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ELECTRONIC STRUCTURE OF TERNARY ALLOYS

FINAL TECHNICAL REPORT

Leonid V. Azároff

1 June, 1978

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The agreement between the two kinds of x-ray measurements and the band calculations that the local densities of 3d holes do not change either at Fe or at Ni sites requires a new model to explain the observed magnetic saturation effects in these alloys. It is proposed that the density of d holes stays constant but their distribution among spin-up and spin-down subbands changes as a function of Al concentration. Evidence already in hand indicates that a similar model fits the x-ray and magnetic saturation data in the binary Ni-Al system also.

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I. INTRODUCTION

The present investigation of Fe-Ni-Al ternary alloys was motivated, in part, by previous magnetic moment determinations of a series of fcc and bcc random solid-solution alloys in this system. Thus Bardos, Aldred, and Beck¹ found, in the fcc alloy series with constant Ni:Fe ratios of 4:1, 3:2, and 2:3, that the decrease in saturation moment of the alloys as the Al content increased to 10 at.% could be accounted for by assuming that the Ni spin moment declined just as it does in binary Ni-Al alloys whereas the Fe moment is taken to be the same as in binary Ni-Fe alloys at comparable Ni:Fe ratios. For iron-rich bcc ternary alloys, Bardos, Beeby, and Aldred² found that this simple model would not work because of an apparent interdependence of the Fe and Ni moments. To explain the experimentally measured alloy moments, they had to devise an eight-parameter phenomenological model in which the transition-metal moments were determined by analogy to Fe-Ni binary solid solutions and the aluminum concentration had a simple dilution effect as in Fe-Al solid solutions. Such a model, like two theoretical models,^{3,4} assume a homogeneous *d* band formed by both transition-metal atom types and depends on the difference in the total number of conduction electrons to explain the magnetic moment changes in the ternary alloys.

By comparison, Vincze⁵ used Mössbauer effect measurements to reveal substantially different behaviors for the Fe and Ni atoms in the presence of aluminum and suggested that a collective *d* band is not formed by the transition-metal atoms. His isomer-shift measurements also did not indicate the required rapid decrease of conduction electron density.

He concluded, finally, that a model proposed by Marshall and cited by Mott,³ which assumes an occupation of the lower-lying 3d holes in nickel atoms by aluminum conduction electrons, satisfactorily accounted for the experimental data of Bardos *et al.*²

Because x-ray spectroscopy can observe independently the electronic structures of each constituent in an alloy, the present study was undertaken to elucidate what changes actually take place at the transition metal sites of ternary Fe-Ni-Al solid solutions. Alloys having the same compositions in the fcc and bcc ranges of the diagram as those used by the above investigators were employed, including some of the very same samples kindly supplied by Drs. Bardos, Aldred, and Vincze. By combining *K* and *L* absorption spectra measurements of both transition metals in each alloy sample, independent determinations of the effects produced by alloy composition were possible. In addition, direct band-model calculations were carried out for some of the selfsame alloys to determine theoretical spectral curves for comparison with theoretical ones.

The measured x-ray spectra clearly show that the electronic structures of the nickel and iron do not change significantly upon alloying in, both, the fcc and bcc solid solutions. This is also confirmed by the theoretical band models calculated for the fcc ternary solid solutions. (This is the first reported band calculation of ternary solid-solution alloys.) A reexamination of previously measured spectra for binary alloys of Fe-Al, Ni-Al, and Fe-Ni confirm that electron transfer from aluminum to the transition metals does not take place to any significant extent. Like the density-of-states curves calculated for the fcc ternary alloys, the x-ray results do not show any evidence for the formation of a collective *d* band by the transition metals. The actual results obtained and a discussion of their implications on the magnetic properties of the two ternary systems studied are presented below.

EXPERIMENTAL RESULTS

The experimental details of sample preparation, characterization, and x-ray spectroscopic measurements have been presented in previous progress reports and are therefore not repeated here. Suffice it to note here that absorption specimens were prepared by flash evaporation from alloys carefully characterized by x-ray diffraction and microprobe analysis to resemble those used by other investigators. A procedure developed in our laboratory for correcting for the effect that compositional variations have on the measured x-ray absorption coefficient (called the *thickness effect*) was systematically applied to all experimental data obtained. Moreover, the fully-automatic two-crystal spectrometer available in our laboratory,⁷ is capable of measuring the x-ray beams transmitted through the absorbing foils with an intensity (counting statistics) and energy resolution (angular precision) representative of the latest state of the art.

To assist in the Discussion of Results presented later, the x-ray measurements are summarized below for the two alloy systems examined.

