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FINAL ANNUAL REPORT

MUON SPIN ROTATION IN SOLIDS

VIRGINIA STATE UNIVERSITY

Supported by NASA grant NSG 1342

February 1983 - February 1984

The February 1983 - February 1984 period saw several important developments in this research program. The most important was the development of a successful muon spin rotation facility at the Alternating Gradient Synchrotron of Brookhaven National Laboratory. During May 1983 this facility, which was proposed in 1978, finally came on line. During a run which lasted three weeks extensive beam studies were conducted and a preliminary measurement of muon depolarization in Aℓ at low temperature with zero magnetic field was conducted. This beam provides about 2,400 μ^+/second on a sample 1 in^2 in area, with 500 good events per second. Additional development work was done in July 1983 while the beam was off for the usual summer maintenance period. Preliminary preparations were made for a run which is anticipated to begin in March 1984. Virginia State University participants in the Brookhaven work include Carey E. Stronach (P/I of NSG 1342), John Bilodeau (former instuctor of electrical engineering technology) and Lucian R. Goode (graduate student). Collaborators from other institutions include William J. Kossler (overall group leader of the Brookhaven MuSR effort) and three graduate students from the College of William and Mary, Anthony T. Fiory of Bell Laboratories, William F. Lankford of George Mason University, and Mohammed Numan of East Carolina University. It is expected that the aluminum data will be published in conjunction with results of related experiments to be done in the 1984 run.

A second major accomplishment was a set of MuSR experiments on nickel alloys, which was conducted at the Swiss Institute for Nuclear Research in

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August/September 1983. Plots of these data are included as Appendix A to this report. These results will be reported at the Virginia Academy of Science meeting in May 1984 in Richmond, and at the Fifth Risø International Symposium on Metallurgy and Material Science in September 1984 in Roskilde, Denmark. The abstract of the latter is included as Appendix B and a paper on this work will be published in the Risø Symposium proceedings. Collaborators on these studies include Robert I. Grynszpan of the Center for the Study of Metallurgical Chemistry, Vitry-sur-Seine, France, Pascal Dassonvalle (a doctoral candidate at the University of Paris), and Bruce D. Patterson of the University of Zürich. We anticipate further studies of nickel alloys at Brookhaven during the 1984 run and, possibly, additional experiments at the Swiss facility and/or the NIKHEF laboratory in the Netherlands. We expect that Lucian Goode will do his masters thesis on the results of the Brookhaven experiments on nickel alloys.

We are also pleased to report that a paper based upon the 1981/82 experiments upon uniaxially strained iron crystals (done at the Swiss facility) was accepted for publication by Hyperfine Interactions in mid-1983, and should appear in that journal in the near future. A preprint is included as Appendix C of this report. Finally, a paper which was accepted for publication in 1982 (in Electronic Structure and Properties of Hydrogen in Metals, Plenum Publishing Corp.) appears in that book, which was published in early 1983. A reprint is included as Appendix D of this report.

Two students have been supported by this grant in 1983-84. Lucian Goode is expected to complete his Master of Science thesis in MuSR by the end of the summer of 1984. Michael Davis is a sophomore who is also an experienced electronics technician. He has been doing repairs and routine maintenance upon a number of electronics units used in the MuSR experiments.

We are most pleased that NASA has supported this basic research, and that this work is continuing to receive support (at a higher funding level) under NASA grant NAG-1-614. This new support, coupled with the success of the Brookhaven MuSR facility, should generate many exciting MuSR studies in the coming years.

Respectfully submitted,

Carey E. Stronach

Carey E. Stronach
Principal Investigator
February 28, 1984

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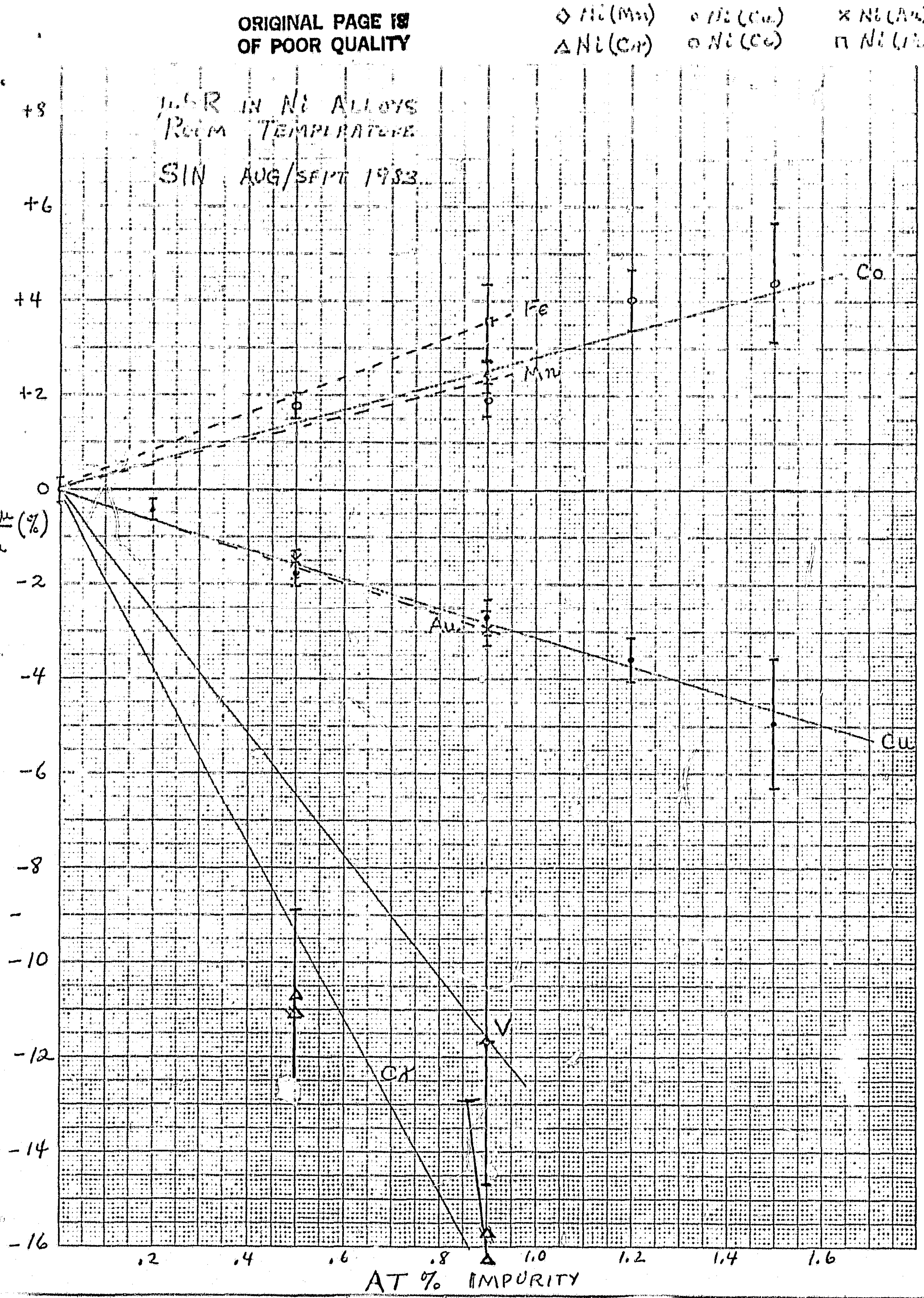

