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Synchrotron Radiation Studies of Local Structure and Bonding
in Transition Metal Aluminides and Rare Earth Transition Metal
Magnetic Nitrides

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I. Studies of Refractory Alloys

(a) Studies of bonding in NiAl, CoAl and FeAl by temperature dependent EXAFS

At the time of our previously submitted proposal, we had reported evidence that the temperature dependant EXAFS of CoAl showed a "stiffer" CoAl lattice, relative to either NiAl or FeAl. We also reported evidence from the literature (x-ray diffraction mean squared displacement (MSD)), and from our interactions with P.C. Clapp of the University of Connecticut, Metallurgy Department, that the Al-Al bond was "stiffer" than the Ni-Ni bond in NiAl, an effect - possibly associated with contacting large Al spheres; recent calculations by Freeman, et. al. also indicate that the Al-Al bond in NiAl is "stronger than the Ni-Ni bond". This finding was of significance in the light of our evidence (reported below) that in the ductilized microalloyed material one substitutes smaller Fe or Ga for the contacting Al spheres.

We have now obtained a much more detailed and rigorously founded picture of the bonding in NiAl, CoAl and FeAl since our last submission. We had become aware of recent neutron diffraction studies of single crystal NiAl which resulted in interatomic force constants for that material. We then embarked on a program in which those NiAl force constants could be used in a Born von Karmen model, and various quantities of interest, such as the EXAFS mean squared relative displacements (MSRD) and diffraction MSD values could be computed. The same computer program can be then used to generate MSD and MSRD values for comparison to experiment, assuming test values of force constants for CoAl or FeAl, for which no comparable neutron diffraction data exists. (To date, we do not believe anyone has produced a useful single crystal of CoAl, due to the brittleness of this material.) At the time of our previous proposal, we had submitted an article to Phys. Rev. B comparing the temperature dependent EXAFS of NiAl and CoAl alone; the referee suggested some extensions and we have now carried out a far more detailed analysis for all three alloys NiAl, CoAl and FeAl, which is being prepared for submission. Our new results are described below.

In Fig. 1 we display our experimental values of $\Delta \delta_{\text{MSRD}}^2$, the change in MSRD for the first three shells relative to a nickel absorber, as a function of temperature. The

$\Delta\epsilon_{\text{MSRD}}^2$ are plotted relative to the lowest temperature value as a reference. On the same plot is shown calculated values, obtained from a Born von Karmen calculation, using the neutron diffraction determined force constants of Mostoller, et. al., as input. Agreement is excellent, furnishing a valuable check on our experimental results. Fig. 2 shows a similar plot for FeAl, in which the experimental $\Delta\epsilon_{\text{MSRD}}^2$ of FeAl are compared to theoretical values obtained using NiAl force constants. (We use force constants out to third neighbors in our calculations.) From the good agreement between theory and experiment in Fig. 2, we deduce that the near neighbor force constants for FeAl (for which no neutron diffraction studies exist) are similar to those for NiAl. On the other hand, the experimental $\Delta\epsilon_{\text{MSRD}}^2$ for CoAl do not fit a curve based on the NiAl force constants. By trial and error, varying the nearest neighbor force constants, we find reasonable agreement with experimental values for CoAl by increasing the matrix diagonal, nearest neighbor force constant, by about 30% and decreasing the off diagonal force constant by 75% relative to the case of NiAl. The matrix diagonal near neighbor force constant is equivalent to a Hooke's law restoring force constant for a $\langle 100 \rangle$ direction force on a Ni atom if an Al atom is displaced in the $\langle 100 \rangle$ direction. This strain is closely related to the C_{44} elastic constant. Since covalent forces are generally associated with strong shear resistance, we deduce that there is something distinctly more "covalent" about the near neighbor forces in CoAl than in either NiAl or FeAl. We note that CoAl is distinctly more "brittle" than NiAl, and probably than FeAl. (The brittleness-ductility question for FeAl has been somewhat confused by the apparent importance of extrinsic factors such as water inclusions on the surface of this material. But for somewhat off-stoichiometric material, at least, very carefully prepared FeAl apparently has some ductility.)

