

## ON THE EFFECTS OF MAGNETIC BONDING IN RARE EARTH TRANSITION METAL INTERMETALLICS

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## ABSTRACT

Neutron diffraction experiments on rare-earth transition metal magnetic alloys  $\text{Er}_2\text{Fe}_{14}\text{B}$  and  $\text{Er}_2\text{Fe}_{17}$  have been carried out at temperatures above and below the ordering temperature ( $T_c$ ). An anomalously large magnetic moment is observed at the crystallographic  $j_2$  site in  $\text{Er}_2\text{Fe}_{14}\text{B}$  which is the intersection point of the major ligand lines in the crystal structure. The interatomic Fe-Fe distances are in the range of strong ferromagnetic bonds ( $\geq 2.66 \text{ \AA}$ ). The analogous  $f$  site in  $\text{Er}_2\text{Fe}_{17}$  does not develop as large a magnetic moment. In addition, the same sites show strong preference for Fe atoms in the respective substituted compounds. Due to poor phase stability of  $\text{Er}_2(\text{Co}_x\text{Fe}_{1-x})_{14}\text{B}$  compounds, iron substitution has been studied in detail in  $\text{Er}_2(\text{Co}_x\text{Fe}_{1-x})_{17}$  alloys for site specific order and lattice distortion effects. However, a nonlinear change in the  $c$  lattice parameter observed in the neutron diffraction results cannot be explained on the basis of site preference alone.

The neutron refinement results indicate iron rich compositions in  $\text{Er}_2(\text{Co}_x\text{Fe}_{1-x})_{17}$  materials, which is related to random substitution of Fe dumbbell pairs on the rare earth sites in the lattice. However, extensive electron microscopy (selected area electron diffraction and high resolution imaging) of  $\text{Er}_2\text{Fe}_{17}$  and  $\text{Er}_2(\text{Co}_{.40}\text{Fe}_{.60})_{17}$  failed to reveal any microscopic inhomogeneity. At this stage, the concept of direct negative exchange interaction between dumbbell Fe atom pairs at very short distances of  $-2.38 \text{ \AA}$  is invoked and its effect is correlated with phase stability. We further suggest that the addition of boron in  $\text{Er}_2\text{Fe}_{14}\text{B}$  suppresses the substitution of dumbbell Fe pairs as a result of which  $T_c$  is raised significantly.

## I. INTRODUCTION

Rare earths are prolific compound formers with most of the elements in the periodic table. Interest in rare earth-transition metal intermetallics stems from the fact that they constitute excellent starting materials for permanent magnetics technology. Outstanding magnetic properties result from the combined magnetic features of the rare earth (4f magnetism) and the transition metal (3d magnetism). Simultaneously, one can benefit from the intrinsic properties of both partners, rare earths providing a very high saturation moment per atom and strong single-ion magnetic crystalline anisotropy, and a high  $T_c$  arising from the high magnetic coupling strengths of the moments of the 3d transition metals. This unique combination is exploited in fabricating high coercivity, high  $T_c$  materials for high temperature applications. Samarium-cobalt was invariably the material of choice until a new hard magnetic phase in the Neodymium-Iron-Boron system was discovered.<sup>1,2</sup> The structure consists of layers of puckered Sigma-phase type nets, formed solely by Fe atoms and sandwiched between triangular nets in the mirror planes formed by Nd, Fe, and B atoms.<sup>3</sup> Thus the environment and the sequences of the layers of Fe atoms are not significantly different from that of cobalt atoms in  $\text{SmCo}_5$  and  $\text{Sm}_2\text{Co}_{17}$  - the prototypes. Presumably, details of the atomic environment and Fe-Fe distances have a profound influence on exchange interactions in iron rich rare-earth phases; this is the subject of the present investigation.

