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Muon Beams, Used for Studying the Solid State

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The positive muon provides a remarkable spectroscopic probe of the solid state. Implanted in virtually any material, its spin polarisation may be monitored to define the sites it occupies in lattices or molecules and to report on local structure and dynamics. Wide ranging applications in solid state science are illustrated in this article by examples in magnetics, chemistry and quantum diffusion.

Primarily, the muon is sensitive microscopic magnetometer: this elementary particle has spin ½ and a magnetic moment about three times that of the proton. The frequencies of its resonance or precession signals provide a direct and accurate measurement of local magnetic or hyperfine fields. Its relaxation functions characterise the distribution in space or the fluctuation in time of these fields.

The muon is rarely a passive probe, however, since it represents a defect carrying unit positive charge. In fact its interactions with the local environment are commonly the main focus of interest; studies of this most fundamental of defects have eliminated complacency in several areas. The interactions, chemical and elastic, are essentially identical with those of the proton, so that their study is invaluable in situations where hydrogen cannot be detected by conventional spectroscopies. Alternatively, when muon and proton behaviour may be compared, the comparison reveals a variety of kinetic and dynamic isotope effects: the muon has about one ninth the proton mass. This order of magnitude ratio greatly facilitates identification of specifically quantum effects, ie those including zero point energy or tunnelling.

1. Muon production and detection: nuclear methods in solid state science

The experimental techniques have come to be known collectively as μSR, standing for Muon Spin Rotation, Relaxation and Resonance (Brewer et al, 1975). They are practised at those accelerator laboratories or meson factories where low energy muon beams are available. The muons derive from the decay of pions, themselves produced when a target of graphite or other light element is exposed to a sufficiently energetic primary proton beam. The success of μSR spectroscopy relies on two circumstances. The first is the intrinsically high degree of spin polarisation of the muon beams (up to 100% for those collected from the decay of pions at rest), and the fact that this initial polarisation is largely preserved when the muons are stopped or thermalised in the sample of interest. The second is the manner in which the subsequent evolution of polarisation within the sample may then be displayed. The muon is itself an unstable particle, and decays with a lifetime of 2.2 microseconds. As each muon decays, it emits a positron in a direction which is closely correlated with its instantaneous spin orientation. This is the classic example of parity violation in radioactive decay, and its discovery by Garwin, Lederman and Weinrich in 1957 represented the starting point for the development of this novel spectroscopy; these authors were well aware of the potential, and concluded their article with the perceptive statement: "... it seems possible that polarised positive and negative muons will become a powerful tool for exploring magnetic fields in nuclei, atoms and interatomic regions".

The decay positrons (β-emission) may be counted in one or more directions using scintillation detectors. This use of "nuclear methods" of single particle counting contributes hugely to the remarkable sensitivity of μSR spectroscopy. It provides a form of trigger detection for magnetic resonance in which transitions of the muon spin (typically in a frequency range between kHz and GHz) are detected at the energy of the radioactive decay. Curious as it sounds to use such a short-lived elementary particle for a solid state probe, the muon lifetime in fact defines an invaluable timescale for certain dynamical studies, whether of hop rates in diffusive motion, or of fluctuating fields in magnetic materials. Various reviews of the techniques and applications are available (see for instance journal reviews by Stoneham, 1979, 1983, and Cox, 1987, and the monograph by Schenck, 1985); the following is a selection of highlights and recent developments.