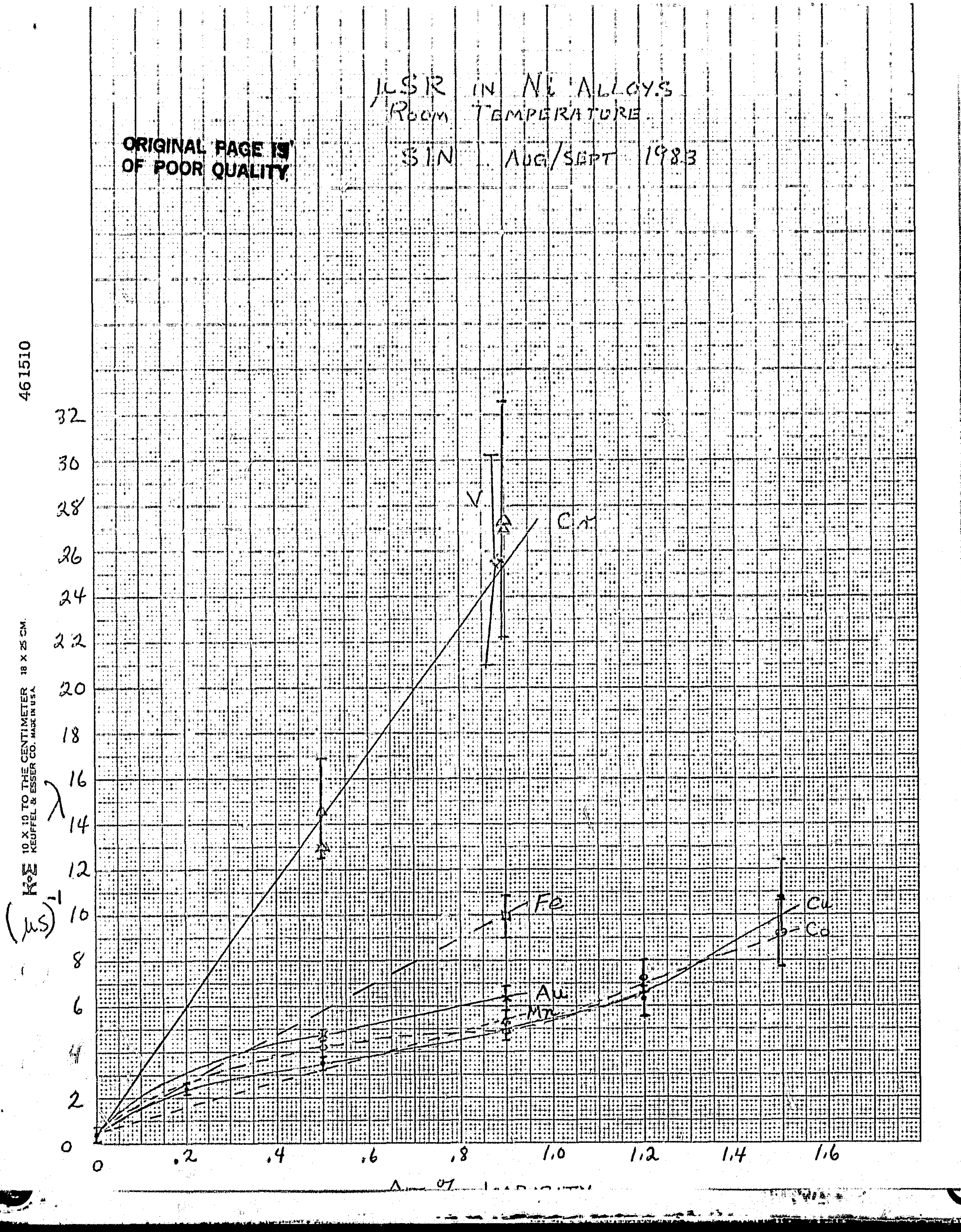

μSR in Ni Alloys
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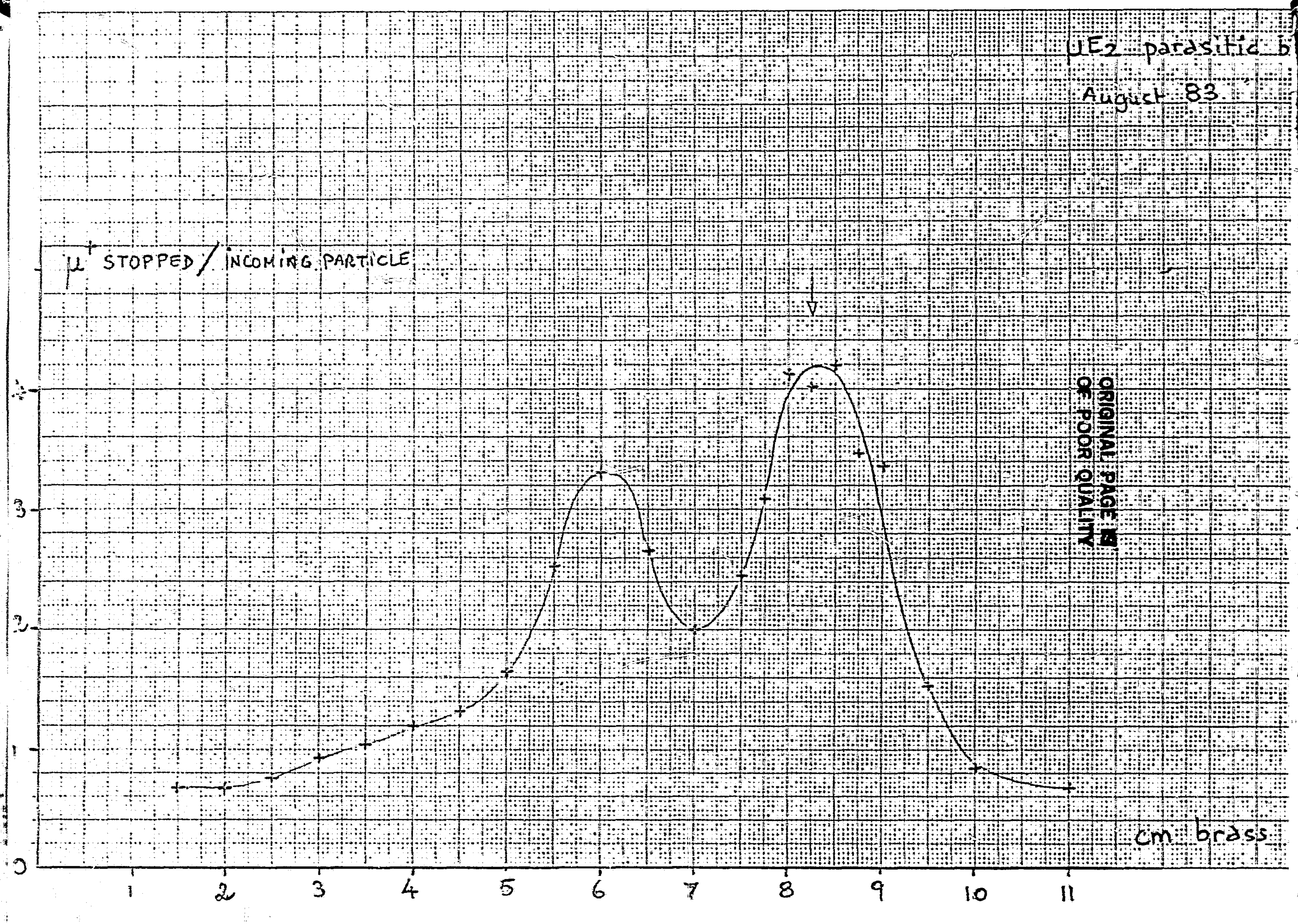
SIN Aug/Sept 1983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Fifth Riso International Symposium on Metallurgy and Material Science
MICROSTRUCTURAL CHARACTERIZATION OF MATERIALS
BY NON-MICROSCOPICAL TECHNIQUES
3-7 Sept. 1984 - Riso Natl. Lab., Roskilde, Denmark

THE INTERACTION OF POSITIVE MUONS WITH IMPURITIES IN NICKEL

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The Muon Spin Rotation (μSR) technique may be used to probe the microscopic electron density in materials. It is found that a positive muon implanted in a solid acts like a light proton ($m_p / m_\mu = 9$), migrating among interstitial sites of the lattice and being attracted or repelled by chemical and/or structural defects (1).

In pure nickel at room temperature (RT), the muon feels a local magnetic field $B_\mu \approx 0.1361(5)$ Tesla. When passing close to a defect it will sense a somewhat different field, and this will change the average B_μ experienced by the ensemble of migrating muons in general by an amount proportional to C_X, the defect concentration ($\Delta B_\mu / B_\mu = K_X C_X$). The constante of proportionality K_X will have a particular value K_{X0} (corresponding to simple dilution) for muon migration which is unbiased by the impurities (i. e. at sufficiently high temperature). The magnetude of K_X will be smaller or larger than that of K_{X0} if the muon is respectively repelled or attracted by the defect (2,3).

We report RT observations of average local field shifts in samples of nickel doped with up to 1.5 at. % of various metallic elements. Our results, in conjunction with the temperature dependent measurements of reference (2), suggest that impurities with full d electron shells (Cu, Au; $K_X = -3$ % per at. %) tend to repel the muon whereas those lacking d electrons with respect to Ni (Co, Fe, Mn; $K_X = +3$ to $+5$ % per at. %) attract the interstitial muon. Negative values of K_X as large as -15 % per at. % have been found for V and Cr impurities, indicating a large local change in the electron spin density. High temperature μSR and magnetization measurements are in progress to allow an unambiguous determination of the muon-impurity interaction and the impurity-induced change in the local spin density.

Additional examples will be mentioned, including interactions of muons with vacancies (4) and dislocations (5).