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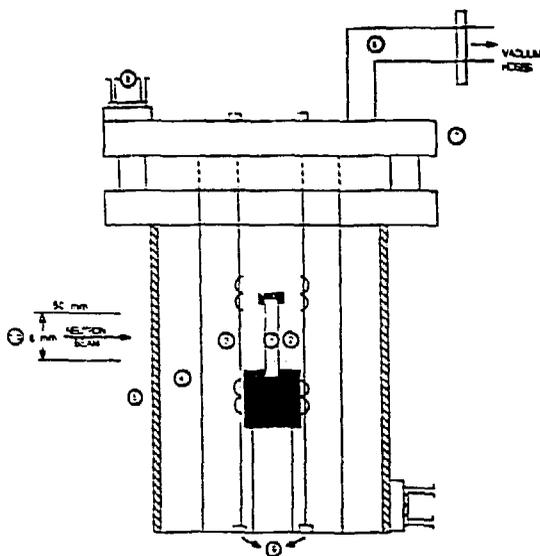
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One of the less favorable properties of the new rare-earth-iron phases is their relatively low curie temperature and a goal of the material scientists has been to modify these materials by site substitution and thereby study the effects on  $T_c$ . There is general agreement on the result that the curie temperature increases considerably for substitution of iron by cobalt at low concentrations, whereas the effect on  $T_c$  tends to saturate at high Co concentrations. This behavior is partly due to a preferential site ordering of the Fe/Co atoms. In principle, Mössbauer spectroscopy and NMR techniques<sup>4</sup> can be used to extract information about the preferential occupancy, but in the case of  $\text{Nd}_2\text{Fe}_{14}\text{B}$  with six inequivalent transition metal sites, the analysis is quite difficult and many different solutions provide equally good fits to the data. Neutron diffraction on the other hand, gives a unique solution and indicates that the Co atoms strongly avoid the  $j_2$  site, prefer the e site, and fill the remaining sites nearly at random with a slight preference for  $j_1$  below 50 at. % Co-substitution. On the other hand, the recent neutron scattering experiments<sup>5</sup> on silicon-substituted  $\text{RE}_2\text{Fe}_{14}\text{B}$  (RE = rare-earth) phases where an enhancement in  $T_c$  observed, find a low occupancy c site which is preferred by Si.

Thus the relationship between preferential site occupation and the increase of curie temperature remains far from being understood. The atomic distances spanned by various crystallographic sites, however provide a clue. The  $16k_2$  and  $8j_1$  sites are characterized by relatively small interatomic distances between the 3d atoms. Simple theory suggests that small interatomic distances favor antiferromagnetic correlations, and thus the presence of sites with short Fe-Fe bonds may account for the relatively low curie temperature in the  $\text{R}_2\text{Fe}_{14}\text{B}$  compounds compared to pure iron. The insertion of Co into the sites with short bonds (all but  $j_2$ ) may enhance the curie point both by the Co-Co exchange and by a redistribution in the number of short Fe-Fe bonds. However, the lack of pronounced preference of Co on the two sites with the shortest average bond lengths ( $j_1$  and  $k_2$ ) makes the interpretation less than unambiguous. The question of antiferromagnetic exchange may be more effectively examined by following the temperature dependence of the bond lengths: above  $T_c$  the bonding will reflect the electrostatic forces, below  $T_c$  this will be modified by exchange, and atoms in an antiferromagnetic environment will change differently in a ferromagnetically ordered material than those in a ferromagnetic environment. A related consequence of the quantum mechanical exchange forces is the observed spontaneous magneto-elastic deformation as a result of Co substitution in  $\text{Er}_2(\text{Co}_x\text{Fe}_{1-x})_{17}$  compounds, which will be dealt with in Sect. III. We have probed these effects by carrying out neutron diffraction at several temperatures above and below the ordering temperature (560 K). We believe a calculation of bond lengths and near-neighbor co-ordination from atomic ordering above  $T_c$  will help us distinguish between magneto-elastic strain and pure lattice strain in  $\text{Er}_2\text{Fe}_{14}\text{B}$ . Neutron diffraction is found to be an ideal probe for fixing the position of the boron atoms in a matrix of heavy ones e.g., erbium and iron.