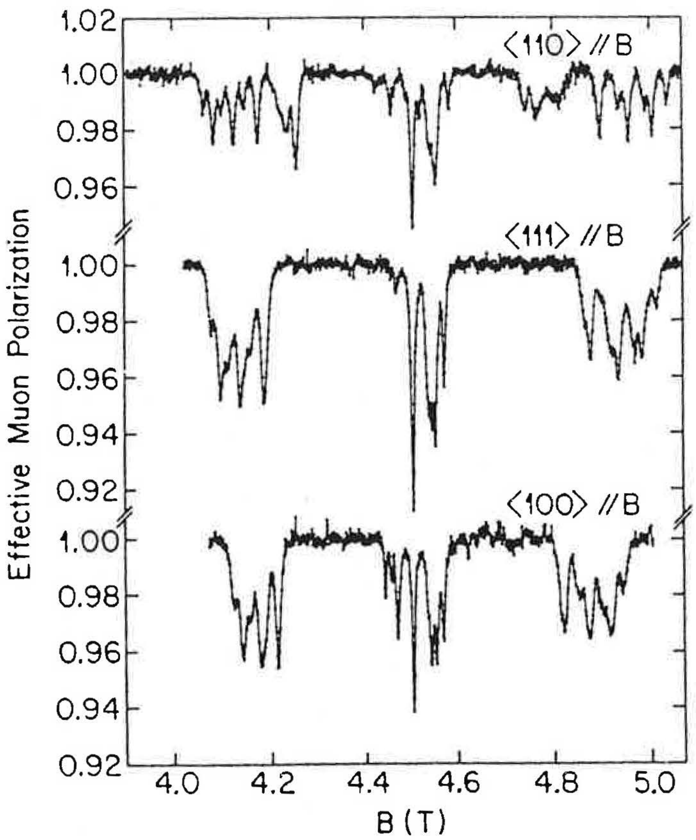

Figure 1. Level crossing resonance spectrum for a muonium defect centre in CuCl (Schneider, 1990). The spectrum represents resonant polarisation transfer from the muon to the neighbouring nuclei, defining the defect site and determining local superhyperfine interactions.

2. Semiconductors and insulators: hydrogen-like defect centres

For a fully polarised muon beam, and counting positrons of all energies, the β-emission is about twice as intense in the "forward" direction (parallel to the muon polarisation) as in the "backward" (antiparallel) direction. Any decrease in forward-backward asymmetry conveniently indicates depolarisation or relaxation of the muon spin. Figure 1 shows an example of *level crossing resonance* in which an external magnetic field, applied parallel to the initial muon polarisation, is tuned so that the combined Zeeman and hyperfine energies of the muon probe exactly match that of the surrounding nuclei. The spectrum also illustrates the formation in certain materials of *muonium*, which may be considered to be the light isotope of hydrogen: just as a proton may pick up an electron to form a hydrogen atom so may a muon pickup an electron to form a muonium atom, $Mu = \mu^+ e^-$ (for a discussion, and an introduction to muonium chemistry, see Brewer et al, 1975 and Walker, 1983). In the example of Figure 1, muonium is formed following muon implantation in one of the cuprous halides and the spectrum indicates that the hydrogen-like atom is trapped and immobile at the tetrahedral interstital site defined by four nearest neighbour Cu^+ ions.

The resonance experiment of Figure 1 measures a time-averaged asymmetry and can benefit from the high intensities now available at certain continuous muon sources. If a pulsed muon source is used, or if the data rate at a continuous source is limited so that individual positrons may be correlated with their parent muons, it is equally possible to display the evolution or relaxation of the muon polarisation, as a function of time following implantation. Measurements of relaxation rate (again in longitudinal field) for an alkali halide, shown in Figure 2, represent spin-lattice relaxation of interstitial muonium due to its diffusive motion through this lattice. The two peaks in relaxation rate (or, in magnetic resonance parlance, T_1 minima) show that muonium mobility is lowest in the vicinity of 100K, and increases both at higher *and lower* temperatures. The low temperature behaviour represents quantum motion of the interstitial defect and is more readily observed for muonium than for hydrogen by virtue of its lighter mass.

