially with distance over a characteristic penetration depth λ (see above).

Since the implanted muons sit at certain crystallographic sites and since the vortex lattice is incommensurate with the crystal lattice. the muon ensemble will randomly sample the field distribution of the vortex lattice (see Figure 6). This field distribution will create a dephasing of the muon polarization, reflected by a relaxation of the µSR signal. Assuming а Gaussian relaxation of the signal, the relaxation rate of the observed precession signal is related to the penetration depth λ by



Figure 6. Schematic field distribution in a type-II superconductor in the mixed (vortex) state. The right-hand side of the figure represents a contour plot of the field *B* (from Blundell SJ (1999) Spin-polarized muons in condensed matter physics. *Contemporary Physics* 40: 175

$$\sigma = \gamma_{\mu} \left\langle B^{2}(\boldsymbol{r}) - \left\langle B(\boldsymbol{r}) \right\rangle^{2} \right\rangle^{1/2} = \frac{0.0609 \gamma_{\mu} \Phi_{0}}{\lambda^{2}}$$

where $B(\mathbf{r})$ is the field at position \mathbf{r} , and the averages are taken over all positions. Therefore the relaxation rate σ of the μ SR precession signal is a measure of the magnetic penetration depth. Its temperature dependence, yields valuable information concerning the symmetry of the superconducting gap and therefore about the pairing mechanism. In addition to the determination of the magnetic penetration depth, a detailed analysis of the data provides information about the coherence length ξ .

Semiconductors

As said, implanted into semiconductors and insulators, the muon often picks up form electron and muonium. an Muonium can exist in three charge states: Mu⁰, Mu⁺ and Mu⁻ which correspond to the three distinct charge states of isolated hydrogen: H^0 , H^+ and H^- . This opens new directions of investigation as the behaviour of muonium provides important information on the charge state, lattice site and dynamics of its hvdrogen analogue. For example. implanted in silicon, neutral muonium has been observed at the tetrahedral site (T) and at a bond-centered (BC) site between two silicon atoms (Figure 7.). While the muonium at the T site was



Figure 7. The crystal structure of silicon, showing the possible muonium (and therefore hydrogen) sites (T = tetrahedral site, BC = bond-center site.

found to be mobile, and rapidly diffusing between such sites, the BC muonium is stationary. The electronic state in the T site is isotropic whilst it is highly anisotropic for the BC site, with axial symmetry along the Si-Si bond. Such site characterization appears rather important since hydrogen is present, as an impurity, in all semiconductors but its low concentration makes it difficult to be investigated using direct spectroscopical methods. In addition, hydrogen is able to passivate dangling bounds known to be present at grain boundaries in the promising new multicrystalline Si photovoltaic cells.

In certain II-VI ompounds, on the other hand, it appears that hydrogen may instead act as a shallow donor - that is to say, it could be used as a dopant in its own right. This has important implications for the electronic properties of new wide-gap materials, now under development for optoelectronic applications. The most recent and compelling evidence for these shallow donor states comes from studies not of hydrogen itself but of its pseudoisotope, muonium. Such results are particularly significant in that they confirm recent theoretical predictions.

Further Reading

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