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The Ferromagnet to Spin Glass Cross-Over: A Neutron Depolarisation Study

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Abstract

Mössbauer spectroscopy, magnetisation, susceptibility and neutron depolarisation have been used to investigate the evolution of magnetic order in a$-Fe_{90-x}Ru_xZr_{10}$. Ru additions cause exchange frustration leading to a rapid reduction in T_c and the appearance of transverse spin freezing at T_{xy}. At $x_c = 2.5$, T_c drops abruptly to meet T_{xy} and the system becomes a spin-glass with no long-range order. The magnetic phase diagram is fully consistent with results of mean field and Monte Carlo simulations. The anomalous peak in the neutron depolarisation signal observed close to x_c in this and other systems is shown to be due to the formation of a uniformly magnetised state induced by the 1 mT guide field.

Keywords: Frustration, Spin-glasses, Mössbauer spectroscopy, Neutron depolarisation, Metallic glasses

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Introduction

Magnetic materials with randomly competing positive and negative exchange interactions exhibit a rich variety of magnetic behaviour. Mean-field calculations [1] and numerical simulations [2] of partially frustrated magnetic systems of Heisenberg spins yield results consistent both with each other and with the experimental measurements. With no frustration, the material is a ferromagnet, with $\infty-$ranged correlations and collinear order. As frustration is introduced, the order at T= 0 becomes increasingly non-collinear and the magnetisation is reduced. The ground state appears to be an xy–spin glass co-existing with perpendicular ferromagnetic order (*i.e.* along the z–axis). On heating from zero temperature, the xy–spin glass melts at T_{xy} to form a collinear ferromagnet with substantial transverse degrees of freedom that fluctuate rapidly and time-average to zero. Further heating takes the system to T_c where the ferromagnet undergoes a conventional 3–D Heisenberg phase transition to a paramagnetic state. As the degree of frustration increases, the features characteristic of ferromagnetic order decline (both T_c and magnetisation fall) while spin-glass character becomes more pronounced (T_{xy} rises and irreversibilities at low temperatures become stronger). Eventually, T_c and T_{xy} meet, and further increases in frustration lead to a pure 3–D spin-glass with a transition temperature (T_{sg}) that is largely independent of the frustration level. Both calculations and simulations predict that the composition x_c at which T_c and T_{xy} meet marks a significant change in properties and there is some evidence from scaling analysis of fluctuations through x_c that a phase transition separates the spin-glass from the ferromagnet [2].

One of the clearest examples of a partially frustrated magnetic system is provided by amorphous iron–rich Fe–Zr alloys [3]. The frustration in these alloys arises through the distance dependence of the direct exchange interaction ($\mathcal{J}(r)$) coupled with short Fe–Fe contacts in the glass which lead to significant numbers of antiferromagnetic (AF) bonds that compete with the dominant ferromagnetic (FM) interactions. The effects of exchange frustration start to appear at $\sim$ 90 at.%Fe and become more pronounced with increasing iron content. All of the features described above have been observed in the $a-Fe_xZr_{100-x}$ system, and quantitative agreement with the predictions has been obtained [3]. Unfortunately, some impurities (in this case Zr) must be present to stabilise a metallic glass, and the FM–SG boundary is expected to lie near x = 94.5 at.% Fe, or about 1.5 at.% beyond the limits of glass stability. The truncated phase diagram has led to a number of other explanations being offered for the magnetic behaviour. These invariably appeal to the presence of magnetically distinct clusters that freeze at some reduced temperature, and destroy the ferromagnetic order established at T_c [4,5,6]. A more complete discussion of the limitations of these models has been presented elsewhere [3]; we note here that there is no evidence for the existence of the distinct spin populations demanded by the models, and that the ferromagnetic order clearly survives to T= 0 and is therefore not destroyed by the second ordering event.

The extrapolated FM–SG boundary in a$-Fe-Zr$ lies well beyond the stability limits of any iron-based metallic glass system, so that it is extremely unlikely that this, or any other iron-based binary glass, will provide access to the cross-over region. However, the compositional versatility of glasses permits extensive chemical changes at essentially constant structure, without the risk of transformations or the precipitation of impurity phases, making a ternary system an attractive alternative route. Of all additions tried so far, ruthenium is by far the most promising [7]. This element is non-magnetic, so that we retain the simplicity of having only one magnetic species in the glass, and the ternary system is an excellent glass-former. Ru additions lead to a very rapid increase in frustration. T_c in $a-Fe_{90-x}Ru_xZr_{10}$ drops at ~30 K/at%. The FM–SG boundary lies at ~2.5 at.% Ru, and by $x = 3$ no trace of long-range ferromagnetic order remains [8].

The work presented here is intended to characterise the Ru-induced changes in the magnetic order, concentrating especially on the FM–SG cross-over region. Magnetisation, neutron depolarisation and Mössbauer measurements were used to probe the magnetic correlations on macro-, meso- and micro-scopic length scales. Our results confirm the destruction of magnetic order and show that by $x \geq 2.5$, no long-range ferromagnetic order is present above 2K. The magnetic phase diagram is fully consistent with the homogeneous exchange-frustration models introduced above [1,2,3]. We also investigate the region around x_c and show that the apparent formation and collapse of ferromagnetic order on cooling is an artefact of the measurement technique.

Experimental Methods

The alloys were prepared by arc-melting the appropriate ratio of pure elements (Fe: 99.95%, Zr: 99.5% and Ru: 99.9% pure) under Ti-gettered argon to yield $\sim 3g$ ingots. Melt-spinning was carried out under a partial pressure of Helium onto a copper wheel at 55 m/s. Absence of crystallinity was confirmed using Cu$-K_\alpha$ powder x-ray diffraction and room temperature Mössbauer spectroscopy.