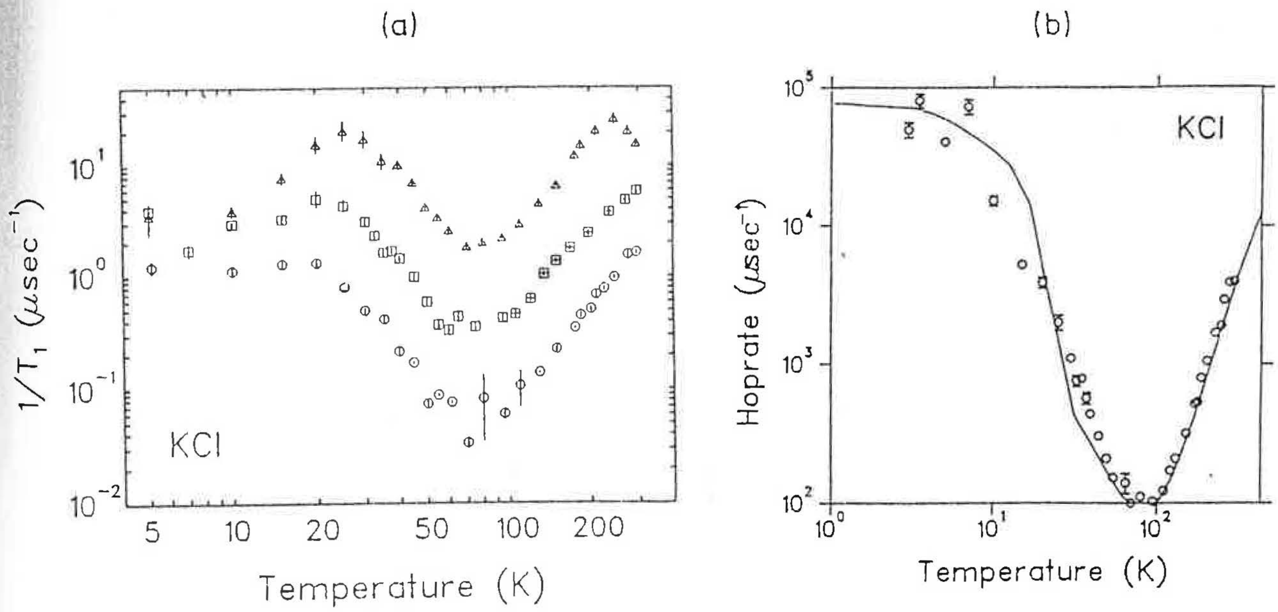

Figure 2. Temperature dependence of muon spin-lattice relaxation rate in KCl, for 3 values of external field, and the extracted rates for interstitial hopping (Kadono, 1990).

For materials in which hydrogen itself is difficult or impossible to detect, muonium proves to be an invaluable substitute. This is the case, for instance, in the elemental (Group IV) semiconductors and in a number of compound (III-V) semiconductors (Patterson, 1988). Figure 3 illustrates a study performed with the magnetic field applied perpendicular to the initial muon polarisation, so that the μSR signal displays oscillations corresponding to precession of the polarisation. (This is muon spin *rotation* the method most commonly employed until recently.) Figure 3 is the Fourier transform of such a signal for gallium arsenide. The remarkable finding, originally made in silicon by Brewer et al (1973), is that there are two distinct muonium states. One of these (known as "normal" muonium) has a hyperfine coupling which is isotropic, though considerably reduced from the vacuum state atomic value; it corresponds as expected to the atom trapped within the tetrahedral interstitial cage. The other, (the so-called "anomalous" muonium state), has a much smaller and highly anisotropic coupling, with axial symmetry along the <III> directions. Its structure remained a puzzle for many years but is now understood to be the more stable of the two and to represent muonium located at a bond-centred site (Cox and Symons, 1986). The consensus of opinion from quantum electronic simulations is now that this structure, which involves a considerable relaxation of the surrounding lattice, is also the most stable for isolated hydrogen defect centres, both in their neutral and positively charged states.