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Uniaxial Stress Induced Frequency Shifts for Muons in Single Crystal Fe

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Abstract: We report the first results on uniaxial stress-induced frequency shifts in an Fe single crystal. Stress was applied along the <100> axis, which was also the axis of magnetization induced by an external field. The observed frequency shift was -0.34±0.023 MHz per 100 micro-strain, which corresponds to $\partial B_\mu/\partial\varepsilon = +25.1\pm1.6$ G/100με. The positive sign arises from the negative sign of B_μ itself. This result is interpreted as follows: The stress induces a statistical population shift between magnetically inequivalent sites. Extrapolations from the calculations of Sugimoto and Fukai from Nb and V to Fe yield order of magnitude agreement. The 4T(0) site system seems more likely.

Introduction

We have measured the muonic precession frequency shift produced by uniaxial strain in an iron single crystal. The strain was along a <100> direction, which was also the axis of magnetization which was induced by a small applied field. The precession frequency is proportional to the average local magnetic field which is often decomposed with the usual definitions for the terms as:

$$\underset{\sim}{B}_{\mu} = \underset{\sim}{B}_{ext} + \underset{\sim}{B}_{dem} + \underset{\sim}{B}_{L} + < \underset{\sim}{B}_{d} > + \underset{\sim}{B}_{hf}. \tag{1}$$

We are interested in the change of $\underset{\sim}{B}_{\mu}$ with applied uniaxial stress: $\Delta\underset{\sim}{B}_{\mu}$. Changes in $\underset{\sim}{B}_{ext}$, $\underset{\sim}{B}_{dem}$ and $\underset{\sim}{B}_{L}$ are small for our purposes, and we find that the changes in the hyperfine field arising from its variation with distance from host lattice ions are small, based upon the results of Butz et al.[1] for precession frequency shifts under hydrostatic pressure. Differences in hyperfind field from one magnetically inequivalent site to another would need to be of the same order as the dipole field differences to be taken into consideration and there seems to be no mechanism to produce such large differences.

What remains, a change in $< B_d >$, is primarily from displacements of the nearest neighbor dipoles and from a change in the thermal average over the magnetically inequivalent sites. Thus we have

$$\Delta\underset{\sim}{B}_{\mu} \cong -2/9(B_{\ell}-B_{t})\Delta E/kT + 1/3\ (B_{\ell}+2B_{t}) \tag{2}$$

where $E = -(\frac{S_{11}-S_{12}}{S_{11}})(p_1-p_2)\varepsilon_{100}$, S_{ij} are the elastic compliances for Fe, p_i are the diagonal elements of the double force tensor[2] for a muon in Fe, $(B_\ell - B_t)$ is the difference in magnetic fields for the magnetically inequivalent sites with tetragonal axes parallel or perpendicular, respectively to the magnetization axis, and ε_{100} is the strain along the <100> direction. $B_\ell + 2B_t = 0$ with no strain and is caused to be finite by strain induced displacement of magnetic dipoles on nearby Fe host lattice sites. B_ℓ and B_t depend strongly on site configuration, local lattice distortion, and the shape of the muon's wave function while undergoing zero point motion. The first term in Eq. 2 is, as it turns out, the most important.

Experiment

These experiments were carried out at the πE3 port of the SIN accelerator using the Mili μSR apparatus. The surface muons passed through a 2 x 5 mm collimator to stop in the sample held in the "puller" illustrated in Fig. 1. The temperature was approximately 300 K held constant by thermal contact to temperature controlled flowing alcohol. The long axis of the 1 x 4.6 x 46.13 mm^3 sample was the stress axis, the magnetization axis, and was within less than 2° of the <100> crystallographic direction. The wide surface deviated from <010> by about 10°. The smaple was ARMCO iron from Monocrystals, Inc. Strain was measured directly on the sample with a strain gauge.

Positron detectors were upstream (B) and downstream (F) and the initial polarization of the muon was upstream and perpendicular to the <100> long axis of the sample. Date were fit to:

$$N_{F/B} = N_{oF/B} e^{-t/\tau_\mu} \{1 +/- P[F_L e^{-t/T_1} + F_T e^{-t/T_2} \cos(\omega_\mu t + \phi)]\} + B_{F/B} \qquad (3)$$

F_L and F_T refer to the fraction of the domains which are magnetized parallel and perpendicular, respectively, to the initial muon polarization. A plot of F_T/F_L versus B_{ext} is shown in Fig. 2. The initial value of 2.5 is not too far from 2 which would be expected for random domain orientations. Above $B_{ext} = 150$ G $F_T >> F_L$ indicating nearly complete domain alignment. F_T/F_L remained high so long as the aligning field was present.

Figure 3 shows our result for precession frequency versus strain. The shift in frequency is -0.34 ± 0.023 MHz per 100μ strain. This corresponds to: $\partial B_\mu/\partial \varepsilon_{100} = +25.1 \pm 1.7$ G per 100_μ strain. The opposite sign since $\underset{\sim}{B}_\mu$ and $\underset{\sim}{B}_{ext}$ are antiparallel.