A susceptibility system with a closed-cycle fridge was used to record χ_{ac} *vs.* T down to 12K. Magnetisation data were obtained in fields of up to 9 Tesla over the temperature range 2 – 300 K. Mössbauer measurements were made on a constant acceleration spectrometer with a 1GBq ^{57}Co**Rh** source calibrated using an $\alpha-$Fe foil. Samples were mounted in a vibration-isolated closed-cycle fridge for spectra at temperatures down to 12K.

Neutron depolarisation data were obtained using the DUALSPEC triple-axis spectrometer at AECL, Chalk River. Initial polarisations of ~96% at λ =0.237 nm were achieved with Cu_2MnAl single crystals as polariser and analyser. Measurements were made between 2 and 300K in a

1 mT guide field, on stacks of one or more 5mm lengths of 20μm thick ribbons. A set of three orthogonal pairs of 1m diameter Helmholtz coils was used to provide guide fields oriented along X (parallel to the neutron flight path), Y (horizontal and parallel to the long axis of the sample) and Z (vertical). These allowed all three components of the sample magnetisation to be probed so that variations in magnetic texture could be detected.

Basic Characterisation

χ_{ac} measurements show a steady reduction in T$_c$ (taken as the point of maximum slope on a plot of χ_{ac} *vs.* T) and an evolution from ferromagnetic behaviour to a spin-glass-like cusp with increasing Ru content. The cusp develops at $x = 2.35$ and there is an abrupt drop between $x = 2.35$ and $x = 2.5$, where T$_c$ falls by 60 K, followed by a more gradual decline. Modified Arrott plots (using standard 3-D Heisenberg exponents) yield ordering temperatures in accord with those derived from χ_{ac} and Mössbauer measurements for $x \leq 2.35$. Beyond this point no spontaneous magnetisation is observed at any temperature. We therefore conclude that the FM–SG boundary lies in the range: $2.35 \leq x_c \leq 2.5$. Magnetisation curves at 5 K show a steady decline with increasing Ru content (Fig. 1), with a gradual increase in high field slope and coercivity. There is no discontinuity in behaviour at x_c.

Mössbauer spectra for these materials exhibit the usual broadened 6-line pattern below the ordering temperature. The decline in average hyperfine field ($< B_{hf} >$) (and hence iron moment) with increasing Ru content is far less than that observed in the magnetisation, reflecting the expected increase in non-collinearity as exchange frustration drives the system towards the spin-glass. $< B_{hf} >$ *vs.* x shows no break in slope at x_c, confirming that the breakdown in long-range order is not related to a collapse in the iron moment. The temperature dependence of $< B_{hf} >$ in Fig. 2 shows two remarkable features. First, an abrupt change in behaviour occurs between $x = 2.0$ and $x = 2.2$, significantly below x_c. Second, for $0.5 \leq x \leq 2.0$ there is a clear break in the temperature dependence at ~80 K. We found that $< B_{hf} >$(T) can be fitted within 0.1 T by a combination of a modified Brillouin function [9] and a linear term that starts at T$_{br}$, the break point [8]. Fitting the data with two Brillouin functions adds more parameters and tends to degrade the fit quality.

The phase diagram in Fig. 3 summarises the characteristic temperatures determined by the various techniques used here. The boundary between ferromagnetic and spin-glass behaviour is clearly visible as an abrupt drop in ordering temperature and a loss of long-range order. T$_{br}$ rises to meet T$_c$ with increasing Ru content and they meet at x_c, behaviour consistent with T$_{br}$ being associated with transverse spin freezing. However, Mössbauer measurements in an applied field are needed before T_{br} can be conclusively identified with T_{xy}. The parallels between the phase diagram in Fig. 3 and that of the binary a-Fe–Zr system [3] are quite striking. Up to $x = 2.0$ the rate of decline in T_c and the composition dependence of T_{xy} are very similar. Indeed if a Mathon plot of T_c^2 $vs.$ x is made, it predicts a loss of magnetic order at $x = 4$ in much the same way as is claimed for a$-Fe - Zr$ [6]. However in the Ru-doped system we can achieve much higher levels of frustration and so observe that magnetic order is not lost. The decline in T_c is cut off by T_{xy} as the spin-glass is formed and T_{sg} is largely independent of x. These results are in full agreement with theoretical predictions for exchange frustration.

Window fits [10] to the Mössbauer spectra yield strongly bimodal hyperfine field distributions $P(B_{hf})$, and while this structure may simply be mathematical in origin [3], it is clearer here than in any other alloy system that we have studied. The bimodal structure is not associated with the break in temperature dependence at T_{br}, as it is present in samples that do not exhibit a break point. Were we to argue that the magnetic behaviour in this system arose from a cluster–matrix competition, then it would be natural to associate the low-field feature in $P(B_{hf})$ with the clusters and the stronger, high-field feature with the matrix. This assignment fails for two reasons. (1) We observe no significant shift in the position of the low-field component on passing through T_{br} so this component cannot be associated with the freezing of clusters. (2) The area of the low-field feature never dominates the distribution, even as we cross into the spin-glass. The low-field feature is always a minority component and likely to be a mathematical artefact.

The most direct evidence for the disappearance of long-range order comes from neutron depolarisation data [8]. Ordering temperatures for $x < x_c$ agree with values determined by other methods and are included in Fig. 3. The depolarisation caused at T=0 decreases with increasing Ru far more rapidly than either the magnetisation or $< B_{hf} >$ (Fig. 4). We attribute this to a rapid reduction in domain size from $1.64\pm0.01\,\mu m$ at $x = 0$ to $0.4\pm0.1\,\mu m$ by $x = 2$. For $x > 3$ no depolarisation is observed at any temperature, ($\Delta P < 5 \times 10^{-4}$) and we can rule out the presence of ferromagnetic correlations on a length scale greater than 10nm. This confirms our conclusion that for $x \geq 2.5$ the system is a spin glass.