## II. HIGH TEMPERATURE DIFFRACTION MEASUREMENTS

Neutron diffraction data on  $\text{Er}_2\text{Fe}_{14}\text{B}$  at 100°C (373 K), 150°C (423 K), 260°C (533 K), 310°C (583 K), and 360°C (633 K) were collected using a high-temperature furnace installed at the neutron powder diffractometer at the University of Missouri Research Reactor (see Fig. 1.). The samples were contained in vacuum tight vanadium cans heated from both top and bottom in an aluminum walled vacuum furnace. The diffractometer is equipped with an oscillating radial collimator which defines the scattering volume seen by the detector (a function of angle). The heat shields in the furnace are sufficiently far from the center that no Bragg peaks are transmitted through this collimator and vanadium, with near negligible scattering length, also contributes nothing to the diagram. The temperature stability was of the order of  $\pm 5^\circ\text{C}$ . Electron diffraction on



- The description of the components are:
1. Sample in the vanadium can
  2. Boron nitride masks
  3. Resistive Ni wound coil
  4. Tantalum heat shield
  5. Furnace aluminum wall
  6. Vacuum connection hoses for diffusion pump
  7. Insulating flanges
  8. Cooling copper tubings
  9. Temperature controller leads
  10. Slits defining the incident beam

Fig. 1. Schematic of the high-temperature neutron furnace.

annealed  $\text{Er}_2\text{Fe}_{17}$  and  $\text{Er}_2(\text{Co}_{.40}\text{Fe}_{.60})_{17}$  is also in progress to characterize the emerging microstructure as a result of the evolution of the atomic defects at the dumbbell site (4f crystallographic site in the structure) which we believe to support strongly antiferromagnetic interactions.

### III. RESULTS AND DISCUSSION

Neutron diffraction intensity from powdered crystals of  $\text{Er}_2\text{Fe}_{14}\text{B}$  at  $360^\circ\text{C}$  ( $>T_c$ ) is shown as function of scattering angle  $2\theta$  in Fig. 2. Data was collected in approximately 12-16 hours for fixed neutron monitor counts using a four arm position sensitive detector installed at the powder diffractometer of the University of Missouri Research Reactor (MURR). A data set was taken in three  $25^\circ$  ( $2\theta$ ) angular spans of the detector position. Data from  $5^\circ$  to  $80^\circ$  was finally rebinned in steps of  $0.1^\circ$ , and the profile intensity so generated was analyzed by MURR-modified Rietveld structure fitting procedure. In this analysis, the detailed intensity profile in the entire powder diffraction peaks is fitted to a model structure. This is accomplished by adjusting the structural parameters in a standard least-squares procedure to minimize the residual between the theoretical and measured intensities. The objective is to make full use of the crystallographic information contained in the accurately measured, but overlapping powder peaks. Neutron refinement of the atom positions was carried out in the tetragonal structure belonging to space group  $P4_2/mnm$  comprising about 350 independent powder reflections in the data. Only one site is fixed by symmetry, the rest involve at least one refinable position. The final refinement included fourteen atomic coordinates, three Gaussian half-width parameters, one counter zero-point parameter, two unit cell parameters, one overall intensity factor, one preferred orientation, and one asymmetry parameter. In the final analysis, boron's isotropic temperature factor was varied along with the rest of the parameters. The final results were further adjusted by iterating the calculations with several fixed values of temperature factor to look for a minimum in the fit indices. There is a provision to refine independently Er/Fe isotropic temperature factors, but the results on atom position are found to be insensitive to this procedure. However, refinement on Boron's thermal factor showed significant changes in the peak height intensity distribution. The refined value of Boron's thermal factor increased to  $3.4 \text{ \AA}^2$  from its room temperature value of  $2.0 \text{ \AA}^2$ . This room temperature value was found to be higher than the refined values in light rare-earth 2-14-1 phases. This may be an important piece of information related to the increased stability of light rare-earth phases, and their crystallization from amorphous phases. The results of