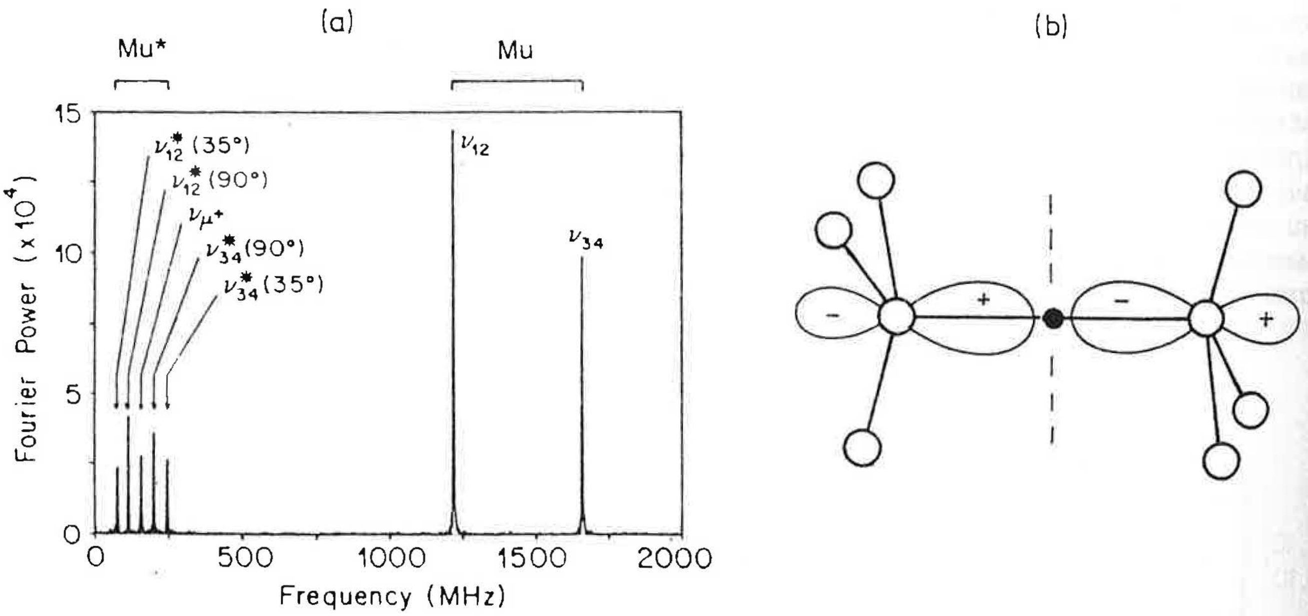

Figure 3. The μSR spectrum of GaAs (a), showing frequencies corresponding to the normal (Mu) and anomalous (Mu*) muonium defect centres (Kiefl et al, 1985) and the "bond-centre" model for Mu* (b) in which the muon lies close to the node of the singly occupied molecular orbital, constructed from sp^3 hybrids on the host atoms (Cox and Symons, 1986)

3. Molecular materials: muon and muonium chemistry

In molecular materials, muonium reacts like atomic hydrogen to become incorporated in a wide variety of molecular species, both diamagnetic (closed shell) and paramagnetic (open shell). Thanks to the unprecedented mass ratio, isotope effects in the reaction kinetics as well as in the one-electron molecular properties of the products are considerably greater than those observed with, for example, deuterium. Most attention has been paid to the hyperfine isotope effect in muonium-substituted organic radicals, which may be traced to the large zero point energy of muonium bound in covalent C-Mu or O-Mu bonds. (Roduner, 1988, Claxton et al, 1990). A similar quadrupole isotope effect has recently been observed in a closed shell species, namely the muonium-substituted water molecule, HMuO (Cox et al, 1990).

The labelling of large molecules by muonium addition is a potentially important tool. In *trans*-polyacetylene, for instance, the unpaired electron introduced by this process is free to migrate along the chain as a bond-alternation defect or *soliton*. The soliton motion in this and other conducting polymers maybe studied via distinctive forms of the muon spin relaxation (Nagamine et al, 1984; Fischer et al, 1991). Most recently, formation of the MuC_{60} radical in Fullerene C_{60} (Ansaldo et al, 1991 ; Kiefl et al, 1991) promises to provide a valuable probe of reorientational dynamics in this fascinating new phase of carbon.

4. Magnetism: spin structure and dynamics

In the presence of atomic moments on the host lattice, μSR proves able to probe both the order parameter and the dynamics. Long range order gives rise to spontaneous static internal fields, and therefore to characteristic frequencies in the muon spin rotation spectra. An example is shown in Figure 4 (a). Fluctuations of the internal fields give rise to muon spin *relaxation*, both in the ordered and paramagnetic phases, whose study nicely complements conventional magnetic resonance and neutron scattering studies (De Renzi et al 1984; Lovesay et al, 1990). Particularly dramatic are the divergence of relaxation rate corresponding to the critical slowing down of the fluctuations and the anisotropy of the correlations. An example of this behaviour is shown in Figure 4 (b).