Face-centered cubic alloys. Ternary alloy samples containing Ni:Fe ratios of 4:1, 3:2, and 2:3 with 0, 2, 5, and 10 at.% aluminum were examined by setting the spectrometer to record separately, the *K* and *L* absorption edges of Fe and Ni. Because of the relatively small concentration of iron in some of these alloys, accurate absorption edges (particularly the L_{III} spectra) could not be obtained for them.

As compared to the Ni *K* absorption spectrum of pure nickel, alloy spectra show no observable change in the initial rise due to $1s \rightarrow 4p$ transitions, and an apparent enhancement of the low-energy side of this peak as a result of alloying. This is consistent with earlier x-ray studies of binary fcc alloys in the Fe-Ni⁸ and Ni-Al⁹ systems when the latter are properly corrected for thickness effects.¹⁰

The Ni L_{III} spectra, due to $2p \rightarrow 3d$ transitions, similarly show variations in relative peak intensity that can be accounted for completely by the atomic concentrations present. Because a very pronounced peak occurs near the Fermi-edge cutoff in the Ni L spectrum, due to transitions to unoccupied $3d$ states at nickel atom sites, even a small filling of such vacant states would be clearly manifest. The absence of a systematic decline with increasing aluminum concentration, therefore, clearly demonstrates that causes other than $3d$ hole filling are responsible for the disappearing moments of nickel atoms.

Quite similarly, the iron edges do not appear to change significantly with alloy composition from their appearance in pure iron. Probably the most significant changes observed are their resemblance to other fcc iron spectra (previously reported¹¹) and a diminution of the $4p$ absorption peak that is considerably more pronounced than the similar decline in the nickel spectrum. The decline in this absorption maximum, moreover, increases with increasing aluminum concentration in a systematic way.

Body-centered cubic alloys. Iron-rich solid solutions containing Ni:Fe ratios of 1:48, 2:23, 4:21, and 1:3 were prepared with, respectively, 0, 2, 5, 10, and 15 wt.% aluminum. Conversely to fcc alloys, best experimental results for all bcc alloy samples were obtained for iron absorption edges. In fact, the L edges of nickel could not be obtained for most samples without using foils thicker than those readily attainable by flash co-evaporation.

As compared to pure bcc iron, the Fe K edges show no changes in the initial rise due to $1s \rightarrow 3d(sp)$ transitions. The first main absorption peak due to $1s \rightarrow 4p$ transitions is notably decreased in the binary alloys (0 at.% Al), in agreement with previous measurements,⁸ but very nearly resembles that of pure nickel in aluminum-containing ternary alloys. An

examination of previously studied Fe-Al alloys having comparable Al concentrations¹² shows that this is an effect due to aluminum. All in all, no significant changes interpretable in terms of changing electronic densities at Fe sites manifest themselves in their *K* spectra.

Quite similarly, the L_{III} spectra are little affected by alloying. It should be reported here that a slight *increase* in the first peak near the main edge appeared to take place with increasing aluminum concentration. Without a corresponding change in the Ni spectra, however, it is not clear what causes the observed changes in Fe.

Like in fcc alloys, the nickel absorption edges do not appear to change in the bcc ternary alloys either with iron concentration or as a function of aluminum concentration. This is consistent with measurements of binary Fe-Ni alloys⁸ in which no changes in the Fe edges were observed and the Ni edges remained unchanged up to 22 at.% nickel, showing a decline above ~~that~~ composition, however.

THEORETICAL CALCULATIONS

The theoretical treatment of the electronic structure of Ni-Fe-Al ternary alloys was carried out within the framework of the average t-matrix approximation (ATA). This approach to the problem of random substitutional alloys was chosen for its relative ease of application as compared to the much more lengthy computational effort required when the coherent potential approximation (CPA) is employed.

In the ATA, the electronic spectrum of an alloy is calculated by assuming that the effect of disorder is to replace the alloy by an averaged lattice of effective scatterers. The t-matrix associated with scattering at each lattice site then is given by a compositionally weighted sum of alloy component t-matrices. In obtaining the average lattice, the configurational averaging used to arrive at the average t-matrix for each

lattice site restores the translational periodicity. This allows one to retain, both, the wave vector as a good quantum number and the concept of the Brillouin zone. The price paid for retaining these familiar concepts, however, is that the scattering phase shifts become complex, and the secular matrix of the regular KKR band theory becomes non-Hermitian, resulting in complex eigensolutions for the electronic energy spectrum. The imaginary part of the energy eigenvalue is the measure of lifetime of the electron in its eigenstate.

