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Are there Giant Magnetic Moments in Fe-nitrides?*

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Abstract

In this paper I will review the situation regarding the claims of giant magnetic moments in Fe–N films. This work is predominantly experimental at present but has serious implications for the theory of 3d magnetism if it proves to be correct.

1. Introduction

In a recent review article entitled ‘Magnetism of thin films and multilayers’, Mark Howson (1994) made the comment that “The study of magnetism is possibly the second-oldest profession in the world [and] unlike the oldest, interest has waxed and waned over the centuries”. Speaking as an *experimental magnetician* I am happy to say that these are exciting times in the study of magnetism and there is so much interesting research being done these days. It is impossible to cover the entire range in two papers. I decided to split my presentation between the transition metal 3d series and the rare-earth 4f series but one is then faced with the problem of deciding what to cover within each paper.

In the present paper I will discuss the question of whether or not Fe shows a *giant* magnetic moment in Fe–N thin films. In the following paper I will consider the 4f series and discuss the $\text{RNi}_2\text{B}_2\text{C}$ (R = rare-earth) magnetic superconductors (Cadogan 1997, present issue p. 1103).

2. Giant Fe Magnetic Moments?

The three ferromagnetic 3d elements are Fe, Co and Ni and their saturation magnetic inductions ($B_S = \mu_0 M_S$) are 2.15, 1.76 and 0.61 T, respectively, at room temperature (RT). These inductions correspond to atomic magnetic moments of 2.2, 1.72 and 0.62 μ_B , respectively. The highest RT bulk saturation induction in a 3d alloy system is 2.45 T for $\text{Fe}_{65}\text{Co}_{35}$, a member of the *permendur* family of magnets. Most people are probably familiar with the Slater–Pauling curve (Fig. 1) which shows the variation of average atomic moment with electron concentration. This curve gives an excellent account of experimentally determined magnetic moments in 3d alloy systems and shows a maximum average magnetic moment of around 2.5 μ_B .

* Refereed paper based on a contribution to the Sixth Gordon Godfrey Workshop on Recent Developments in Magnetism and Magnetic Materials, held at the University of New South Wales, Sydney, in October 1996.

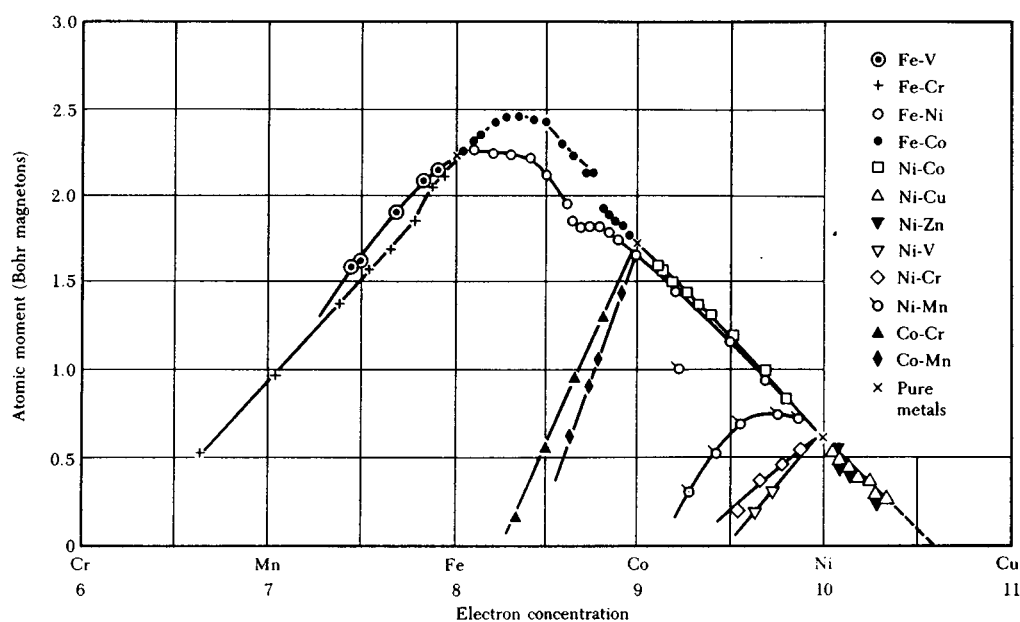


Fig. 1. Slater–Pauling curve showing average magnetic moment versus electron concentration in the transition metal series (reproduced from Kittel 1996).