The FM – SG cross-over region

The change in behaviour at x_c is remarkably sharp. Over a range of ~0.2 at.% in Ru content, T_c changes by nearly a factor of 2 and all long-range order is lost. We emphasise that this is solely an exchange effect, as Mössbauer spectra show that there is no significant change in either the average iron moment or its distribution through this region.

The critical region is the only place that there is a significant difference in ordering temperature determined by the various techniques, and then only for the Mössbauer data at $x = 2.2$ and 2.35. The Mössbauer values lie ∼30 K below the other methods. While it is easy to attribute an upward shift in the Arrott values to the large applied field used in their measurement, it is far less obvious that the 1 mT and 0.5 mT used in the depolarisation and χ_{ac} measurements could have a significant effect. However, in view of the field effect described below, it is possible that some shift is present. Furthermore, as the cusp in χ_{ac} develops there is an associated ambiguity in the definition of T_c. It is not clear at which composition it becomes appropriate to switch from using the point of maximum slope to define T_c (used below x_c), to the the peak of the cusp (used above x_c). No such ambiguity is present in the Mössbauer data and the onset of magnetic splitting marks ordering in both spin-glass and ferromagnetic systems. It is likely that χ_{ac} overestimates T_c for these two alloys.

For x =1 and 2, we previously observed a striking recovery in the neutron depolarisation below about 80K [8] and suggested that a stress-driven re-orientation of the domain magnetisation might be an explanation. This was consistent with changes in the line intensities in the Mössbauer spectra of these alloys. However, measurements of all three components of the depolarisation signal do not support such a rotation. Furthermore, as we move into the cross-over region the recovery structure becomes even more pronounced, even as the maximum effect weakens. For the $x = 2.35$ sample shown in Fig. 4 there is a striking peak at 120 K with only weak depolarisation observed below 80 K. We observed such peaks as far as $x = 3$. Similar behaviour has been reported for $a-(Fe_{0.3}Mn_{0.7})_{75}P_{16}B_6Al_3$ [11], $Au_{84}Fe_{16}$ [12] and $a-(Fe_{0.15}Ni_{0.85})_{75}P_{16}B_6Al_3$ [13]. In this last study, the polarisation recovery evident in our less heavily Ru–doped samples was also reported.

For a multi-domain sample the series of brief precessions of the neutron moment in each domain leads to a polarisation that decays as: $P = \exp(-\alpha\lambda^2)$ with $\alpha = \frac{1}{2}c^2 < B_\perp^2 > d\delta$. d is the sample thickness, δ is the mean domain size along the neutron flight path, $< B_\perp^2 >$ is the mean square domain magnetisation perpendicular to the beam polarisation, and c has the value $4.633 \times 10^{14} m^{-2}T^{-1}$ [14]. Given this expression, the simplest explanation for a depolarisation signal that fades on cooling is that either or both of the domain size δ or the domain magnetisation, B, are decreasing. Lorentz microscopy studies (albeit of less frustrated alloys), showed no evidence of domain shrinkage on cooling, and attributed the irreversibilities observed at low temperatures in these systems to pinning of domain walls [15]. We therefore eliminate changes in domain size from consideration. A reduction in magnetisation on cooling could be considered attractive for many reasons. As we move through the composition range around x_c where the peak occurs, the saturation magnetisation of the samples clearly falls. The formation and subsequent collapse of a ferromagnetic state would be consistent with the various cluster models identified in the introduction [4,5,6]; however such agreement is only found in the extremely narrow composition range around the FM–SG boundary, and appears coincidental. Alternatively, results of a homogeneous 2–D 'local mean field' simulation [16] have been used to support a canting driven loss of magnetisation at low temperatures. Apart from the inconsistency in the use of this 2–D model (the canting transition is only observed on heating, but it is interpreted as a cooling curve) we repeated the simulations in the course of our own 3–D Monte Carlo work [2] and found that the 'local mean field' model exhibits extremely poor dynamics and is easily trapped in local minima. The canting transition is a artefact of such trapping: a system quenched into a low magnetisation state remains there until the temperature is raised enough for it to jump to the equilibrium high magnetisation state. A drop in magnetisation was never observed on cooling. Our own 3–D simulations showed no evidence for a loss of magnetisation; on the contrary, we observed a consistent increase in magnetisation on cooling for all those systems that initially entered a ferromagnetic state. Finally, is is not clear that the spontaneous loss of a ferromagnetic state on cooling is thermodynamically reasonable.

If the domain size is fixed, and ferromagnetic order does not decay, where does the peak in the depolarisation signal come from? To answer this question we turn to the full polarisation data shown in Fig. 5. For an isotropic domain distribution, the depolarisation signal should be independent of the direction of polarisation of the incident neutron beam, and this was generally observed for the ferromagnetic samples. However Fig. 5 shows that the Z signal is always the weakest, indicating that the magnetisation tends to lie in the horizontal plane defined by the ribbon long axis and surface normal. This is consistent with low-temperature Mössbauer spectra recorded in zero applied field which also showed a clear tendency for the moments to lie perpendicular to the ribbon plane, *i.e.* along X. The data shown in Fig. 5 were obtained on heating, following cooling in the 0.5 mT stray field along Z from the guide boxes. We attribute the differences between the X and Y channels to imperfect guide fields that were not truly orthogonal for these axes. A conventional interpretation would call for the development of a ferromagnetic domain state on heating, followed by its collapse at T_c. Similar curves were obtained on cooling and presumably would be explained in the same way. However, a comparison of the depolarisation in Fig. 5 with the Mössbauer data in Fig. 2 shows that the signal persists about 40 K above the temperature at which the hyperfine field, and hence all spontaneous magnetic order, disappears. It is therefore extremely unlikely that conventional ferromagnetic order is causing the depolarisation signal, especially in view of the excellent agreement in ordering temperatures observed at less frustrated compositions (Fig. 3).