Detailed calculations were carried out for Ni-Fe-Al alloys in the nickel-rich region with Ni Fe ratios of 4:1 and 3:2 and with aluminum concentrations of 0, 2 and 5%. The only experimental inputs into the calculations were the appropriate lattice constants and the crystal structure (fcc). The alloy crystal potentials for each alloy component were constructed via the "superposed atom" method from Hartree-Fock-Slater atomic charge densities. From these scattering phase shifts were obtained. The only other inputs necessary for the calculation were the KKR structure constants for the fcc lattice which were computed for 131 $\vec{k}$ -points lying along 13 lines. These 13 lines are the Bansil's "special directions" which allow one to perform Brillouin zone integrals of functions in $\vec{k}$ -space which have the symmetry of the cubic lattice.

Using the ATA formalism of Bansil and Schwartz, spectral densities (i.e. densities of states at a $\vec{k}$ -point) were calculated at the 131 point Bansil mesh. Unlike the case for ordered structures where the spectral densities are Dirac's delta functions, for alloys the spectral densities are broadened by the disorder. The total density of states is the Brillouin zone sum of the spectral densities. In spite of recent advances in integrating these steep functions it should be noted that present-day techniques still produce considerable noise in the final result.

One of our original goals in this calculation was to calculate the x-ray absorption and emission spectra for each alloy component. Since such x-ray processes are local in nature, this required projecting out the appropriate component densities of states. Unfortunately, in the ATA formalism of Bansil and Schwartz,¹³ the component densities of states can and do become negative. This very unphysical feature of the formalism forced us to abandon our original aim of calculating the alloys' x-ray excitation spectra. Instead, we concentrated on the total densities of states where the negative densities-of-states problem is found to occur infrequently and is surmountable.

With the spectral densities calculated and stored on tape, the final Brillouin zone integration for the total densities of states were performed. The line integrals along Bansil's directions were done by first interpolating the spectral densities to a denser mesh and then making a polynomial fit to the data. Finally, analytic forms for the integrals were provided which led to the desired densities of states. This calculation is, to our knowledge, the first muffin-tin ATA application to ternary alloys.

Although the negative component densities in the ATA prevented us from carrying out the desired x-ray spectral analysis, there is a wealth of information that can be obtained from the computed total densities of states as a function of varying concentrations. We find that the Ni-Fe-Al density-of-states spectra are principally characterized by two types of behavior. First, there are two strong *d*-state resonances, one due to Ni and the other to Fe, with large densities of states. The Ni *d* resonance occurs at energies at and below the Fermi energy, E_F , since most Ni *d* states are occupied (pure Ni has ~ 0.6 *d* holes). The Fe *d* resonance lies above E_F reflecting the fact that pure Fe has about 2.2 *d* holes per atom. The position of the *d* resonances remains the same for all the alloys. In

this spectral range, for the concentrations studied, there is no change on alloying except for broadening upon addition of Al. Otherwise, the density of states faithfully mirrors the strength and structure of that for Ni which here is the majority component of the alloy.

DISCUSSION OF RESULTS

Combined x-ray experiment and band theory. Because of the negative component densities of states resulting from the ATA formalism, it proved impossible to calculate the absorption spectra of the component atoms in the ternary alloys studied. Although CPA has been used successfully for binary alloy calculations, the much more lengthy calculational efforts required by ternary alloys decided us not to pursue this approach. The remaining time limitations similarly dictated against carrying out full calculations for bcc ternary alloys. Instead, the fcc band calculations were completed for the alloys noted in the previous section and it is of interest to compare them here to the experimental x-ray results.

Clearly, the agreement between the calculated density-of-states curves and the corresponding features of the K and L absorption curves is remarkable. Theoretical curves show separable d -state resonances attributable to Ni and Fe, respectively, that reflect the relative concentrations of the two transition metals present but not an overlap indicative of a collective band formation. The x-ray absorption edges, similarly, portray no changes attributable to local variations in their electronic structures with alloying. Both, the features of the calculated density-of-states curves and the corresponding x-ray fine structures show a certain broadening with increasing Al concentration. In this connection it should be noted that the decline in the $4p$ absorption peaks reported above can be readily explained by a broadening effect which is further consistent with the slight energy shifts observed in adjacent peaks.

An interesting "side effect" of this investigation is the confirmation of a long believed but not hitherto verified assumption regarding the K absorption edges of transition metals of the first long period. Starting with the pioneering working of Beeman over thirty years ago, it has been assumed that the initial rise in the K edge is due to transitions to unfilled $3d$ states which satisfy dipole selection rules by having admixed $4sp$ symmetry. By comparison, the corresponding rise in absorption in the L_{III} spectrum does not require admixture since dipole selection rules are satisfied by transitions to states having d -type symmetry directly. (The correspondingly higher transition probabilities are the reason, of course, why a very distinct peak appears in the L spectra.) Any changes in the unoccupied density of $3d$ states caused by alloying, therefore, should be manifest in both spectra, if the above postulates are correct. The self-consistency of both kinds of spectra for each measured alloy in the present study clearly supports the above.