Discussion of the Results

To compare with experiment we must evaluate Eq. 2. B_ℓ and B_t have been calculated taking lattice site, lattice relaxation, and muon wave function into account. Results for B_ℓ are shown in Table I. Since the second term of Eq. 2 is much smaller than the first term we make comparison between a theoretically based $(p_1-p_2)_{th}$ and a $(p_1-p_2)_{exp}$ which fits the observed frequency shift. $(p_1-p_2)_{th}$ was obtained by extrapolating from the results of Sugimoto and Fukai[3] for the bcc Nb and V to Fe by the lattice parameter. Sugimoto and Fukai consider two site configurations for the muon which they call 4T(0) and 1T. The 4T(0) site is an octahedral site with simultaneous occupancy of the 4 neighboring tetrahedral sites, while a 1T site is a single tetrahedral site. This extrapolation yields: $(p_1-p_2)_{th} = 3.73$ eV 4T(0) and -1.23 eV 1T. The experimental results were corrected for the smaller second term using the same percentage lattice distortion as Sugimoto and Fukai obtained for Nb and muon wavefunctions of similar extent ($|\psi_\mu|^2 = \frac{1}{\alpha^2\beta\pi^{3/2}} \cdot e^{\frac{-x^2-y^2}{\alpha^2}} \cdot e^{-z^2/\beta^2}$; $\alpha = 0.2a$; $\beta = (1/\sqrt{2})\alpha$ 4T(0), $\beta = \sqrt{2}\alpha$ 1T). They are $(p_1-p_2)_{exp} = 1.8$ eV 4T(0) and -3 eV 1T. There is order of magnitude agreement for either site, slightly better for the 4T(0). Even better agreement would be obtained for the 4T(0) if the neighboring ions have their moments reduced by the presence of the muon.

Acknowledgements

We would like to thank W. Kündig and P. F. Meier for their hospitality, cooperation and tolerance. This work was supported in aprt by NSF Grant DMR 8007059 and NASA Grant NSG 1342.

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Figure Captions

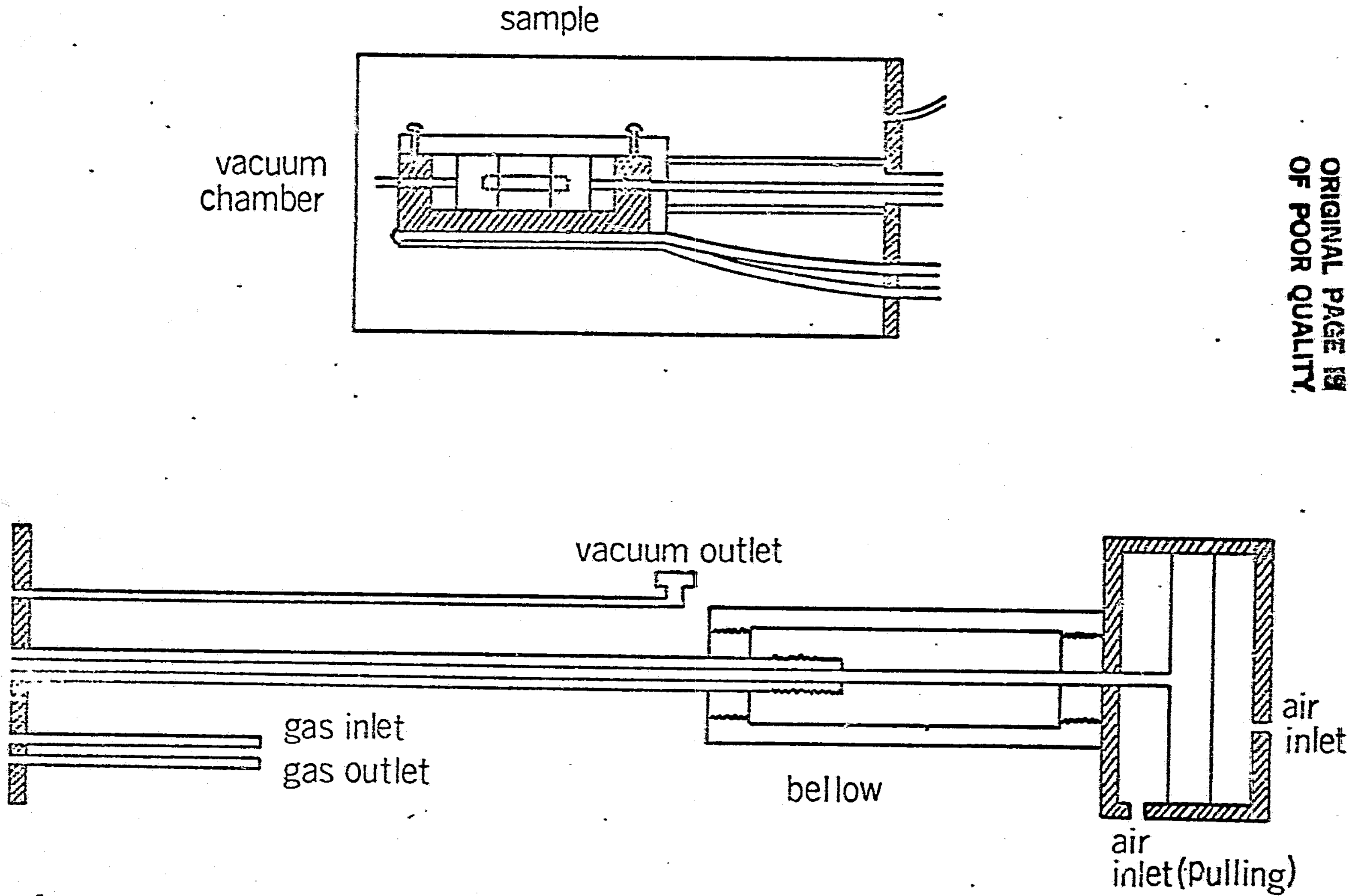
Figure 1 Experimental Apparatus.

Figure 2 F_T/F_L versus B_{ext}. $F_T(F_L)$ is the fraction of domains aligned transverse (parallel) to the muon's initial polarization.

Figure 3 Muon precession frequency versus strain. The open circles represent the observed frequency observed immediately after the stress was released which produced the indicated strain for the full circle point.

Table I. Dipolar Fields under Various Conditions

Lattice		Rigid	Relaxed			
Muon		Point	Point	$\alpha = \beta$ .15–.19	$\alpha = .19$ $\beta = .15$	$\alpha = .19$ $\beta = .25$
B_ℓ(kG)	4T(0)	18.64	13.56	13	9.3	–
	1T	–5.24	–3.73	–3.7	–	–4.8


sample
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air
inlet (pulling)

Fig. 1.