In 1972, Kim and Takahashi (1972) from Tohoku University claimed an average Fe moment (at RT) of $3.0 \mu_B$ in Fe-nitride thin films, based on magnetometry measurements, but somewhat surprisingly little notice was taken of this claim, perhaps due to an unshakeable faith in the Slater–Pauling curve but more likely due to the well-known difficulties associated with making accurate measurements of magnetisation in thin films. However, in 1990 the Hitachi group of Sugita and co-workers (see Komuro *et al.* 1990) reported a value of $3.2 \mu_B$ for $\langle \mu_{Fe} \rangle$ in Fe–N films. This report sparked much work on the magnetism of Fe–N films in an effort to determine if such large magnetic moments were possible in alloy systems. (Of course, Fe in non-metallic oxides can reach $5 \mu_B$ in a high-spin configuration, although B_S reaches only 0.6 T since much of the volume is occupied by non-magnetic O^{2-} ions.)

To place this work in context we need to go back to 1951 when Jack (1994) was undertaking PhD work at Cambridge, studying phase formation in the Fe–N binary system. The solubility of N in bcc α -Fe is negligible but Fe–N phases can be obtained by heating α Fe in a mixture of NH_3/H_2 . At temperatures around 900–975 K, NH_3 is unstable relative to H_2 and N_2 and with a suitable catalyst, such as Fe, NH_3 decomposes to form N_2 at a very high effective pressure (the equilibrium pressure of N_2 at such temperatures is ~ 2400 atm). By contrast, fcc γ -Fe does dissolve N and was therefore used as a precursor to forming an α -Fe–N phase.

γ -FeN exists above 590°C with N randomly occupying octahedral interstices. By quenching γ -FeN from say 600°C one can form a bcc α' -Fe–N phase through a martensitic transformation. After suitable heat treatment the α' -Fe–N forms a mixture of α -Fe and α'' -Fe₁₆N₂, the latter comprising eight distorted bcc cells of

α -Fe with N occupying two of the 48 available octahedral interstices. α'' -Fe₁₆N₂ is tetragonal with the lattice parameters $a = 5.72 \text{ \AA}$ and $c = 6.29 \text{ \AA}$ and its crystal structure is shown in Fig. 2. There are three Fe sites in this structure: Fe(4e) is a *first* nearest neighbour of N (2a site); Fe(8h) is a *second* nearest neighbour of N and Fe(4d) is a *third* nearest neighbour of N.

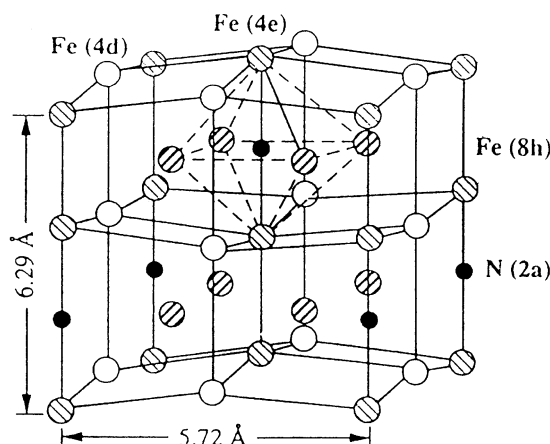


Fig. 2. Crystal structure of α'' -Fe₁₆N₂ (reproduced from Coey 1994).