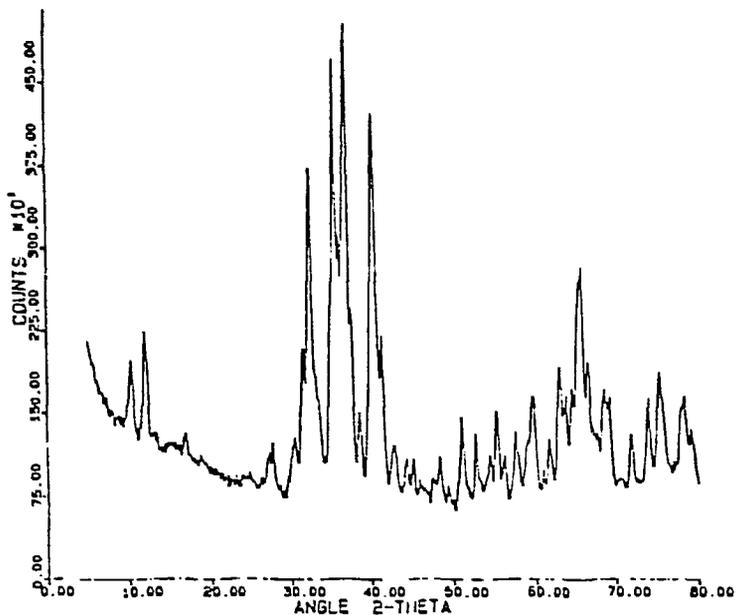


Fig. 2. Neutron diffraction spectrum at 633 K.

structure refinement are presented in Table 1, and the fitted diffraction diagram at 633 K is shown in Fig. 3, where the dots represent the observed intensity and the solid lines are the calculated intensity. Similar refinement on atom positions was carried out at temperatures of 583 K, 533 K, and 423 K. In all cases, the weighted profile R-factor remained within a factor of 2 of  $R_{exp}$ , the R-factor based on counting statistics. The positive deviations in the difference curve in Fig. 3 are mostly the contaminant scattering by unreacted elemental iron in the sample. Those regions of data could be excluded from the analysis, but do not affect the results other than the marginal improvement in the quality of fit indices. The refined atomic positions were finally introduced in a subsidiary crystallographic subroutine to calculate the interatomic distances. The distances then reflect the relative changes in the a and c lattice parameter as well as the coordination changes. These distances, were averaged over the symmetry related equivalent positions and the average distances are plotted in Fig. 4. The e and  $j_1$  sites show a strong negative magnetostriction, the bond lengths expanding as the temperature decreases. The remaining sites show only small changes in bond lengths over the same temperature range. This suggests that the e and  $j_1$  sites support the most important antiferromagnetic interactions and that the bond lengths readjust to weaken those effects as the crystal becomes more ferromagnetic. Thus the antiferromagnetic interactions are not merely dependent on average bond length, but must depend on the details of site environment (symmetry, co-ordinations, etc.). Interestingly, the Co does show a distinct preference for the e site on substitution as well as a slight preference for the  $j_1$  site at low Co concentration. A contradictory picture is given, however, by the increase in  $T_c$  on Si substitution, as this atom strongly prefers the c site.