Implications on magnetic properties. The absence of any evidence that the local $3d$ densities of states at Fe and Ni atom sites change with alloy composition strongly argues that the measured magnetic moment changes must be explained in different terms. Present band calculations similarly fail to show changes suggestive of any "filling" of empty $3d$ states at either Fe or Ni in these alloys. By comparison, magnetic saturation measurements only measure an "average" moment per atom and cannot, of themselves, discriminate the moments at different atomic sites. In fact, in dealing with ternary alloys, one is forced to make inferential deductions from binary alloy measurements.

Magnetic measurements of Fe-Al solid solutions show that the saturation value decreases linearly with the addition of Al in a way

that corresponds with simple dilution up to 18 at.% aluminum. For Ni-Al solid solutions up to 10 at.% Al, a much more rapid decline in saturation value sets in which has been explained variously by rigid-band-like models involving occupation of normally empty $3d$ states in Ni, shifts in the Fermi energy, or both. Because of the obvious relevance of the electronic structures of these alloys to the present study, their L x-ray spectra have been measured and no significant changes in the $3d$ state density were observed.* Thus it is necessary to postulate a model in which the net moments at nickel atom sites decline with decreasing Ni concentration without changing the local density of vacant d states.

A possible model that does not require such correlation with the density of empty states is one in which the distribution among spin-up and spin-down subbands changes with alloying rather than the number of empty states. Alternatively, an interaction between Al conduction electrons and transition-metal d electrons may cause a rearrangement (randomization) of their spin states (net atomic moments) that accounts for the declining saturation magnetization. This is not unlike a model proposed by Beeby¹⁴ in which the increasing number of conduction electrons introduced by Al additions causes a rearrangement of the d bands due to an increasing s - d admixture. Such a model adequately explains the reported magnetic measurements and is consistent with our observations of the K and L spectra of binary and ternary alloys of Fe, Ni, and Al. Others also are beginning to lean to such a model for explaining their observations like the electronic specific heat variations in similar alloys.**

*The x-ray study, combined with magnetic saturation measurements of identical alloys, is nearing completion and will be prepared for publication in mid-summer.

**We thank Professor P. A. Beck for his personal comments to this effect.

CONCLUSIONS

Several important results were obtained in the present study and several new lessons were learned. It was shown that ATA calculations can be applied successfully to ternary solid-solution alloys. It was learned that this approach prevents calculation of x-ray spectra, however, because they require component densities of states. Given the present state of the art in band calculations, a CPA approach can be used but at a prohibitive cost in computer time. Fortunately this is not the case for binary alloys so that it is recommended that further studies concentrate on such systems.

It also has been demonstrated that a combination of two parallel but fully independent measurements of the "same" effect gives greater confidence in the results adduced to each. Thus, corresponding measurements of K and L absorption edges on a series of identical alloys led to an unambiguous conclusion that the density of $3d$ holes at Fe and Ni atoms did not change (except as noted) with alloying and did not correlate with Al concentration whereas their respective spin moments did. Although it is possible to concoct an elaborate explanation for the absence of a manifest x-ray spectroscopic effect, a far more plausible model is provided by an invariant local hole density with a changing proportion of unoccupied up and down spin states.

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APPENDICES

Participating Personnel

The following individuals have contributed to the work described in this report and received partial support from the grant during the reporting period 1 June 1975 through 31 May 1978:

Leonid V. Azároff, Professor of Physics and Director of IMS

Philip E. Best, Associate Professor of Physics

Douglas M. Pease, IMS Postdoctoral Fellow

C. C. Chu, Graduate Assistant

Bernard F. Cordtz, III, Graduate Assistant

Steven Kao, Graduate Assistant

C. K. Vaccaro, Graduate Assistant

Of the above, Mr. Chu completed his Ph.D. requirements in 1976, Mr. Kao earned his M.S. degree in May, 1977, while Dr. Pease has been appointed Assistant Professor in Physics in 1978.

Publication List

The list of publications presented below includes two papers describing work on nickel which had been initiated under a previous NSF grant but completed under the present one. The results of our work on the ternary system has only now been completed so that the last two titles listed represent manuscripts currently in preparation. It is expected that they will be submitted for publication this summer.

"Augmented-plane-wave calculation and measurements of K and L x-ray spectra for solid Ni," Phys. Rev., Frank Szmulowicz and Douglas M. Pease, 17B (1978) 17.

"Bremsstrahlung Isochromat from Nickel," C. C. Chu and P. E. Best, Submitted to Phys. Rev.

"X-Ray Spectroscopy of Metals and Alloys," L. V. Azároff, J. Metals 29
(1977) 38.

"Electronic Structures of Ternary Fe-Ni-Al Alloys," to be submitted this
summer.

"Electronic Structures and Magnetic Moments in Ni-Al Solid Solutions,"
to be submitted this summer.