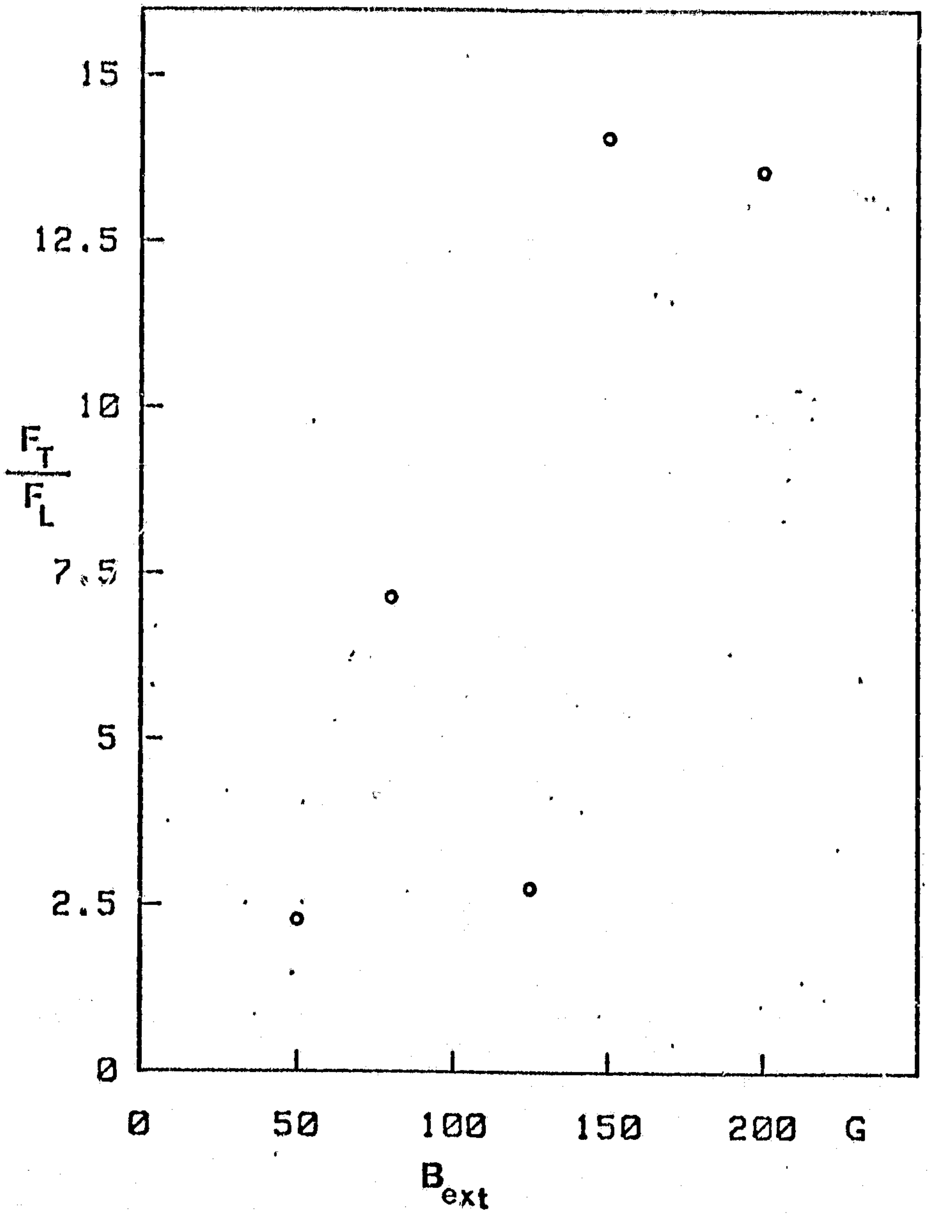

Fig. 2.