We believe our results are an interesting challenge to electronic structure calculations. We have become aware of very recent calculations, by Davenport's group at BNL, of the electronic structure of NiAl, FeAl, and CoAl which, so far, do not show any striking evidence for why CoAl should be particularly brittle. Motivated by our EXAFS results, however, we embarked on an extensive study of existing elastic constant measurements, both published and in the form of internal reports, for these materials. The comparison is complicated somewhat by the fact that no single crystal coefficients are

available for CoAl, therefore no elastic moduli C_{11} , C_{12} or C_{44} have been measured for this material, and by the fact that the polycrystalline Young's and shear moduli can be obtained from C_{11} , C_{12} and C_{44} values only within upper and lower bounds determined by the Hashin-Shtrikman limits. To briefly sum up our findings analyzing the literature results, we find that the bulk moduli of all three alloys are quite similar. (For a cubic material, the bulk modulus does not depend on C_{44} .) However, the polycrystalline shear modulus, and by implication the value of C_{44} , of CoAl is distinctly larger than that of either NiAl or FeAl. On the other hand, as far as the cohesive energy is concerned, FeAl is "odd man out" having a much lower cohesive energy than either NiAl or CoAl. Thus, the existing elastic moduli data are consistent with our EXAFS results; however the EXAFS gives a much more detailed picture of the differences in bonding in terms of diagonal and off diagonal force constants between specific atomic pairs, than can be obtained from elastic constant measurements alone.

We next consider the relationship to the diffraction MSD values obtained for NiAl by Georgopoulos and Cohen and by Fox, and for CoAl by Fox. We use our Born von Karman program to compute Ni versus Al MSD values in NiAl. We find in agreement with Georgopoulos and Cohen and the simulations of Clapp, that the lighter Al atom has a distinctly smaller MSD value than the Ni atoms in NiAl. (This result would be non intuitive of one thinks of a light Al atom hooked by a spring to a heavy Ni atom in a two atom model.) The MSD results again furnish further evidence for the "contacting Al sphere" picture. Furthermore, when we use the adjusted force constants which increase the "covalent", diagonal near neighbor constant of CoAl relative to NiAl, thus giving agreement with our EXAFS MSD values for CoAl, we find the calculated average CoAl MSD values are about 20% smaller for room temperature than the NiAl MSD values. This result agrees with the diffraction results of fox on CoAl, again furnishing a check on our procedures.

Having now established the validity of comparing EXAFS MSD values with values computed from a force constant Born von Karman approach, we next re-approached the question of MSD values as seen relative to Ni or Al atoms in NiAl. As pointed out previously, there are considerable experimental problems in obtaining good temperature dependant Al K edge EXAFS. We have now calculated MSD values from the standpoint

of the Al atom, however. First, the Ni-Al MSRD near neighbor value is calculated to be the same as seen from the Ni or Al vantage point. This result has to be true for logical consistency and furnishes another check point on our procedures. We find however, that the 2nd neighbor Al-Al MSRD values are much less than the 2nd neighbor NiNi MSRD values, by a considerable amount. This result again illustrates the importance of stiff forces between Al spheres to the lattice dynamics of these systems.

Finally, although there exist extensive analysis of pure metallic systems in terms of EXAFS MSRD values and theory, and an analysis of the AgI system has also been carried out, we are unaware of our present approach being used previously, in which close neighbor force constants are chosen by trial and error in order to obtain fits to EXAFS MSRD values for an alloy, in a Born von Karman model. We believe that, in addition to our findings about refractory alloys, the procedures we are developing may lead to an advancement in the utilization of EXAFS to obtain local lattice dynamical information.