The Fe–N system is no stranger to unusual magnetic behaviour. For example, cubic fcc γ -FeN is non-magnetic whereas cubic γ' -FeN is ferromagnetic with a Curie temperature of about 760 K and a saturation magnetisation of about 186 J/T/kg (emu/g for the non-SI among us). The only structural difference between these two γ phases is the arrangement of the N interstitial atoms: in γ -FeN the N atoms are disordered and randomly occupy the octahedral interstices whereas in γ' the N atoms are ordered. It seems that the ordering of the N atoms is enough to alter the Fe band structure sufficiently to support a sizable magnetic moment.

Once the α'' -Fe₁₆N₂ phase had been formed by Jack in 1951, Chikazumi studied its magnetic properties but found nothing unusual (cited by Coey 1993). The 1972 claim by Kim and Takahashi was based on Fe–N films formed by evaporating Fe onto glass in an N₂ atmosphere. The samples produced were multi-phase, containing mainly α -Fe and α'' -Fe₁₆N₂. These authors measured a sample B_S of 2.64 T, from which they deduced an induction of 2.76 T for the α'' -Fe₁₆N₂ phase, corresponding to $\langle \mu_{Fe} \rangle = 3.0 \mu_B$, but they were unable to repeat this measurement. There are a number of problems associated with work of this nature including:

- (i) ambiguity in identifying the α'' -Fe₁₆N₂ phase (there are eight binary phases in the Fe–N system),
- (ii) difficulty in determining the volume fraction, mass, and density of α'' -Fe₁₆N₂ in the sample (necessary to deduce B_S of α'' -Fe₁₆N₂ from the measured sample B_S), and
- (iii) lack of reliable B_S values for all Fe–N phases.

It is difficult to derive reliable magnetisation values from measurements on thin films and these are probably the reasons for the fact that the 1972 claim went largely unheard.

Komuro *et al.* (1990) deposited Fe–N films by molecular beam epitaxy (MBE) onto α -Fe which had been grown epitaxially onto $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$ (these authors claimed to see a chemical reaction between the Fe–N and InGaAs which produced FeAs_2 , hence the use of an α -Fe buffer). They reported $B_S = 2.90$ T for $\alpha''\text{-Fe}_{16}\text{N}_2$, corresponding to $\langle\mu_{\text{Fe}}\rangle = 3.2 \mu_{\text{B}}$. As $T \rightarrow 0$, $\langle\mu_{\text{Fe}}\rangle \rightarrow 3.5 \mu_{\text{B}}$. This group (Sugita *et al.* 1991) estimated a Curie temperature of $\sim 540^\circ\text{C}$ from fits to magnetisation curves obtained up to $\sim 400^\circ\text{C}$.

Numerous experiments were carried out around the world in an attempt to reproduce these remarkable findings but, by and large, most were unable to do so. The great problem facing experimentalists trying to resolve this controversy is that it has so far proved impossible to prepare single-phase *bulk* samples of $\alpha''\text{-Fe}_{16}\text{N}_2$. All samples prepared to date contain significant amounts of phases such as α -Fe and $\gamma\text{-FeN}$. For example, typical samples prepared by Wallace and co-workers (Wallace and Huang 1994; Huang *et al.* 1994*a,b*) contain 50–56% α'' , $\sim 15\%$ α -Fe and 30–35% $\gamma\text{-FeN}$. It is no surprise, therefore, that the reported $\langle\mu_{\text{Fe}}\rangle$ values for $\alpha''\text{-Fe}_{16}\text{N}_2$ span an enormous range from $2.3\text{--}3.5 \mu_{\text{B}}$. In Table 1 we summarise the results of magnetometry measurements made on a variety of $\alpha''\text{-Fe}_{16}\text{N}_2$ samples. As noted by Metzger *et al.* (1994) these data fall into two camps: those supporting the *giant moment* idea (thin film samples prepared by MBE, sputtering, medium and small particles [$6\text{--}9 \mu\text{m}$]) and those opposing the *giant moment* idea (foils $25\text{--}100 \mu\text{m}$, large particle [$30 \mu\text{m}$], N_2^+ -implanted films, dc-sputtered films). It seems that some thought should be given to determining whether or not these samples contain Fe oxides which could be responsible for the magnetisation values claimed (*vide infra*).