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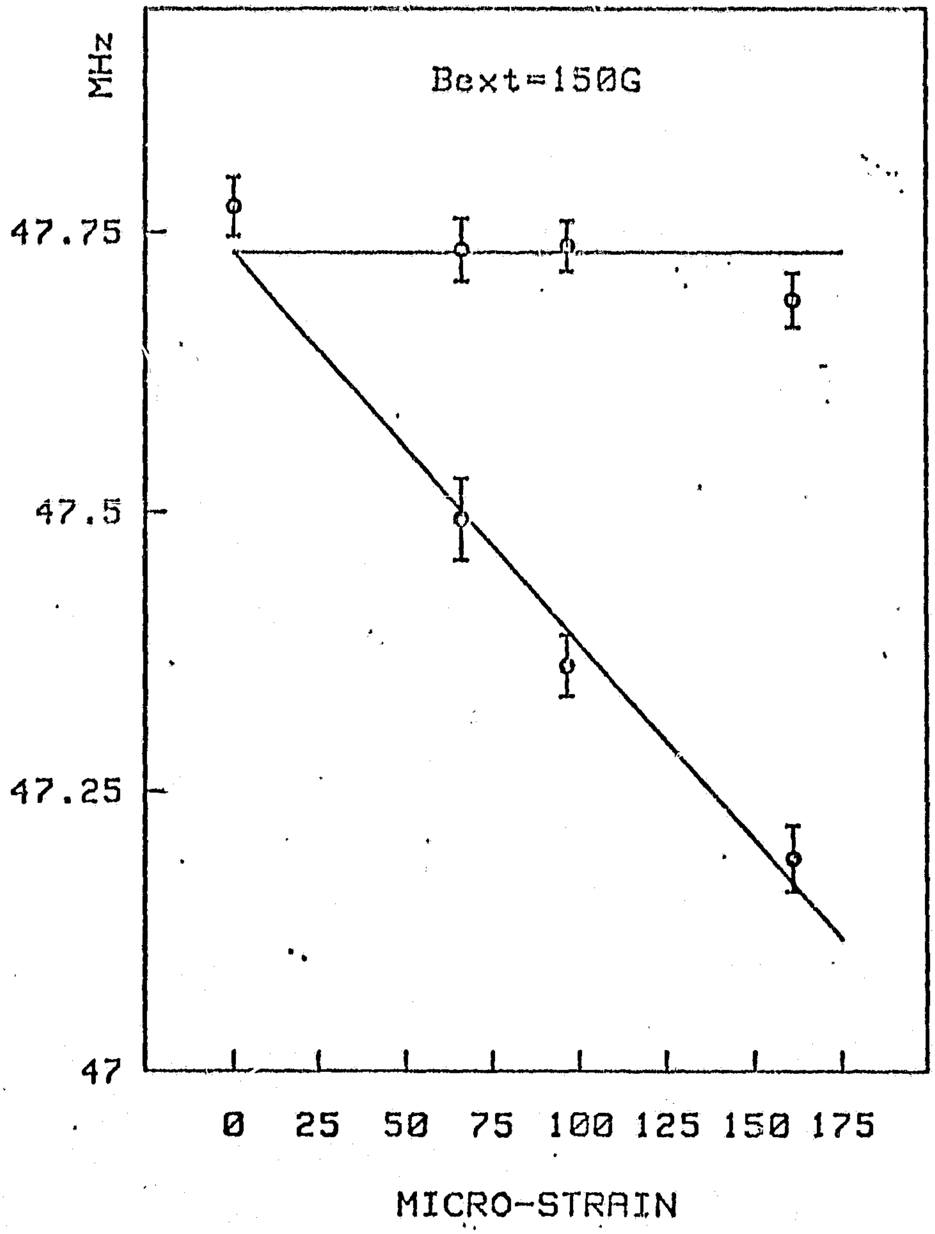

Fig. 3.

From: ELECTRONIC STRUCTURE AND PROPERTIES OF HYDROGEN IN METALS
Edited by P. Jena and C.B. Satterthwaite
(Plenum Publishing Corporation, 1983)

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BEHAVIOR OF POSITIVE MUONS IMPLANTED IN IRON ALLOYS

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ABSTRACT

Muon spin rotation measurements were made upon Fe alloyed with small amounts of N, Al, Si, Ge, Ti, V, Cr, Mn, Co, Ni, Nb, Mo and W. The measurements are described and the results are discussed in terms of the effect of impurities and associated strain upon B_μ and B_{hf}.

INTRODUCTION

We employed μSR for a study of iron alloys. The time dependent angular correlation of the muon's positron decay precesses at 13.552 MHz$\cdot B_\mu$ (KG) where B_μ is the average field sensed by a μ^+ implanted into the sample. See ref. 1 for more detail on the technique.

The experiments were conducted at the TRIUMF cyclotron facility. As previously mentioned, one obtains the local field B_μ directly from the precession frequency. With no external field applied, and with the dipolar fields averaged to zero by the motion of the muon, B_μ has only two major contributions, the Lorentz cavity field $B_L = 4\pi M/3$, and the contact hyperfine field B_{hf}, which arises from a polarized electron density about the μ^+. The hyperfine field is then given by

$$B_{hf} = B_{\mu} - B_{L}.$$

For Fe at room temperature B_L is about 7.2 KG and B_{μ} is about -3.6 KG, so values for B_{hf} are of the order of -10.8 KG.

We parameterize the effect of the impurity upon the hyperfine field by calculating the fractional change in the hyperfine field, normalized to the impurity concentration, $\Delta B_{hf}/c\ B_{hf}$. For most of the alloys this has been determined only at room temperature, but for Fe(Mo) and Fe(Al) it has been measured over a range of temperatures.

HYPERFINE FIELDS

The table below summarizes our determinations of $\Delta B_{hf}/c\ B_{hf}$ at room temperature:

Impurity	$\Delta B_{hf}/c\ B_{hf}$
N	-8
Al	-0.23
Si	-0.42
Ge	0
Cr	-0.09
Mo	-0.72
W	-0.79
Ti	-13
V	-1.10
Cr	-0.09
Mn	-1.10
Co	0.2
Ni	-4.4

If an impurity served only to dilute the hyperfine field by acting as a non-magnetic hole at a substitutional lattice site, with random site sampling by the μ^+, one has $\Delta B_{hf}/c\ B_{hf} = -1$. We note that Mo, W, V and Mn approximate such behavior at room temperature, while Cr, Al and Si decrease the magnitude of B_{hf} considerably less than predicted by pure dilution. These results for Al and Si may be at least partially explained by the mechanism described in ref. 2, in which the temperature dependence of B_{hf} in Fe(Al) was measured. An increase in $\Delta B_{hf}/c\ B_{hf}$ with increasing temperature suggests that μ^+ are repelled from solutes with p-wave bonding electrons, and thus experience smaller solute effects. Even so, the high-temperature limit (or random sampling limit) of $\Delta B_{hf}/c\ B_{hf}$ for Fe(Al) is still only ≈ -0.35.

Co, which increases both the moment per Fe atom and the Curie temperature (Tc), is the only solute which makes B_{hf} more negative. Ge, which also increases the moment per Fe atom and Tc, has the next most positive effect, zero.

Cr is out of line in both the vertical Cr, Mo, W and horizontal W, Cr, Mn sequences in the periodic table. To the best of our knowledge there is no other characteristic of these alloys for which this is seen. We can only speculate that Cr impurities produce less internal strain in the crystals than the other impurities do.