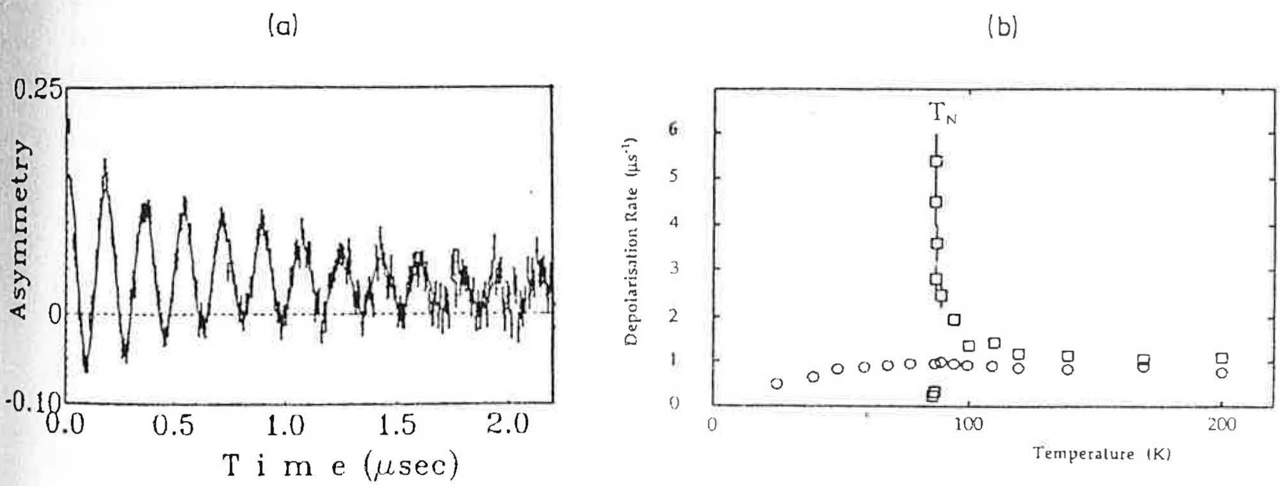

Figure 4. The muon spin rotation signal (a) recorded for La_2CuO_4 in zero external field displays the presence of (antiferro) magnetic order (Budnik et al, 1987). The muon spin relaxation rates (b) recorded for single crystal erbium with muon spin parallel (squares) and perpendicular to the c-axis reveal a striking anisotropy in the critical spin fluctuations (Hartmann et al, 1990).

Since the muon invariably adopts an interstitial position, it is particularly sensitive to changes in the spin structure, via the alteration in dipolar sum. μSR will, in fact, detect the slightest softening of a spin structure before other techniques, and has the unique ability to distinguish a change in static structure from the onset of slow fluctuations. Direct measurements of muon relaxation time cover a dynamic range of four decades (centred roughly on the muon lifetime);

the spin *correlation times* which may be extracted depend on the amplitude of the field modulation involved but range from 10^{-4} seconds in weak moment systems to 10^{-12} seconds in the hard magnets (in many instances opening a time window previously inaccessible: Kalvius et al, 1986).

There is no need for a polarising field or field cycling sequence with μSR detection, so measurements may be made directly in zero external field. This makes the muon an invaluable probe of "fragile" systems, such as spin glasses, or certain heavy fermion systems, where the order can be disturbed or suppressed even in low external fields.

A topical example involving rather weak-moment systems is the identification of the antiferromagnetic phases which are the parent compounds to the various families of high temperature superconductors. The first such is illustrated in Figure 4(a). For the muon, "long range" magnetic order need only mean a correlation length longer than the convergence radius for the dipolar sum, so that much less perfect order is detectable by this local probe than is possible by diffraction techniques. μSR has succeeded in identifying magnetic parent compounds for all the various families of cuprate superconductors, both electron-doped and hole-doped (Luke et al, 1989; De Renzi et al, 1990).