To determine the magnetic order on the transition metal atom sites, neutron diffraction data of  $Er_2Fe_{14}B$  and  $Er_2Fe_{17}$  for the full four arm position of the detector ( $5^\circ$ - $105^\circ$ ) were taken at 373 K and 250 K, respectively. Data above room temperature (373 K) were collected with a furnace and low temperature data (250 K) were collected using a closed cycle refrigerator in which temperature can be continuously controlled between 293 K and 8 K. Neutron diffraction instruments at MURR optimize the data quality at low  $Q = (\frac{4\pi}{\lambda} \sin\theta)$  for the simultaneous analysis on the magnetic and nuclear scattering to yield site specific magnetic moments and their orientation in a ferromagnetically ordered uniaxial system. The theoretical calculation of the magnetic scattering requires the  $Er^{3+}$  and  $Fe^{2+}$  Hartree-Fock magnetic form factors as input in the Murr-modified

Table 1. Atomic positions obtained by structure analyzing the neutron data.

Crystall. Site	533 K (260°C)			633 K (360°C)		
	X	Y	Z	X	Y	Z
Fe 16k <sub>1</sub>	0.2241(7)	0.5671(7)	0.8720(5)	0.2239	0.5677	0.8702(5)
Fe 16k <sub>2</sub>	0.0364(7)	0.3588(7)	0.1731(5)	0.0386(7)	0.3576(7)	0.1724(5)
Fe 8j <sub>1</sub>	0.0999(7)	0.0999(7)	0.2036(5)	0.0987(7)	0.0987(7)	0.2025(5)
Fe 8j <sub>2</sub>	0.6794(6)	0.6794(6)	0.7570(7)	0.6791(6)	0.6791(6)	0.7570(7)
Fe 4e	0.0	0.0	0.3842(10)	0.0	0.0	0.3848(11)
Fe 4c	0.0	0.5	0.0	0.0	0.5	0.0
B 4g	0.3724	-0.3724	0.0	0.3745	-0.3745	0.0
R <sub>w</sub> = 6.05	R <sub>exp</sub> = 4.77	χ <sup>2</sup> <sub>av</sub> = 5.20		R <sub>w</sub> = 6.29	R <sub>exp</sub> = 5.50	χ <sup>2</sup> <sub>av</sub> = 4.8
a = b = 8.7077(15), c = 11.9549(23), V <sub>cell</sub> = 906.48(42) @ 533 K						
a = b = 8.7178(16), c = 11.9758(28), V <sub>cell</sub> = 910.17(49) @ 633 K						

	583 K (310°C)			423 K (150°C)		
	X	Y	Z	X	Y	Z
Fe 16K <sub>1</sub>	0.2249(7)	0.5667(7)	0.8705(5)	0.2228(8)	0.5663(7)	0.8718(5)
Fe 16k <sub>2</sub>	0.0385(7)	0.3572(7)	0.1724(5)	0.0373(8)	0.3590(7)	0.1723(5)
Fe 8j <sub>1</sub>	0.0991(7)	0.0991(7)	0.2034(6)	0.0999(7)	0.0999(7)	0.2021(6)
Fe 8j <sub>2</sub>	0.6790(6)	0.6790(6)	0.7571(8)	0.6794(6)	0.6794(6)	0.7581(8)
Fe 4e	0.0	0.0	0.3849(10)	0.0	0.0	0.3833(11)
Fe 4c	0.0	0.5	0.0	0.0	0.5	0.0
B 4g	0.3727(24)	-0.3727(24)	0.0	0.3756(26)	-0.3756(26)	0.0
R <sub>w</sub> = 6.23	R <sub>exp</sub> = 5.52	χ <sup>2</sup> <sub>av</sub> = 4.58		R <sub>w</sub> = 5.60	R <sub>exp</sub> = 7.36	χ <sup>2</sup> <sub>av</sub> = 2.05
a = b = 8.7192, c = 11.9787(27), V <sub>cell</sub> = 910.57(47) @ 583 K						
a = b = 8.7178(16), c = 11.9459(16), V <sub>cell</sub> = 907.90(46) @ 423 K						

The numbers in ( ) are the estimated standard deviation on the last digits of the refined parameters.