Ti, Ni and N impurities produce very large reductions in the magnitude of B_{hf}, each being several times the prediction of pure dilution. Again, this may be due in part to non-random site sampling by the μ^+. The temperature dependence of B_{hf} was measured for Fe(Mo) and it showed an effect opposite to that found for Fe(Al): the μ^+ is apparently attracted to Mo impurity sites in Fe.[3] The effect of Ti is about double what one would obtain if B_{hf} were reduced to zero at all sites adjacent to Ti impurities. Studies of Fe(Ti) at higher temperatures show that while $|\Delta B_{hf}/c\ B_{hf}| >> 1$, it is decreasing with increasing temperature. This suggests that the Fe(Ti) result may arise, in part, from preferential sampling by the μ^+ of sites adjacent to Ti atoms.

A study of the temperature dependence of B_μ in Fe(Ge) showed an hysteresis effect upon annealing (Fig. 1). The form of the $B_\mu(T)$ curve changed upon annealing, apparently because of the release of internal strains in the annealing process.

A large decrease in B_μ was found in the two-phase alloy Fe + Fe_2Nb, compared with the pure Fe from the stock material from which all the alloys were formed. This sample consists of Fe_2Nb inclusions in a pure Fe matrix. Two samples with different Nb concentrations showed that the reduction in B_μ is more pronounced with greater Nb concentration. This is contrary to the naive expectation that, since all μ^+ spin rotation takes place in the pure Fe, the frequency of the signal would be unchanged by the Fe_2Nb inclusions with only the amplitude changing. This reduction in magnitude of B_μ is of the order of magnitude of Fe anisotropy fields around inclusions and may result from weak trapping of the μ^+ around these inclusions.

STRAIN EFFECTS

The observation that in some cases the presence of impurities in Fe decreases the magnitude of B_{hf} more than pure dilution, the shift of B_μ in Fe(Ge) upon annealing, and the decrease in the magnitude of B_μ with addition of Fe_2Nb inclusions all lead us to the

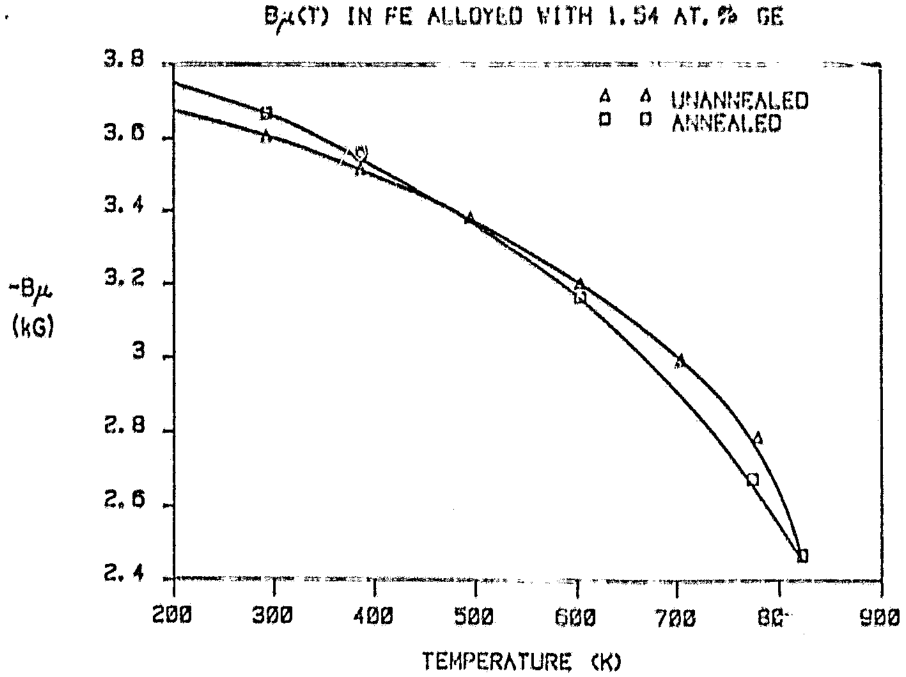

Figure 1

belief that strain plays a major role in determining the field seen by the μ^+.

Shear strains break the symmetry of the dipole lattice in Fe. With complete bcc lattice symmetry the diffusion of the μ^+ averages the dipolar fields to zero. However, shear strains could give a net dipolar field perpendicular to the magnetization. Computer simulations of these fields show that the net effect upon μ^+ is to decrease the precession frequency.[4]

The recent observation that uniaxial tension decreases B_μ in single crystals of Fe supports the motion of strain sensitivity to B_μ.

HIGH TEMPERATURE MEASUREMENTS

The problem of non-random sampling of sites by the μ^+ can presumably be overcome by providing the muons with sufficient thermal energy that kT is much greater than the attractive/repulsive potentials at interstitial sites adjacent to impurity atoms. Measurements at such high temperatures (approaching Tc) are fraught with difficulties. Assuming that the practical problem of constructing ovens which can maintain stable gradient-free high temperatures is overcome, one must also determine the magnetization of the alloy as a function of temperature. This varies rapidly near Tc, and the approximation heretofore used of scaling the magnetization curve of pure Fe to the M(T = 0K) and Tc values of the alloy probably is not sufficiently accurate. Therefore both precision μSR experiments and magnetization measurements will be necessary to fully exploit this area of study.

ACKNOWLEDGMENTS

These studies were performed at TRIUMF, which is operated by the University of Alberta, the University of British Columbia, Simon Fraser University, and the University of Victoria with the support of the Natural Sciences and Engineering Research Council of Canada. The experiments were supported in part by the National Aeronautics and Space Administration (CES and JJS) and the National Science Foundation DMR8007059 (WJK). We appreciate the help received from J. Brewer, P. Percival, T. Suzuki and Y. Uemura. A. T. Fiory and B. D. Patterson participated in the initial studies of Fe alloys.

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