5. Superconductors: mapping internal fields

But it is perhaps the study of the superconducting phases themselves which has recently brought μSR measurements to the attention of a wide audience. The elimination of anyon models of high-T_c superconductivity (Kiefl et al, 1990) is a striking illustration of the sensitivity of the muon magnetometer. For the mixed state of all type II superconductors, conventional and high-T_c, the μSR frequency spectrum serves to map the distribution of internal flux within the vortex system (Herlach et al, 1990); the linewidth is directly proportional to the field contrast (rather than the square of this quantity, as for diffraction methods) and undoubtedly provides

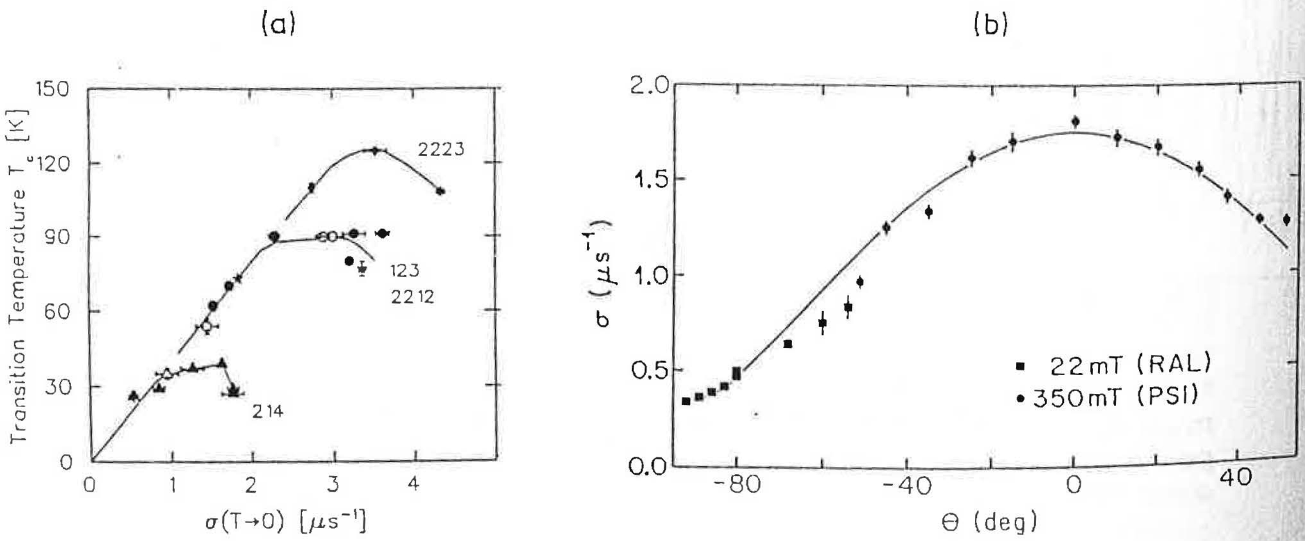

Figure 5. Correlation of μSR linewidth σ with transition temperature T_c for various superconductor families (a) (Uemura et al, 1990) and its variation with orientation for single crystal $YBa_2Cu_3O_{7-\delta}$ (b) (Forgan et al, 1990). Linewidth is proportional to the variance of the internal field and to the inverse square of the London penetration depth.

the most reliable measurement of London penetration depth. This quantity is intimately related to the pairing mechanism of the superconducting carriers, via their concentration and effective mass. Its measurement for such a wide variety of materials as the cuprate high-T_c superconductors, heavy fermion systems, chevrel phases and even organic superconductors, and the discovery of a systematic variation with transition temperature which embraces all of these (figure 5a) would be unthinkable without μSR. Single crystal measurements also bear out the predicted tensor properties (Barford and Gunn, 1988) relating to the almost two-dimensional conduction in the layered cuprate systems (Figure 5b).