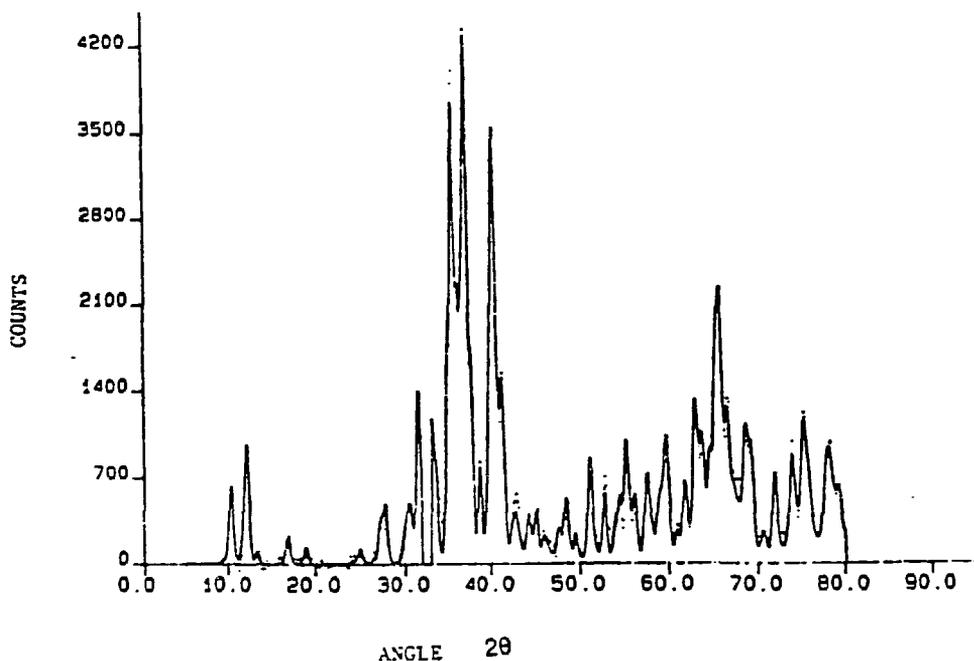


Fig. 3. Comparison of the observed and calculated diffraction intensities.

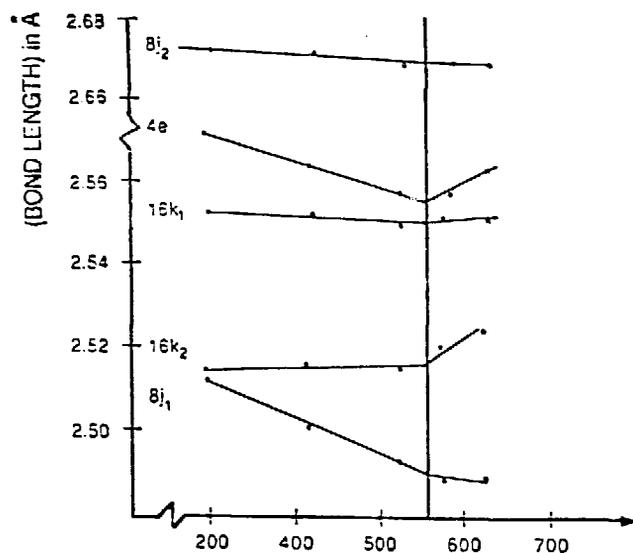


Fig. 4. Average bond lengths as function of temperature.

Rietveld code to fit the measured magnetic contribution in the powder pattern. Approximately 41 least-squares fitting parameters for the  $\text{Er}_2\text{Fe}_{14}\text{B}$  data and 31 for the  $\text{Er}_2\text{Fe}_{17}$  data have been varied simultaneously to minimize the disagreement factors in the refinement of the structure. The refined magnetic moments in Table 2 are dominated by the iron sublattice alone, because the rare earth sublattice is disordered at these temperatures in both systems. This helps to disentangle the present analysis on transition metal atoms from the rare earth atoms which fully order only at low temperatures. A detailed mechanism of the magnetic order in the two sublattices and its effect on the concomitant spin reorientation phase transitions will be published elsewhere.<sup>7</sup>

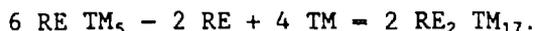
Table 2. Transition metal site magnetic moments in Bohr magneton ( $\mu_B$ ).