Table 1. Saturation inductions B_S , magnetisation M_S and average Fe magnetic moments of $\alpha''\text{-Fe}_{16}\text{N}_2$ prepared by various methods (all values are at RT)

| B_S (T) | M_S (J/T/kg) | $\langle\mu_{\text{Fe}}\rangle$ (μ_{B}) | Sample preparation | Ref. |
|-------------|---------------------------------------|--|------------------------------|------|
| 2.78 | 298 | 3.07 | Film (MBE) | a |
| 2.94 | 315 | 3.25 | Film (MBE) | b |
| 2.40 | 257 | 2.65 | Sputt. or plasma evap. films | c |
| 2.94 | 315 | 3.25 | Film (MBE) | d |
| 2.06 | no larger than $\alpha\text{-Fe}$ 220 | 2.27 | dc sputt. film | e |
| 2.94 | | 3.25 | Sputt. or plasma evap. films | f |
| 2.67 | 286 | 2.95 | Powder | g |
| 2.66 | 285 | 2.94 | Powder | h |
| 2.20 & 2.53 | 236 & 271 | 2.43 & 2.79 | Foil ($100 \mu\text{m}$) | i |
| 2.10 & 2.22 | 225 & 238 | 2.32 & 2.45 | Foil ($25 \mu\text{m}$) | i |
| 2.25 | 241 | 2.48 | Powder | i |
| 2.90 | 310 | 3.20 | Powder (+Mn) | j |
| 2.80 | 300 | 3.09 | Powder (+Mn) | j |
| 2.90 | 310 | 3.20 | Film (MBE) | k |
| <2.24 | <240 | <2.47 | Film (reactive sputt.) | l |
| <2.24 | <240 | <2.47 | Film (reactive sputt.) | m |

(a) Kim and Takahashi (1972), (b) Komuro *et al.* (1990), (c) Nakajima and Okamoto (1990), (d) Sugita *et al.* (1991), (e) Shoji *et al.* (1993), (f) Gao *et al.* (1993), (g) Wallace and Huang (1994), (h) Huang *et al.* (1994*a*), (i) Coey *et al.* (1994), (j) Bao *et al.* (1994), (k) Sugita *et al.* (1994), (l) Takahashi *et al.* (1994), (m) Takahashi *et al.* (1996).