6. Muons in metals: quantum dynamics

In metals, the use of muons to mimic proton behaviour has been exploited to the full, and every variant of the μSR techniques brought to bear on questions relevant to hydrogen in metals. In particular, muons in metals have been used to clarify issues of quantum mobility: they serve as a testing ground for the fundamental problem of a quantum mechanical system (here an interstitial defect light enough to tunnel) coupled to a classical system (the electron and phonon baths). The coupling is large, so that the initial muon state (which must be extended on entry at epithermal energy) collapses to a localised state, "self-trapped" within a single interstitial well. In semiconductors, the phonon coupling is responsible for the localisation; in metals the electron coupling is dominant. Screening of the muon potential by a local accumulation of electron density is such as to preclude the formation of muonium (ie no paramagnetic moment is centred on the muon) and the local electronic structure is diamagnetic, as it is for interstitial protons. Phonon coupling is also evident from a slight local relaxation of the lattice, so that the overall defect has "small polaron" character *(Figure 6a)*.

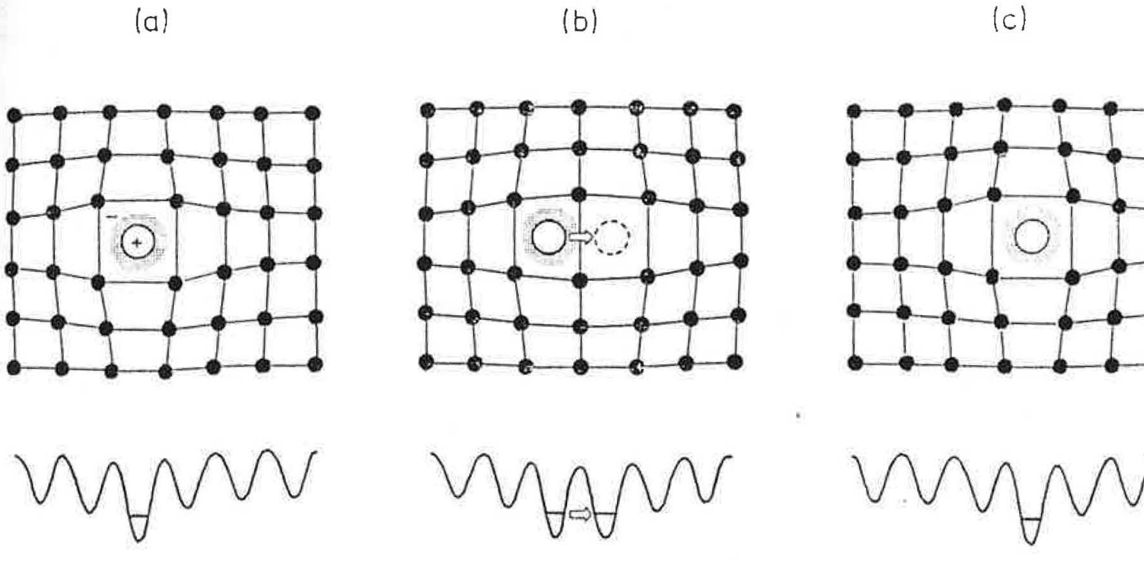

Figure 6. Interstitial localisation of the positive muon as a small polaron defect in metals (a) and the elemental diffusive step via phonon-assisted tunnelling (b,c). thermal fluctuations provide a favourable intermediate configuration for tunnelling between initial and final sites.

Of interest is how this entity, the interstitial muon together with its screening charge and lattice distortion, can move from one site to the next in an elemental diffusive step. The onset of the slowest motion is apparent in the zero-field and low longitudinal-field muon spin-lattice relaxation function. The zero-field spectrum of Figure 7a is for copper, which is a good example of a metal in which hydrogen is too insoluble to be studied by conventional spectroscopies. Faster diffusion becomes apparent in the transverse-field muon spin rotation spectra, as a motional narrowing of the linewidth analogous to that familiar in conventional NMR. The hop rates deduced from these various measurements reveal a broad minimum in mobility around 40K and an increase both to higher and lower temperatures (Figure 7b). The behaviour is characteristic of small polaron states (compare the case of muonium in insulators: Figure 2). Level crossing resonance confirms that equivalent interstitial sites are involved at all temperatures (Luke et al, 1991). The thermally activated hopping reveals an activation energy which is much less than the classical barrier height between neighbouring interstitial sites, and is well described by phonon-assisted tunnelling, of the sort propounded by Flynn and Stoneham and illustrated in Figure 6. The low temperature increase in mobility is much weaker than for the neutral small polaron defect in insulators, and finds its explanation in a departure from adiabatic behaviour, namely a surprising reluctance on the part of the electronic screening change to follow the muon motion (Kondo, 1985).