Some small guide field is essential to maintain the neutron polarisation, and it can also be used, as here, to control the polarisation direction. The field of 1 mT used here is typical, and generally does not significantly influence the sample magnetisation, especially when samples have saturation magnetisations of 1–1.5 T. For such samples the guide field may bias the domain magnetisations slightly but the dominant effect is still the randomising of the polarisation due to transits through multiple, uncorrelated domains. Even quite severe bias will not suppress the signal. For samples near x_c the situation is totally different. The spontaneous magnetisation is close to or actually zero, so the unperturbed sample can not affect the beam. However, both *dc* and *ac* susceptibility yield significant signals for these materials, and modest magnetisations are readily achieved by quite small fields. The origin of this magnetisation is easily understood. Both numerical [2] and analytical [1] work show that at the FM–SG boundary, 75–85% of the exchange bonds are still ferromagnetic, so that a field applied near T_{sg} can elicit a significant ferromagnetic response. At higher temperatures, thermal fluctuations dominate and no polarisation of the spins is possible, while at low temperatures frustration effects dominate, again preventing polarisation. In small fields the frustration prevents the field cooling of a polarisation, and χ_{dc} follows the decline of χ_{ac}. Our explanation of the depolarisation peak observed in samples close to x_c is therefore as follows: it is an artefact of the guide field that induces a weak uniform polarisation of the sample.

To test this model we cooled a $100\mu m$ thick sample of $a-Fe_{93}Zr_7$ in a field of 76 mT parallel to Y and recorded the signal shown in Fig. 6 on heating in the orthogonal guide fields. This material is on the FM side of x_c and shows only a weak ($\sim$3%) depolarisation signal [17]. Two features are immediately apparent: (i) there is no signal in the Y channel at any temperature confirming that the sample is indeed polarised along Y and that the decay of this polarised state does not involve the formation of domains; (ii) the polarised state is uniform as the neutrons are able to complete a full 2π rotation within the sample. As the temperature is increased, the magnetisation relaxes and the neutrons complete progressively smaller fractions of a rotation. Similar results were obtained for a$-Fe_{90}Sc_{10}$ which is a spin-glass and does not depolarise the beam at any temperature [18], however the internal magnetisation is weaker and we did not obtain a complete inversion of the neutron polarisation. The $a-Fe_{93}Zr_7$ data are shown here because the neutron polarisation is clearly rotated by a uniform field, and not scrambled by domains.

For the uniformly magnetised case, the behaviour of the neutron polarisation is given by: $P = \cos(cd\lambda B)$ where the symbols have the same significance as above. Using this to fit the data in Fig. 6 yields an internal field of 0.57 T. The vertical scales in Fig. 5 were calculated using this model. The dashed lines also shown in Fig. 5 are zero-field-cooled magnetisation curves obtained on heating in 1 mT for the same samples. The magnetisation curves were rescaled by up to 30% (see caption) which is a minor correction in view of the fact that the neutron depolarisation data were obtained in fields of $\pm X$, $\pm Y$ and $\pm Z$ at each temperature, rather than the static value used in the magnetometer. Despite these limitations, the agreement in shape, position and amplitude is remarkably good. An examination of the a$-Fe-Mn$ [11] and a$-Fe-Ni$ [13] data suggests that uniform polarisations of 16 mT and 10 mT respectively would also account for those observations.

Conclusions

$a - Fe_{90-x}Ru_xZr_{10}$ provides a clear example of the evolution from ferromagnet to spin-glass under the influence of exchange frustration. Two magnetic transitions are observed, the first at T_c as ferromagnetic order appears, while at the second (T_{xy}) transverse degrees of freedom freeze to form a co-existing xy-spin-glass. There is a clear break in behaviour at the FM–SG boundary with $x_c \sim 2.5$ at.%Ru. At x_c, T_c and T_{xy} merge and the system becomes a spin-glass with no ferromagnetic order. The loss of ferromagnetic correlations is confirmed by neutron depolarisation. We have also shown that the maximum in the depolarisation signal seen near x_c in this and other systems does not reflect the formation and decay of ferromagnetic order, but rather is an artefact of the guide field used in the measurement.

Acknowledgments

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Figure captions

Figure 1 Magnetisation curves for a$-Fe_{90-x}Ru_xZr_{10}$ measured at 5 K.

Figure 2 $< B_{hf} >$ vs T for a$-Fe_{90-x}Ru_xZr_{10}$ showing break in slope at $\sim$80 K for $0.5 \leq x \leq 2.0$. Solid lines are fits to the model described in the text.

Figure 3 Magnetic phase diagram for a$-Fe_{90-x}Ru_xZr_{10}$ showing the abrupt change at $x_c = 2.5$ as the system becomes a spin glass. Note how the decline in T_c is cut off by T_{br} ($\equiv T_{xy}$).

Figure 4 Normalised neutron depolarisation data for a$-Fe_{90-x}Ru_xZr_{10}$ obtained on cooling (∇) and heating ($\Box$) in a guide field of 1mT in the Z direction. Notice the strong maximum in the signal from the $x = 2.35$ sample and the absence of a depolarisation signal at low temperatures.

Figure 5 Comparison of neutron depolarisation signal (points) and low-field magnetisation data (dashed lines) for zero-field cooled a$-Fe_{90-x}Ru_xZr_{10}$ in the region around $x_c = 2.5$. The vertical scale was calculated assuming a uniformly magnetised sample (see text) and the magnetisation was re-scaled for two of the samples as indicated. Note the excellent agreement between the magnetisation and depolarisation data.

Figure 6 Neutron polarisation obtained on heating for a 100 μm thick sample of a$-Fe_{93}Zr_7$ field cooled in B_Y =76 mT. At 10K the neutron moment completes a full 2π rotation within the sample. The sample is cooled into a uniformly magnetised state which decays on heating without forming domains.