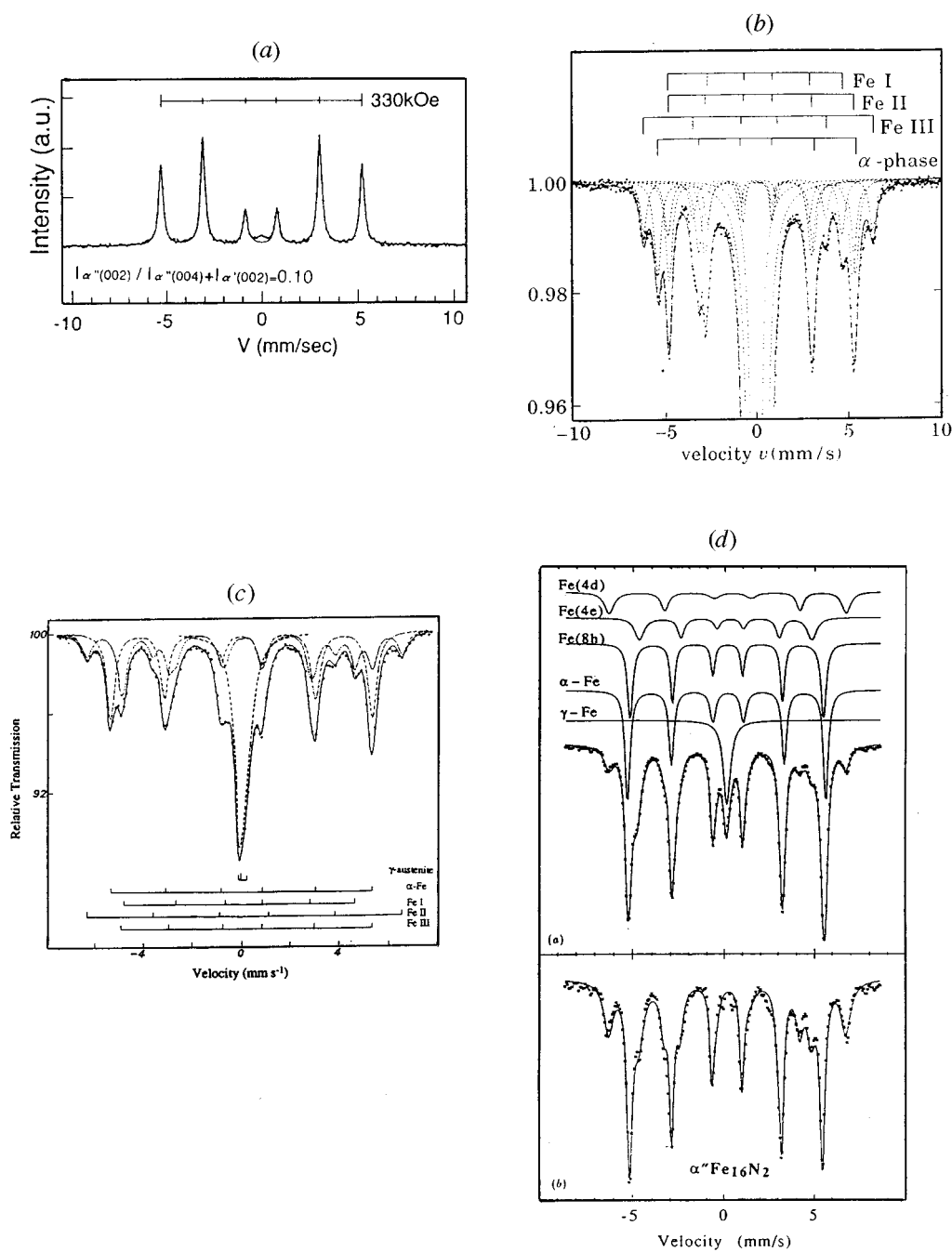


Fig. 3. A selection of ^{57}Fe Mössbauer spectra of α'' -Fe₁₆N₂ reproduced from (a) Sugita *et al.* (1994), (b) Hinomura and Nasu (1996), (c) Bao *et al.* (1994) and (d) Coey *et al.* (1994). (A B_{hf} of 48 T would yield a sextet with a splitting between the outer lines of ~ 15.5 mm/s).

3. Mössbauer Spectroscopy

Mindful of the difficulties associated with carrying out accurate magnetometry measurements on thin films, many workers turned to ^{57}Fe Mössbauer spectroscopy, reasoning that a $\langle\mu_{\text{Fe}}\rangle$ of $3.2\mu_{\text{B}}$ should produce a hyperfine magnetic field B_{hf} at the ^{57}Fe nucleus of about 48 T at RT (B_{hf} in $\alpha\text{-Fe}$ is 33.0 T at RT which is a well-known value used for calibrating Mössbauer drives). In principle, the Mössbauer effect should provide a reliable measure of the local Fe moment in the $\alpha''\text{-Fe}_{16}\text{N}_2$ films and, to a first approximation, the Mössbauer spectrum of $\alpha''\text{-Fe}_{16}\text{N}_2$ should comprise three magnetically split sextets in the area ratio 4:8:4, this being the Fe site population ratio in the $\alpha''\text{-Fe}_{16}\text{N}_2$ structure (Fig. 2). In Fig. 3 we show a selection of published Mössbauer spectra and we summarise the findings of the Mössbauer spectroscopy experiments in Table 2. The conclusions to be drawn from the Mössbauer results are:

- the average Fe magnetic moment in $\alpha''\text{-Fe}_{16}\text{N}_2$ is *almost exactly* the same as in $\alpha\text{-Fe}$ ($2.2\mu_{\text{B}}$);
- the magnetic moment at the Fe 4d site is larger than that in $\alpha\text{-Fe}$ by 20%, which is to be expected since the 4d site is quite far from the N interstitial, being only a third nearest neighbour and thus relatively unaffected by electron hybridisation effects with the N atom.