$\text{Er}_2\text{Fe}_{14}\text{B}$ (373 K)		$\text{Er}_2\text{Fe}_{17}$ (250 K)	
Er 4f	-1.84 (16)	Er 2b	-2.56 (20)
Er 4g	-2.00 (15)	Er 2d	-3.29 (19)
Fe 16k <sub>1</sub>	2.83 (18)	Fe 12k	1.95 (12)
Fe 16k <sub>2</sub>	2.38 (19)	Fe 12j	2.06 (14)
Fe 8j <sub>1</sub>	2.98 (18)	Fe 6g	1.35 (15)
Fe 8j <sub>2</sub>	3.70 (18)	Fe 4f	1.91 (14)
Fe 4e	2.18 (25)		
Fe 4c	2.01 (22)		

As expected, the rare earth couples antiparallel with the transition metal moments, and ordered transition metal moments show significant variation among crystallographic sites consistent with the site moments in isostructural compounds. The refined magnetic moments in  $\text{Er}_2\text{Fe}_{14}\text{B}$  are slightly large, perhaps due to systematic errors in the data analysis, but follow the same pattern as has been observed in this series when the magnetic moments have an axial configuration. Whereas the  $j_2$  site in  $\text{Er}_2\text{Fe}_{14}\text{B}$  and the f site in  $\text{Er}_2\text{Fe}_{17}$  are quite similar crystallographically, their refined magnetic moments differ by nearly a factor of 2. The  $j_2$  site has 12 Fe near neighbors in two hexagons (one above, one below), while the f site has 13 neighbors, the 12 of the  $j_2$  site plus 1 short (dumbbell) neighbor. It appears that this difference is critical and that further investigation of the site substitution in these compounds could provide a means of further diluting the prominent anti-ferromagnetic bonds. In one such recent study<sup>8</sup> at MURR, Mn substitution on transition metal sites has been extensively characterized by neutron diffraction, and the results on microscopic magnetic coupling at various crystallographic sites are as follows: Mn atoms prefer the  $j_2$  site and the Fe  $j_2$  magnetic moment remains highest at all Mn concentrations. The other sites,  $k_1$ ,  $k_2$ ,  $j_1$ , e, and c, show varying degrees of antiferromagnetic coupling between the near neighbor Mn atoms. This may well be related to anomalous expansion in a lattice parameter and the resulting net volume magnetostriction observed in the atomic structure analysis. It is

abundantly clear by now that occurrence of exotic magnetism at transition metal sites in rare earth transition metal hard magnetic alloys revealed by neutron diffraction experiments, is critically short of a unifying first principles mechanism.

In comparison to the  $RE_2TM_{17}$  compounds, the substitutions in the  $RE_2TM_{14}B$  phase is a simple problem (except with heavy rare earths). The latter phase is stoichiometric with all sites fully occupied; the former compounds support a variety of additional complications. If one looks at the progenitor phase  $CaCu_5$ , then one finds that the  $RE_2TM_{17}$  phase is formed by substitution of 1/3 of the RE atoms by pairs of TM atoms in a dumbbell arrangement along the c-axis according to the solid state reaction:



The substitution of dumbbells of the smaller TM atoms leads to an increase in the c-axis and a decrease in the a-axis. However, the resulting structure is highly defective and substitution of additional Fe atoms on some of the remaining RE sites is favored, leading to composition shifts towards the Fe rich side as we and others<sup>9,10</sup> have previously found. The effect of Fe/Co substitution demonstrates further the complexity of the problem. The room temperature lattice parameters of  $Er_2(Co_xFe_{1-x})_{17}$  obtained from the Rietveld analysis are plotted in