(b) Site selectivity studies in ductilized, β phase NiAl

We have been studying the alloys formed when Fe or Ga is microalloyed into an NiAl matrix. Dariola reported that for NiAl doped with 0.25 atomic percent Fe and for NiAl doped with 0.1 atomic percent Ga there was a significant ductility enhancement which disappeared at higher doping levels. At the time of our previous proposal, we reported preliminary studies indicating that for an alloy such as $\text{Ni}_{50}\text{Al}_{49}\text{Fe}_1$, the entire 1% of Fe atoms go exclusively to the "Al type sites" (transition metal (TM) nearest neighbors), rather than have a site selectivity in which the 1% excess TM atoms are randomly chosen between Ni and Fe. Since that time we have found out much more about this problem. We have made alloys that were Al rich, such as $\text{Ni}_{46}\text{Al}_{52}\text{Fe}_2$ and here find that the Fe atoms, in contrast to the TM rich case, go exclusively to TM type sites. We examined published temperature dependent magnetization measurements carried out on this system by Ikeda, and have reinterpreted his results based on our new findings. He reported a magnetic moment per iron atom of $24\mu_B$. We show that this value was based on an erroneous model of the site selectivity, resulting in a too low value of N, the concentration of Fe moments, appearing in his expression for the Curie constant. Re-analyzing his data, we now obtain a consistent moment value between magnetization curves (previous results did not show this

consistency) and the moment per iron atom is of order 7.3 Bohr magnetos. This is close to the moment of a central iron atom having the moment of iron in pure alpha iron, surrounded by eight TM atoms of moment 0.6 B (Ni in pure Ni), and seems much more reasonable than $24\mu_B$. This reanalysis strongly supports our site assignment.

We have also worked with Prof. Clyde Kimball of Northern Illinois University, who has carried out Mössbauer measurements on these specimens. He finds, in agreement with our results, that the iron in $\text{Ni}_{50}\text{Al}_{49}\text{Fe}_1$, while surrounded by TM near neighbors, is not present as ferromagnetic iron precipitates and has a shift consistent with nickel near neighbors. Finally, previous Kondo effect measurements on Fe doped NiAl, carried out by Ochai and Brittain, should be interpreted based on our finding of Fe site selectivity. A paper on site selectivity in Fe doped NiAl has been written up and is ready for submission.

Based on our findings to date, we speculate that the Fe dopants of Dariola may change near neighbor force constants and enhance ductility partly because of the relief of stiff Al-Al forces upon substitution of smaller Fe atoms for one of a pair of contacting Al spheres.

At the time of our previous proposal, we had reported that Ga, which is also a ductilizing impurity in small concentrations, appears to go to the Al site in TM rich Ga doped NiAl, just as Fe does in Fe doped NiAl. However, we have since amassed evidence that this story is more complicated. The previous Ga EXAFS was on as-cast materials; the EXAFS was rather consistent with an "Al type of site" selectivity for the Ga, but the fit to this model was not as good as one would hope. Furthermore, upon giving the samples a lengthy temperature anneal, we found the Ga EXAFS had completely changed in character. The EXAFS became fairly consistent with a theoretical simulation of pure gallium. Thus, our EXAFS studies are indicative of Ga precipitation in this material. We have had problems investigating this finding with x-ray emission electron microscopy (EDAX) so far, possibly because our grain boundary etchants may preferentially be attacking the Ga. We need to do more thorough studies, varying the annealing temperature, but now have a working hypothesis that the Ni-Al-Ga system may have some kind of miscibility gap (we can find no ternary phase diagram) despite the fact that NiAl and NiGa are isostructural. This behavior may have gone undetected by Dariola because he used very high annealing

temperatures (1300°C). Of course, a tendency for the ductilizing element to go out of solution, as we seem to be finding, would be a real practical problem for possible structural use of this material.

We have also done some measurements to try and investigate further the relationship between ductility and composition in these materials. We have measured the Ni EXAFS in 1% Fe doped NiAl, for room temperature versus 43°K, and compared our results to similar measurements on annealed, undoped, stoichiometric NiAl. We do find a somewhat larger temperature dependance in the Ni-Al mean squared relative displacement (MSRD) in the doped material indicating a slight weakening of the near neighbor Ni-Al bond due to Fe doping. This is interesting, but more work needs to be done on other compositions before we can speculate on the significance of this result.