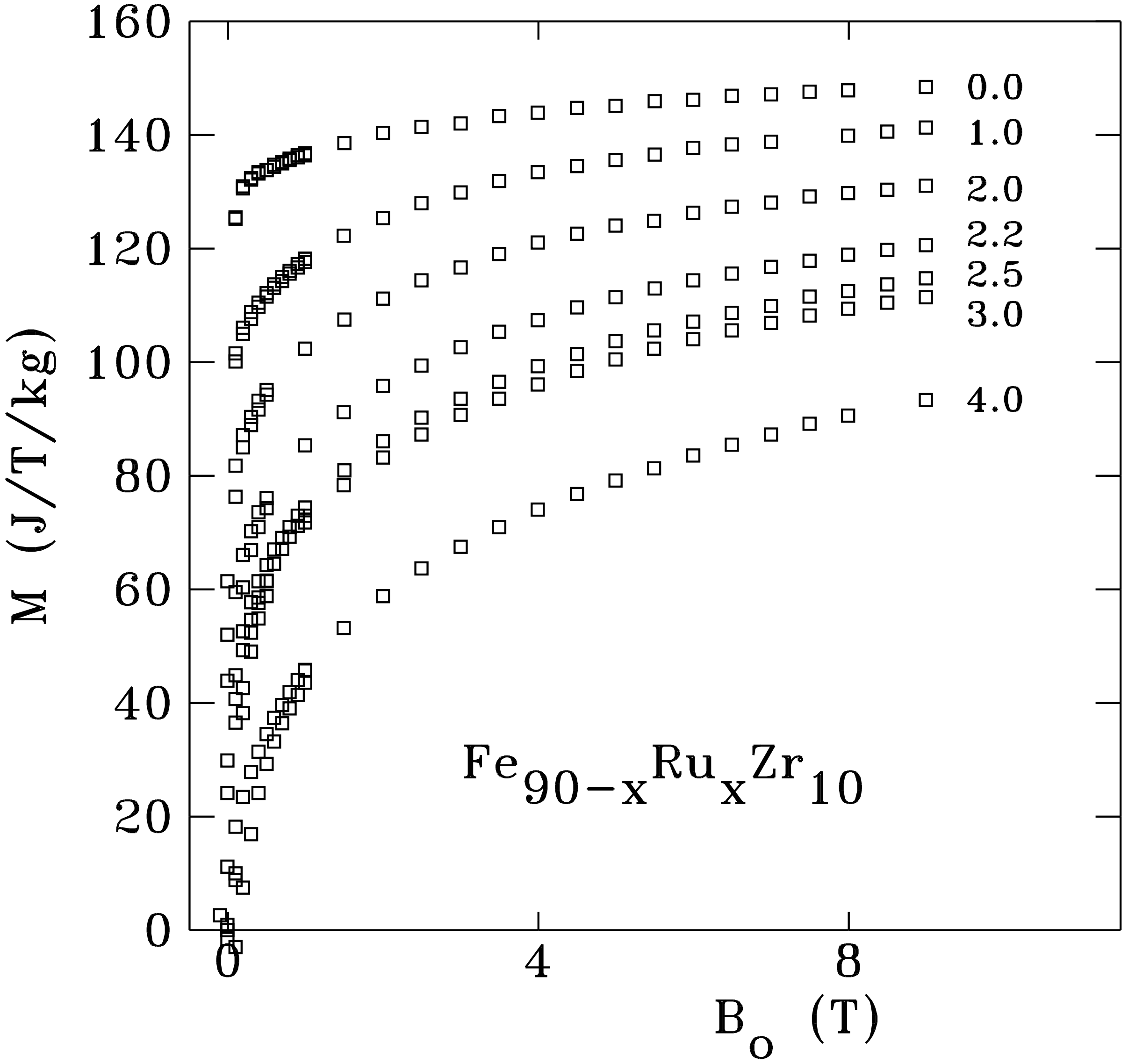
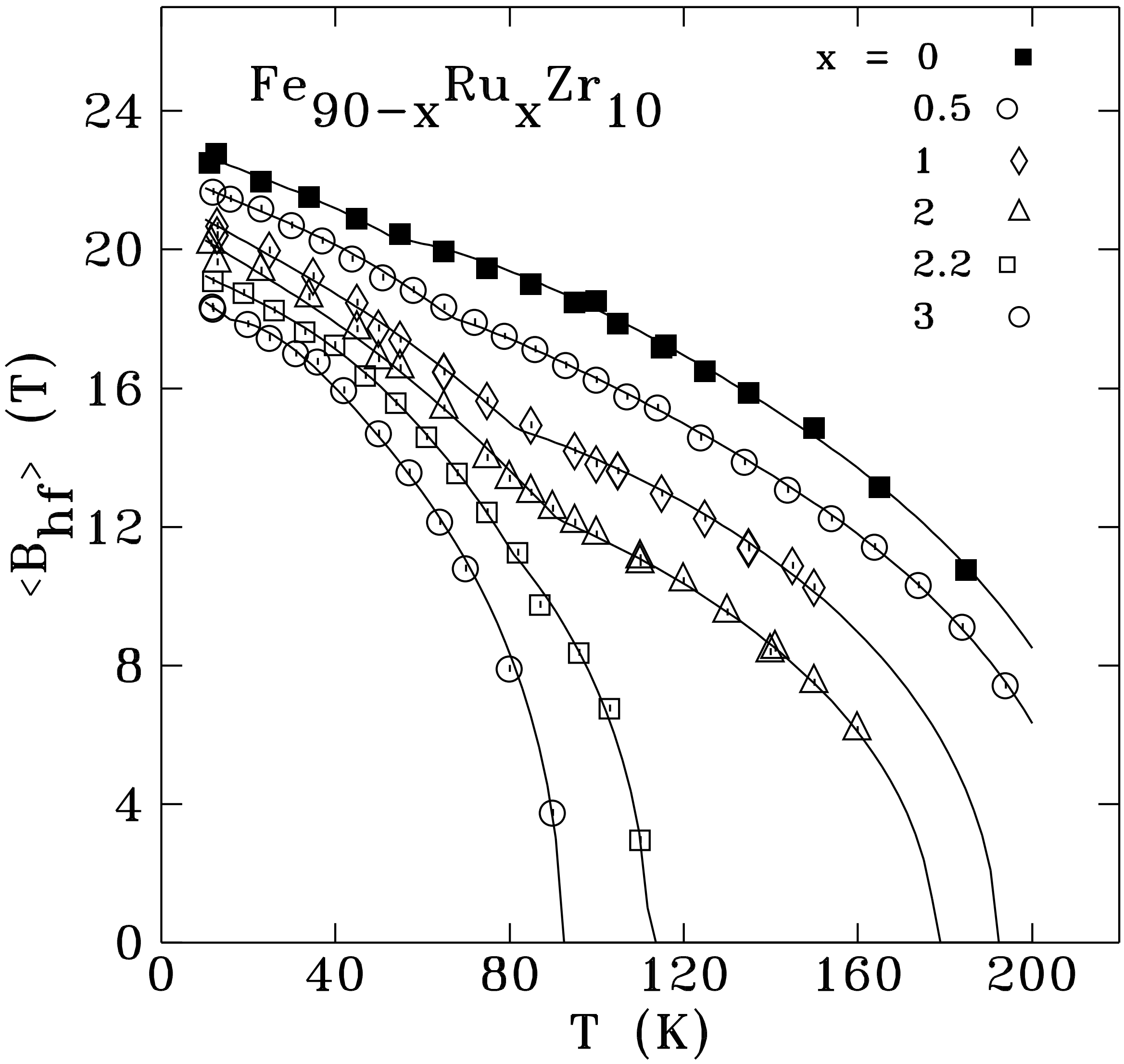