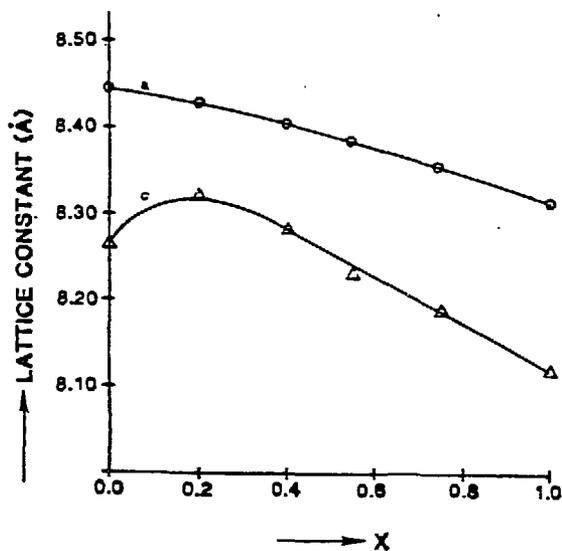


Fig. 5. Hexagonal Lattice constants of Fe-substituted 2-17 compounds.

Fig. 5. There are several competing effects here; the Fe atoms are larger than the Co atoms and yet for Fe rich compositions (small x) both a and c counteract. This may be the consequences of a negative magnetostriction which would affect the Co rich side (large x) more strongly than the Fe rich side, due to the high  $T_c$  in the Co rich alloys. The peak in the c parameter for 80 at. % Fe is almost certainly a consequence of the strong Fe preference for the dumbbell site (f) observed in the neutron refinements, which leads to the effects discussed above. As previously noted, this site has 12 near neighbors at long distances (~2.66 Å) plus a very short Fe-Fe bond (2.38 Å). This short bond may result in a large mechanical strain and low temperature phase

instability. Annealing of 2-17 materials with B present gives a strong tendency to transform to the  $Re_2Fe_{14}B$  phase<sup>11</sup> which maintains the same atomic environments minus this short dumbbell bond. The addition of B presumably restricts the Fe substitution in pairs and promotes a different rearrangement of the sigma phase type nets. The addition of Zr in a commercial grade composition of  $Sm(Co, Fe, Cu, Zr)_2$  system is also helpful in preparing high coercivity, high  $T_c$  magnets. It has been suggested<sup>12</sup> that the Zr atoms preferentially occupy one of the two dumbbell sites, relieve the strain, and reduce the antiferromagnetic bonding, but the conclusive evidence of the Zr location is lacking. Boron segregation to grain boundary phases in 2-14-1 and the preferential diffusion of the transition metal additions at the defected dumbbell site may be controlling the kinetics of the evolution of the specialized microstructure in the respective materials. However, our initial TEM micrographs of the high temperature (1200°C) annealed hexagonal phases did not show any evidence of rhombohedral twinning due

to layer faulting in  $\text{Er}_2\text{Fe}_{17}$ . Samples of  $\text{Er}_2\text{Fe}_{17}$  and  $\text{Er}_2(\text{Co}_{.40}\text{Fe}_{.60})_{17}$  annealed at  $650^\circ\text{C}$  will be examined by high resolution TEM and selected area diffraction to study the phase decomposition reaction. Further neutron diffraction is planned on the rare earth substituted  $(\text{Er}_x\text{Pr}_{1-x})_2(\text{Co}_x\text{Fe}_{1-x})_{17}$  and  $\text{Er}_2(\text{Co}_x\text{Fe}_{1-x})_{14}\text{B}$  compounds for crystal and magnetic structure studies on the high resolution powder machine being installed at the University of Missouri Research Reactor.

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