(c) Development of Novel Experimental Techniques

At the time of submission of our previous proposal, we had carried out preliminary measurements characterizing our annular detection method of measuring glancing emergent angle (GEA) fluorescent EXAFS. We had previously demonstrated, without an annular detector, that the method enables one to obtain accurate EXAFS and XANES on bulk, concentrated absorbers, and that the method can be applied to the β phase aluminides. Because of the fact that the GEA method can be used for insulators and is readily adaptable to temperature dependant experiments in a duplex refrigerator, the GEA technique has certain advantages over the electron yield detection method. However, the GEA method can result in somewhat low count rates, and our annular silicon photodiode detection apparatus optimizes the counts and improves the convenience of the set up. We have now constructed a working GEA detector, based on the annular photodiode scheme, optimized the electronics, and characterized the improvements to data collection resulting from the annular detector variant to the GEA technique. These results, obtained in cooperation with Chuck Bouldin of NIST, are now published in Review of Scientific Instruments. By this time we have collected XAS data for concentrated absorbers in a number of alloy systems, by both electron yield and GEA. Our experience has been that often there are slight distortions introduced by the electron yield technique which we do not understand; we find

the GEA to be more reproducible and reliable as a probe of bulk properties, even for conducting samples.

In cooperation with Eric Jordan of the Mechanical Engineering Department at the University of Connecticut, we have been investigating a new method of measuring internal strain by seeding refractory test materials of aerospace interest with small (30 micron) particles and obtaining transmission radiographic images before and after strain using a double exposure. We had reported a proof of principle measurement at the time of our last proposal. We have now applied the method to a practical engineering material (graphite-epoxy composites) as well as to aluminum and titanium based test material. The collimated synchrotron radiation from the X-11 A line at the NSLS was used; recently by using a wiggler line at CHESS we have been able to image markers in titanium. The first paper on the method is now ~~submitted for publication~~ published, and included in the enclosed *publications*

Accurate position changes of the micro markers are obtained using a double exposure technique; where the sample is strained between exposures and the film is translated after the first exposure so that one does not have interference between the exposures. Marker shifts are detected using a combination of optical enlargement and laser imaging on centering diodes. We are investigating improving the resolution, which is already 100 microstrain, by imaging using the white line peak of small particles of heavy transition metals such as tungsten, thus using the enhanced white line absorption to enable us to use smaller marker particles. In conclusion, we note that this newly developed method is the only technique we know of for obtaining an internal strain map of stressed materials, and is of interest in characterizing novel, high performance aircraft materials and alloys.

We have written a proposal to the Connecticut Department of Economic Development to develop a versatile diffraction EXAFS apparatus (DAFS). The proposal was funded and including \$9,000.00 matching funds from the University of Connecticut Research Foundation, has given us a total of \$42,000.00 in equipment money to spend developing such a device. The apparatus is to be used in the alloy studies discussed in the present proposal. By monitoring diffraction peaks from thin alloy films, and maintaining the proper Bragg angle as the EXAFS is scanned, we should be able to obtain single crystal, polarized EXAFS on polycrystalline specimens (for example, TiAl, which forms as a layered

compound), obtain separate EXAFS for the same element occurring in different phases, and obtain EXAFS from different inequivalent sites of the same element in complex structures. Much of the basic physics of the concept has been worked out by others, and depends on the fact that there is an absorption factor component in the atomic scattering factors of the atoms in a unit cell. In principle, one can use the different weightings of different atomic structure factors in different reflections to separate the EXAFS from inequivalent atoms in the same unit cell.

Finally, we have purchased several calibration foils, consisting of different thicknesses of Cu deposited on glass slides and will use these to characterize our GEA technique for depth profiled EXAFS studies.