Figure 1: Ryan, Tun and Cadogan, ICM-97



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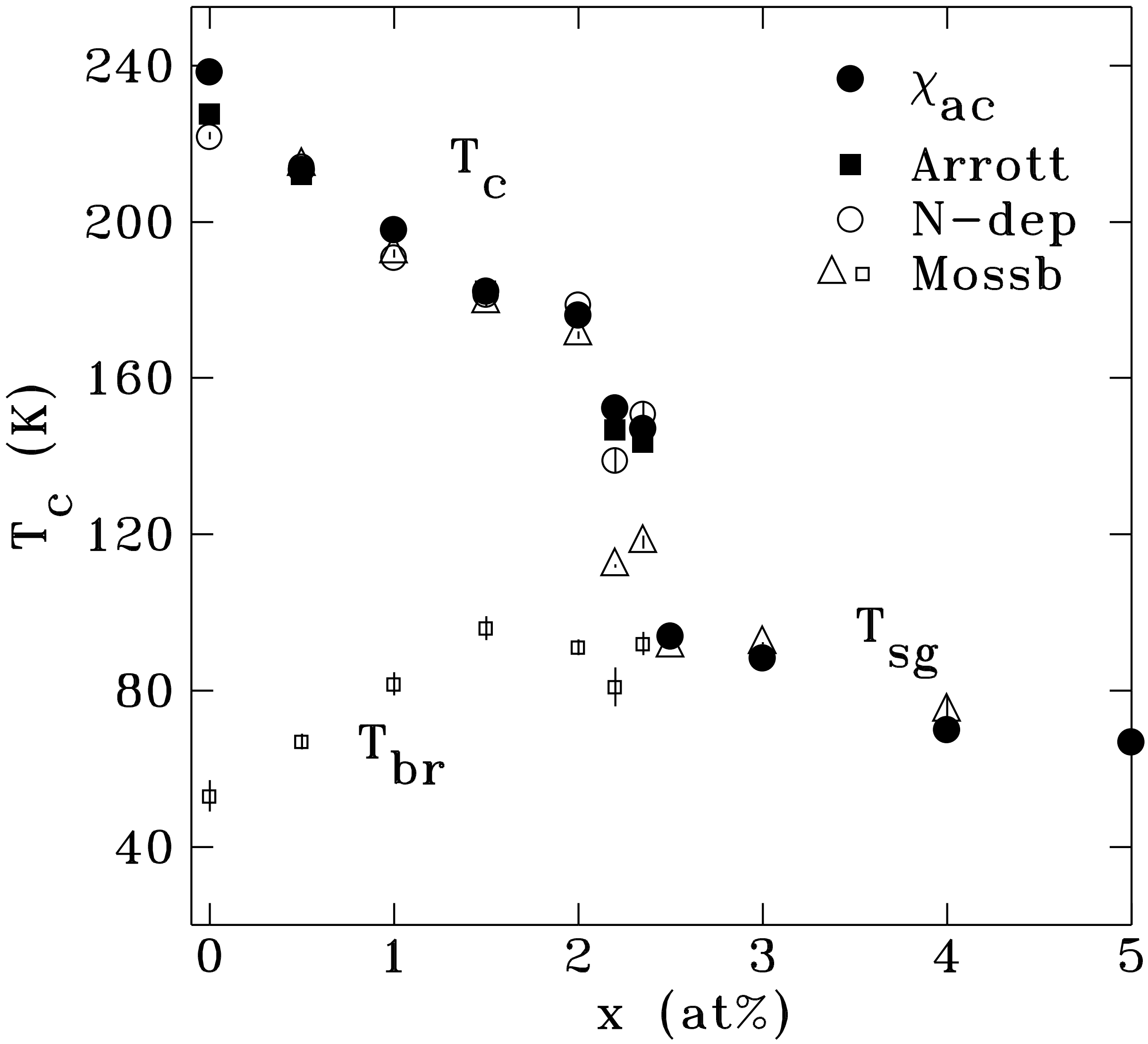