The Mössbauer spectra of $\alpha''\text{-Fe}_{16}\text{N}_2$ presented by Sugita *et al.* (1994) are significant because they show only one magnetically split sextet with a B_{hf} of 33.0 T at RT. These authors employed conversion electron Mössbauer spectroscopy in their study of thin film samples and so are only probing the surface regions. The observation of a single sextet with a field of 33.0 T is strongly suggestive of the formation of $\alpha\text{-Fe}$ ($B_{\text{hf}} = 33.0\text{ T}$) rather than $\alpha''\text{-Fe}_{16}\text{N}_2$ (Fig. 3a).

One interesting result is that of Hinomura and Nasu (1996) who observed two rather broad magnetic sextets in a Mössbauer spectrum of $\text{FeN}_{0.63}$ at 10 K. They reported a B_{hf} of 49 T for one of the sextets which corresponds to an Fe moment of about $3.3\mu_{\text{B}}$ in this NaCl-type Fe nitride. They also claimed that Mössbauer measurements carried out in an externally applied magnetic field indicate that this nitride is antiferromagnetic. However, it should be noted here that Fe oxides have B_{hf} values in the range 47–50 T at RT and it is possible that these observations are of oxides rather than nitrides. The avoidance of oxidation in such samples is by no means trivial.

An alternative hyperfine study is that of Zhang *et al.* (1996) who employed ^{57}Fe spin-echo NMR as the hyperfine probe rather than Mössbauer spectroscopy. As expected, these authors found that their nominal $\alpha''\text{-Fe}_{16}\text{N}_2$ phase consisted of $\alpha''\text{-Fe}_{16}\text{N}_2$, $\alpha\text{-Fe}$ and $\gamma\text{-FeN}$. The advantage of NMR is that the resonances from individual Fe sites can be quite sharp and, for example, $\alpha\text{-Fe}$ is readily observed from its sharp peak at 46.7 MHz (at 1.3 K). The NMR results yield maximum moments of $\langle\mu_{\text{Fe}}\rangle = 2.9\mu_{\text{B}}$ and $\mu_{\text{Fe}}^{4\text{d}} = 3.5\mu_{\text{B}}$ at 1.3 K (see Fig. 4).

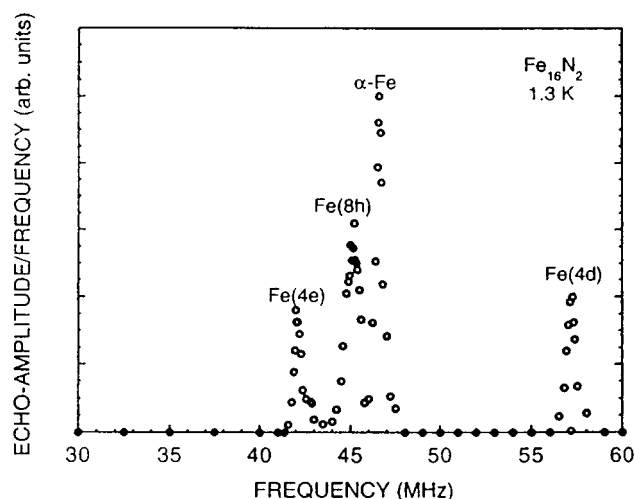
4. Band Calculations

Confirmation of giant Fe magnetic moments would have quite substantial consequences for band theory and the experimental reports prompted an acronymic frenzy of band calculations using all manner of methods and approximations. In Table 3 we summarise the main results of these calculations. Virtually all

Table 2. Average ^{57}Fe hyperfine fields, average Fe magnetic moments and corresponding values at the Fe 4d site in $\alpha''\text{-Fe}_{16}\text{N}_2$, determined by Mössbauer spectroscopy