II. Studies of Rare Earth Transition Metal Nitrided Permanent Magnets

Introduction

The addition of nitrogen to the $(\text{RE})_2\text{Fe}_{17}$ systems substantially expands the lattice and brings about very large changes in magnetic properties. For example $\text{Sm}_2\text{Fe}_{17}$ shows strong uniaxial symmetry at high temperatures, as well as a large enhancement in magnetic ordering temperature T_c . In an effort to explore the local basis for the enhancement of the magnetic properties we are carrying out studies of the near edge (XANES) and EXAFS behavior in samples based on the $(\text{Re})_2\text{Fe}_{17}$ structure. Our goal is to explore the specific effects of the nitrogen on both the rare earth and Fe atoms in this structure. Does the nitrogen bond more strongly to the iron or to the rare earth? Does the lattice expand uniformly with the filling of the interstitial sites by the nitrogen? What is the specific effect of the nitrogen on the Fe and rare earth magnetic moments? To address these questions are some of the goals of our study.

To date we have prepared a number of $(\text{Re})_2\text{Fe}_{12}$ systems by general techniques such as rapid quenching, and initially have focussed on the Y_2Fe_{17} system which we have nitrated for a series of compositions up to $\text{N}=2.5$. Samples have been prepared which are single phase and with essentially no α iron. In addition to X-ray characterization, and magnetization studies we have carried out a series of NMR experiments, in which we have observed resonances from the Y, Fe, and nitrogen nuclei.

a) EXAFS study of nitrided versus non nitrided Y_2Fe_{17}

Our studies using NMR have yielded specific information about the distribution of N atoms in nitrided Y_2Fe_{17} . (The yttrium compounds are useful simplifying models for studying aspects of R_2Fe_{17} alloys, since Y does not carry an atomic magnetic moment, and the magnetization measured from the corresponding compound largely represents the Fe sublattice. Also, one can measure both the ^{89}Y and ^{14}N NMR signals.) From our nitrogen NMR studies, it is known that the NMR spectrum is single peak, verifying that N atoms only occupy the octahedral interstitial site in the Th_2Zn_{17} type structure; the ^{89}Y NMR studies show that there is a distribution of N atoms surrounding a Y atom. These results on the location of nitrogen atoms are in good agreement with previous EXAFS studies carried out by others.

Based on our experience using EXAFS as a local probe of lattice dynamics in TM-Al based alloys, we explored the EXAFS spectrum on nitrided versus nonnitrided Y_2Fe_{17} , using the Y absorption spectrum to monitor effects of "packing" N atoms into the lattice. Our first comparative results are shown in Fig. 4. One can see that the fourier transform (FT) is distinctly higher in the nitrided sample. At first glance this result might seem surprising, since NMR data show that there are an array of different environments surrounding each Y atom in nitrided Y_2Fe_{17} ; thus, one might expect by this last effect a lowering of the FT due to interference between the EXAFS arising from different environments. However, there may be a competing effect in that there may be "packing" of N around the Y atoms so as to reduce the thermal vibration of the lattice. (Recall our findings on the effects of "contacting Al spheres" in TM-Al based systems.) Another interpretation may be simply overlap between Fe and N contributions to the backscattering, resulting in some N area under the first FT peak. At present, the interpretation is speculative, and awaits our studies of temperature dependent EXAFS, which can be used to disentangle structural versus thermal disorder effects.