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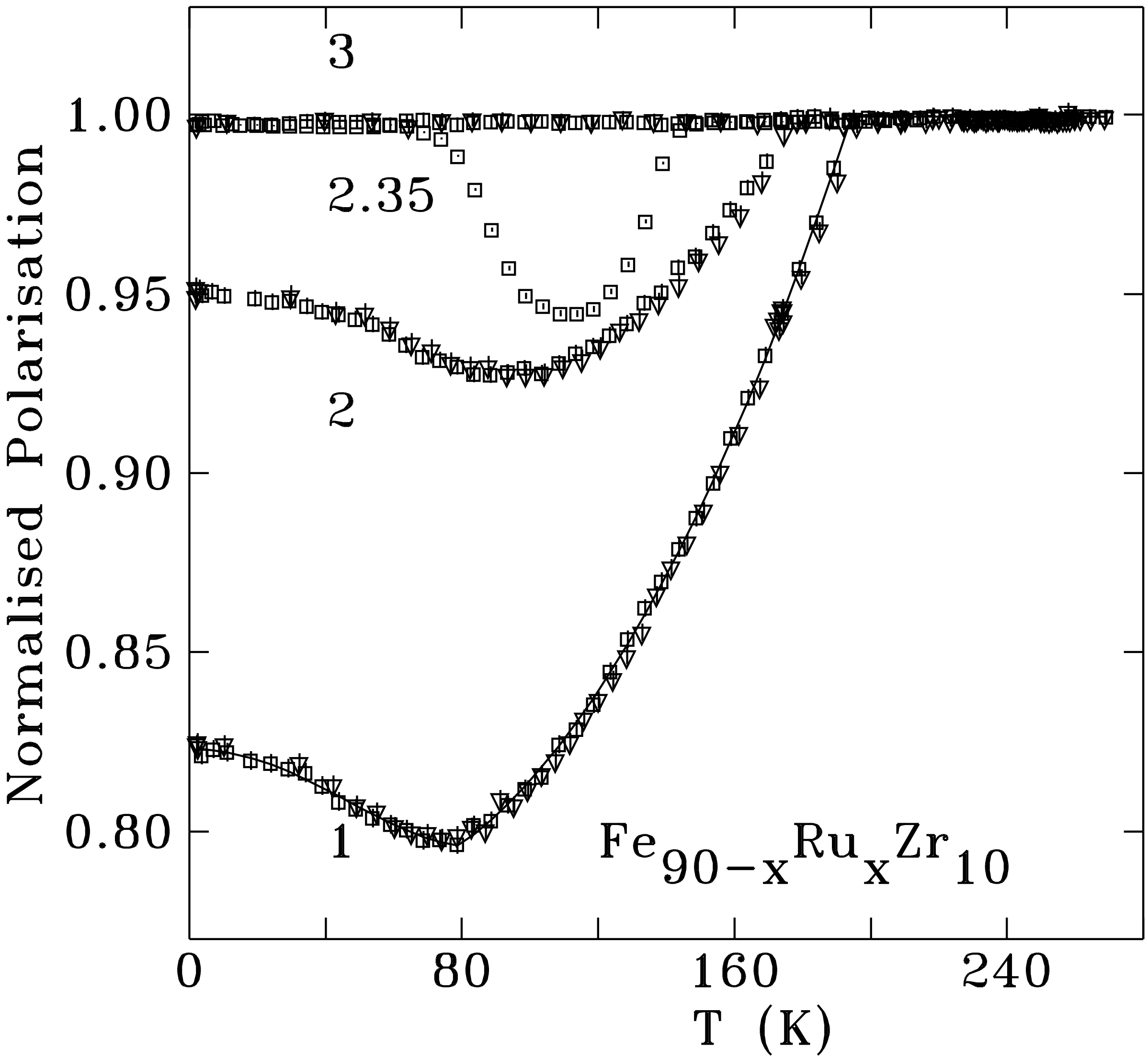

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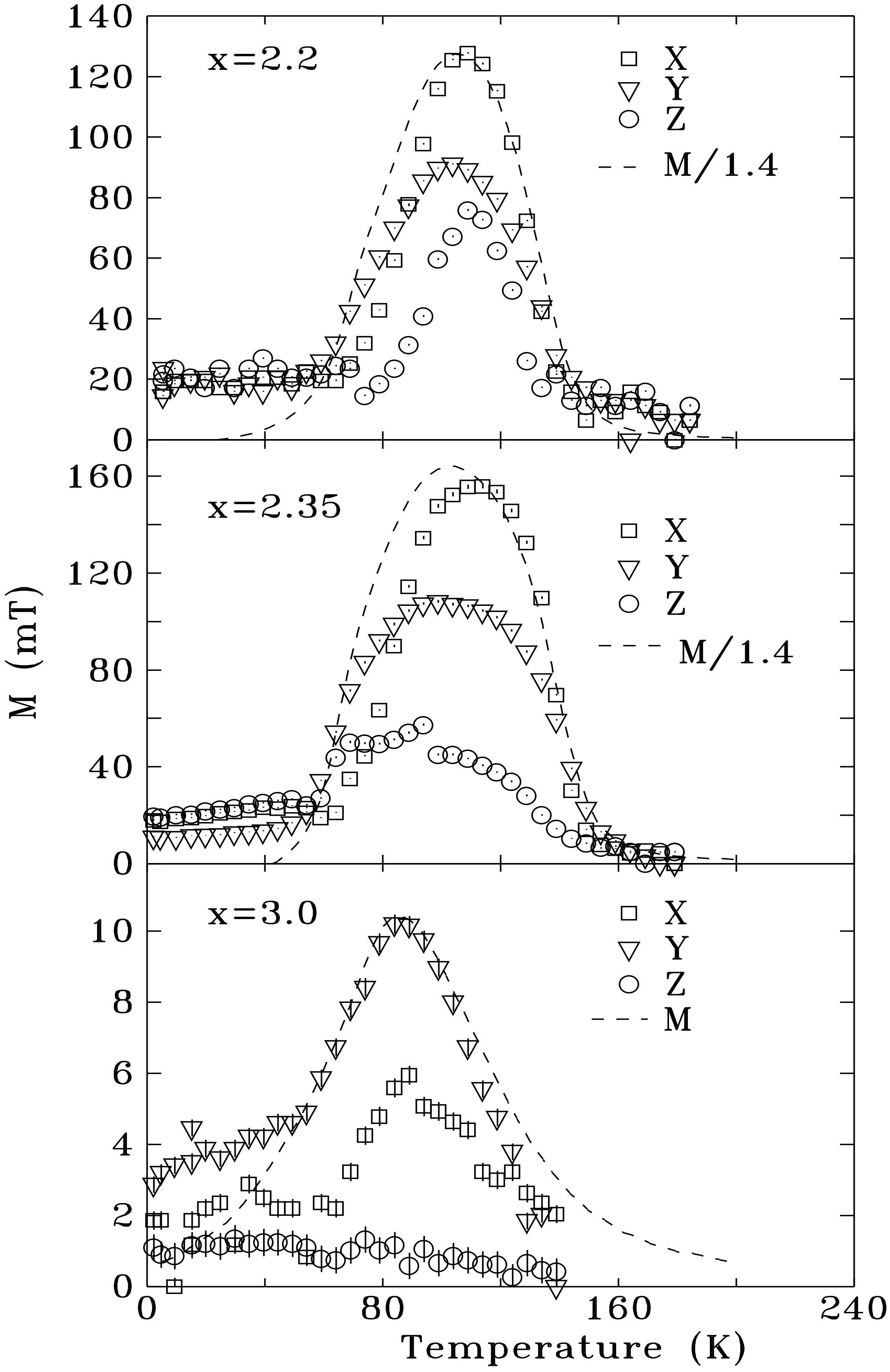