All values are at RT unless otherwise stated. A conversion factor of 15 T/ μ_B (e.g. Cadogan 1996) was used to determine the moments from the hyperfine field values

| $\langle B_{\text{hf}} \text{ (T)} \rangle$ | $\langle \mu_{\text{Fe}} \rangle$ | $B_{\text{hf}}^{4d} \text{ (T)}$ | $\mu_{\text{Fe}}^{4d} \text{ (}\mu_B\text{)}$ | Reference |
|---|-----------------------------------|----------------------------------|---|---------------------------------------|
| 34.2 | 2.28 | 41.8 | 2.79 | Moriya <i>et al.</i> (1973) n.b. 78 K |
| 32.6 | 2.17 | 37.3 | 2.49 | Nakajima <i>et al.</i> (1989) |
| 33.3 | 2.22 | 40.0 | 2.67 | Nakajima and Okamoto (1990) |
| ~ 37 | ~ 2.5 | 46.0 | 3.07 | Sugita <i>et al.</i> (1991) |
| 33.6 | 2.24 | 40.6 | 2.71 | Coey <i>et al.</i> (1994) n.b. 15 K |
| 33.1 | 2.21 | 39.7 | 2.65 | Bao <i>et al.</i> (1994) |
| 33.0 | 2.20 | 39.5 | 2.63 | Coey (1994) |
| 34.1 | 2.27 | 41.2 | 2.75 | Coey (1994) n.b. 15 K |
| 33.0 | 2.20 | — | — | Sugita <i>et al.</i> (1994) |
| 32.5 | 2.17 | 39.1 | 2.61 | Takahashi <i>et al.</i> (1994) |
| 32.7 | 2.18 | 40.0 | 2.67 | Takahashi <i>et al.</i> (1996) |
| 33.0 | 2.20 | 39.0 | 2.60 | Hinomura and Nasu (1996) |

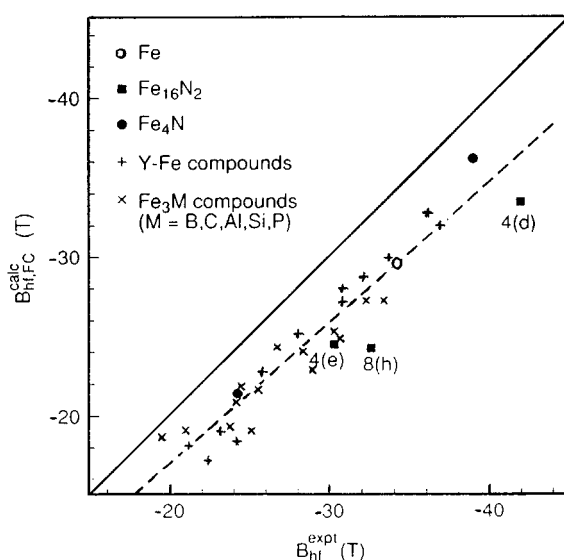
**Fig. 4.** Pulsed ^{57}Fe spin-echo NMR spectrum of $\alpha''\text{-Fe}_{16}\text{N}_2$ at 1.3 K (reproduced from Zhang *et al.* 1996).

calculated average magnetic moments fall in the range 2.3–2.5 μ_B , with only the LMTO calculations of Lai *et al.* (1994) yielding a larger moment of 2.85 μ_B , still well short of the experimental claims. The main conclusions to be drawn from the band calculations are:

- relative to $\alpha\text{-Fe}$, μ_{Fe}^{4e} is reduced by hybridisation of the Fe(3d) band and N(2p) orbitals, giving a moment of 2.13 μ_B ;
- μ_{Fe}^{4d} has an enhanced moment because it is the furthest away from the N site and has a larger Fe–Fe separation than in $\alpha\text{-Fe}$, giving a moment of 2.85 μ_B ;
- the orbital contribution to the Fe moment is small (0.07 μ_B); and