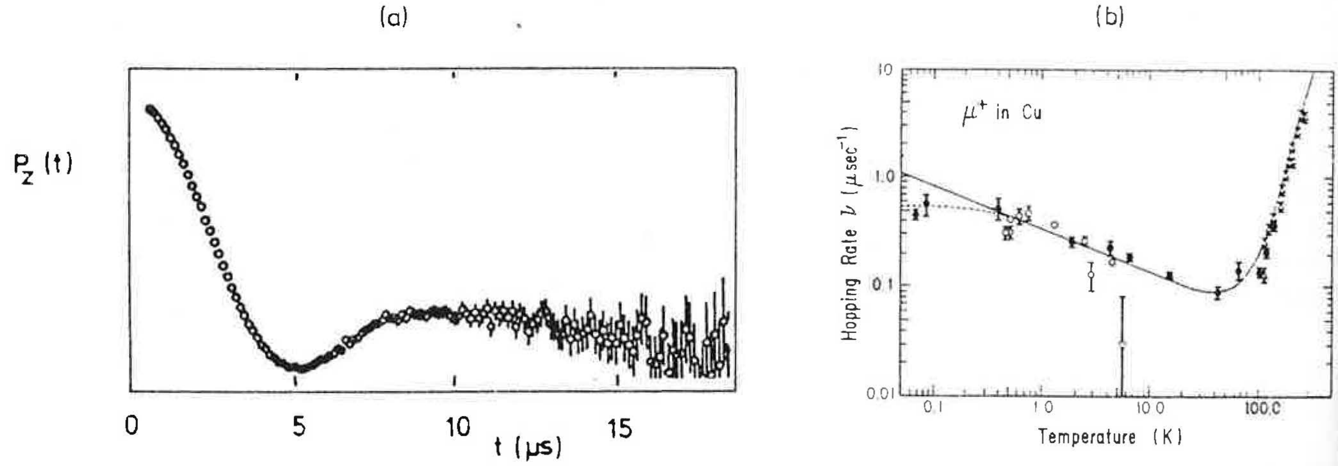

Figure 7. The zero field relaxation function for muons in copper metal is shown in (a) (this ISIS test spectrum is remarkable for displaying evolution of the polarisation beyond eight muon lifetimes: Karlsson, 1989). μSR provided the first demonstration of this distinctive function, in which the polarisation $P_z(t)$ recovers at long elapsed time to $^1/_3$ of its initial value, as predicted by Kubo and Toyabe (1987). Relaxation of the $^1/_3$ tail proves sensitive to extremely slow diffusion. Collected results for muon hop rates are reported in (b) and reveal quantum mobility at low temperatures as well as thermally activated motion (Kadono et al, 1986).

7. Perspectives

These examples give a glimpse of the broad range of application of implanted muon studies. Classed with the nuclear-probe spectroscopies, μSR provides information on local structure which ideally complements the k-space information obtained from diffraction techniques, and contributes to a detailed microscopic picture of matter.

By virtue of the mass and lifetime of the muon probe, μSR studies significantly extend the range of dynamical phenomena accessible to conventional magnetic resonance.

The present status of this relatively novel spectroscopy is best compared with that of magnetic resonance some decades ago. Results on model systems validate the technique, but have also produced some surprises (notably as regards hydrogen defects in semiconductors and light interstitial diffusion in metals) which cause established views to be revised. New and more complex systems may now be tackled with confidence. In amorphous and mesoscopic structures for instance, metallic and semiconducting, issues of site occupancy, site competition with hydrogen and diffusion dynamics are well suited to muon study; such experiments are in their infancy, but have considerable potential.

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