Figure 5: Ryan, Tun and Cadogan, ICM-97

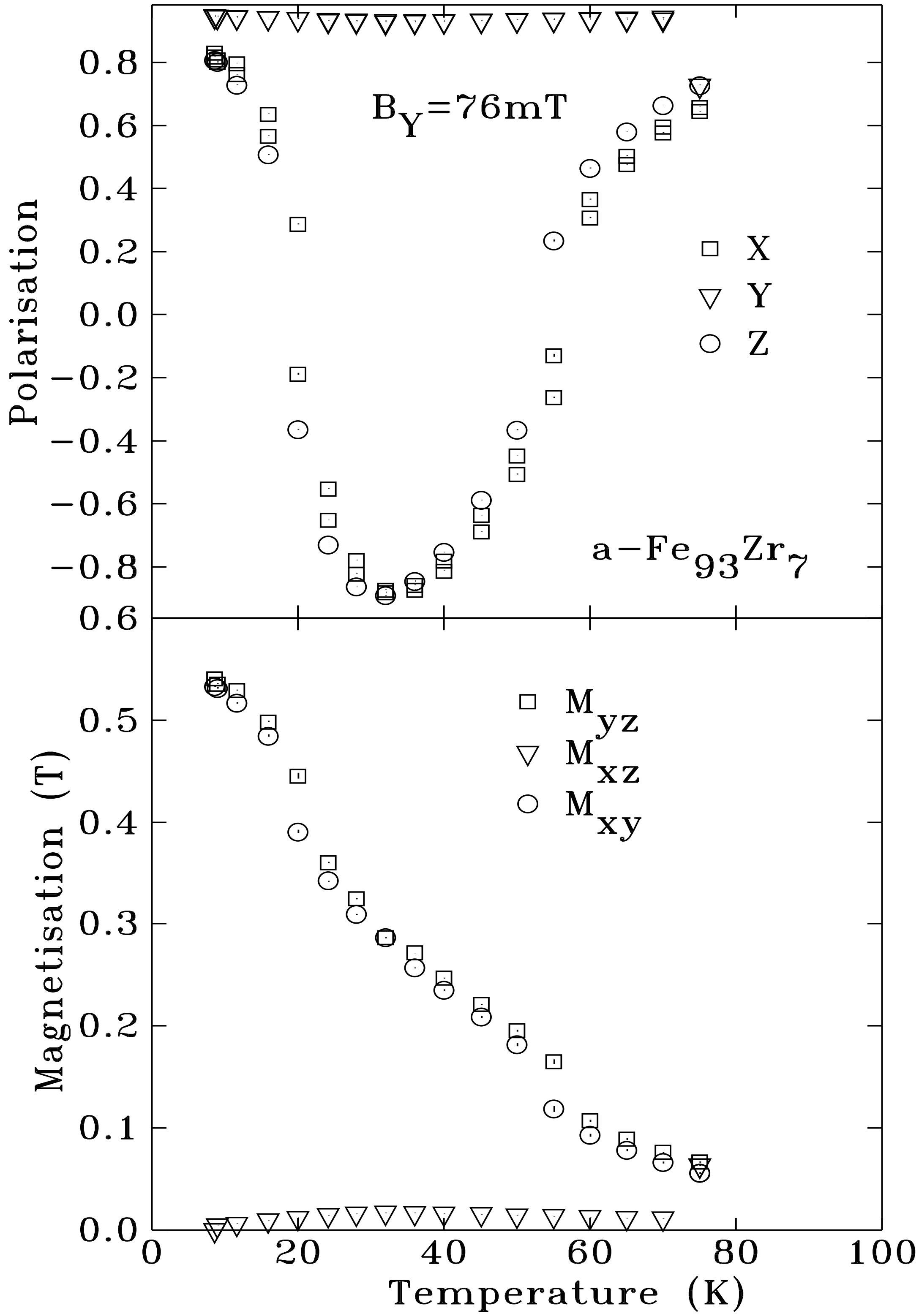

Figure 6: Ryan, Tun and Cadogan, ICM-97