Table 3. Average Fe magnetic moments in α'' -Fe₁₆N₂ deduced from band calculations

| Method | $\langle \mu_{\text{Fe}} \rangle (\mu_{\text{B}})$ | Reference |
|---------------|--|-------------------------------|
| LMTO | 2.39 | Sakuma (1991, 1996) |
| LMTO | 2.42 | Ishida <i>et al.</i> (1992) |
| LMTO | 2.50 | Min (1992, 1993) |
| ASW | 2.42 | Matar (1992) |
| FLAPW | 2.37 | Coehoorn <i>et al.</i> (1993) |
| DV-X α | 2.27 | Miura <i>et al.</i> (1993) |
| LMTO | 2.45 | Coey <i>et al.</i> (1994) |
| APW | 2.40 | Sawada <i>et al.</i> (1994) |
| LMTO(LDA+U) | 2.85 | Lai <i>et al.</i> (1994) |
| OLCAO | 2.44 | Huang and Ching (1995) |
| SIC-LDA | 2.39 | Umino <i>et al.</i> (1996) |
| LDA | 2.35 | Umino <i>et al.</i> (1996) |

**Fig. 5.** Comparison of measured and calculated B_{hf} values in a number of alloys and compounds (reproduced from Coehoorn *et al.* 1993).

- LMTO calculations will produce $\langle \mu_{\text{Fe}} \rangle = 3.0 \mu_{\text{B}}$ but this requires an unreasonable volume expansion of 50% which would result in a rather low B_{S} of 2.0 T (the actual volume expansion, relative to α -Fe, is more like 5–10%).

At this point one should perhaps question the use of *standard* band calculational methods as applied to thin film samples. Would two-dimensional calculations yield unusually large magnetic moments?

5. Coehoorn's Question

Coehoorn *et al.* (1993) reviewed the state of band calculations on the Fe–N compounds with a view to answering the question: *Could the band calculations*

really be SO wrong? It is well known that band calculations generally underestimate the dominant Fermi contact contribution to B_{hf} by about 5 T and in Fig. 5 we show a plot of calculated B_{hf} values versus experimental values for a number of series of compounds including Fe, Y-Fe intermetallics, Fe_3X , $\text{X} = \text{B}, \text{C}$ etc. along with $\alpha''\text{-Fe}_{16}\text{N}_2$. A difference in B_{hf} of 5 T translates to a difference in Fe moment of about $0.3\text{--}0.35 \mu_{\text{B}}$. Thus, the band calculations could conceivably yield an Fe moment of up to $2.8 \mu_{\text{B}}$. The contribution of a $0.07 \mu_{\text{B}}$ orbital moment on the Fe translates to a change in B_{hf} of about 3 T, with the opposite sign to that of the dominant spin contribution, making the discrepancy even worse! The inescapable conclusion is that band calculations might underestimate $\langle\mu_{\text{Fe}}\rangle$ by $0.3\text{--}0.4 \mu_{\text{B}}$ but *not* by $1.2 \mu_{\text{B}}$!

6. Conclusions

The magnetometry studies carried out to date seem to be almost equally divided into the *for* and *against* camps; the ^{57}Fe Mössbauer work seems to be predominantly *against* as are the band calculations. My own reading of the situation is that there is no conclusive evidence for *giant magnetic moments* in $\alpha''\text{-Fe}_{16}\text{N}_2$, although no satisfactory explanation for the *pro* magnetometry claims has been advanced so far. Being a Mössbauer spectroscopist, and perhaps therefore somewhat biased, I am reasonably persuaded by the Mössbauer evidence against giant moments in $\alpha''\text{-Fe}_{16}\text{N}_2$. It seems that $\alpha''\text{-Fe}_{16}\text{N}_2$ does not have a giant magnetic moment on the Fe atom, although it is closer to strong ferromagnetism than $\alpha\text{-Fe}$. One of the Fe sites in the $\alpha''\text{-Fe}_{16}\text{N}_2$ structure does have a larger magnetic moment than $\alpha\text{-Fe}$. Band calculations support these views. Nevertheless, the controversy continues, fuelled by the recent claims by Sugita *et al.* (1996) of $\langle\mu_{\text{Fe}}\rangle = 3.5 \mu_{\text{B}}$.

Acknowledgments

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