The above studies, however, point to the possibility of modifying the nitriding effect by making more "room" for the nitrogen; consequently, we for the first time have begun studies of the effect of La dopants in Y_2Fe_{17} nitrided versus non nitrided materials. We have made such samples by a rapid quenching technique. We find that we can dissolve La into Y_2Fe_{17} up to 10 atomic percent. The lattice expands, due to the substitution of La, in

an anisotropic way, as we have determined by x-ray diffraction (XRD). The basic R_2Fe_{17} phase appears significantly more "stable" to heat treatment and nitriding as a result of La substitution. Thus, when we nitride Y_2Fe_{17} at 550°C, unwanted α iron appears and the R_2Fe_{17} diffraction peaks become weak. On the other hand, this effect is not observed in La doped material; also we can insert more nitrogen into the La doped material. We have performed thermogravimetric analysis (TGA) experiments as a follow up. Preliminary studies indicate that heating nitrated Y_2Fe_{17} to 500°C results in a phase separation whereas this result is not seen in the La doped, nitride material. We intend to extend these studies in order to study the variation in Sm_2Fe_{17} magnetic properties due to La doping. A side benefit of studying La in these systems is the availability of a dipole x-ray XANES transition to unoccupied 5d states, which we can monitor using a convenient x-ray wavelength. We can thus investigate local d electron charge transfer or bonding effects in La doped systems.

We have obtained new results in which we obtain, EXAFS data for both the RE and Fe edges in nitrated versus non nitrated Y_2Fe_{17} .

A cursory inspection of the data would seem to indicate that the addition of nitrogen clearly expands the structure local to the RE site, but it is not so clear that the expected uniform expansion is found local at the Fe sites: However, because the Fe edge is comprised of EXAFS from four inequivalent Fe sites, a firm conclusion must await further analysis and studies, to be described later. We point out, however, that in other alloy systems, EXAFS investigations have often found that overall expansion of a unit cell due to alloying substitution does not, in general, translate into uniform expansion local to particular sites. If one could obtain data relevant to the local expansion, relative to Fe sites, due to nitriding, this would be most useful in testing a model that assumes the iron moment enhancement, due to nitriding, is strictly due to increased, uniform separation of Fe-Fe distances and consequent enhancement of the exchange interaction.

We have also obtained Gd L_3 and L_2 XANES data on Gd_2Fe_{17} and $Gd_2Fe_{17} N_x$. These XANES spectra probe, by a dipole allowed transition, unoccupied local RE 5d states.

We observe a small, but definite sharpening and narrowing of the white lines due to nitriding. (This effect is also evident from inspection of the data of Capehart, et. al. on the L_3 edges of $Sm_2Fe_{17}N_x$; these authors did not comment

on this aspect of their data.) The fact that this white line sharpening is observed due to nitriding in both the Gd and Sm based systems is of interest. A simple interpretation is "narrowing" of the local RE 5d band due to lattice expansion, an observation which may pertain to interpretations of magnetic alignment in these systems associated with interactions between RE and Fe d electrons.

III. Studies of Transition Metal X-Ray Spectra as Related to Magnetic Moments

At the time of our last proposal, we had begun a theoretical study of the L_3/L_2 emission ratio in X-ray emission spectroscopy (XES) of 3d transition metals. A motivating idea was that, whereas in absorption, the L_3/L_2 "white line" ratio depends on magnetic moment, in emission the influence of moment should be much weaker since both majority and minority spin bands contribute to the XES. Therefore, if the L_3/L_2 emission ratio could be obtained, perhaps this quantity could ultimately be subtracted from the absorption ratio, isolating the magnetic moment influence on the absorption edge line ratio in a simple way.

We report that we have completed one step in this procedure, in that the L_3/L_2 ratio in emission, corresponding to the absorption ratio studied by Leapman and Grunes, has for the first time been isolated. Using a previously developed formalism to correct XES for Coster-Kronig distortions we have obtained the L_3/L_2 ratio for the $2p \rightarrow 3d$ emission of elements Ti through Cu (excluding V and Mn) and compared this result to previous appearance potential spectroscopy and electron energy loss spectroscopy (EELS) results. This study has now been accepted by Phys. Rev. B. We find a magnetic moment enhancement in the absorption relative to the emission ratio, as hypothesized, but evidence that the absorption ratio in nickel metal is also enhanced by a very small spin orbit splitting of the 3d band, resulting in an increased concentration of $d_{5/2}$ relative to $d_{3/2}$ holes. L_3/L_2 ratios for experimental XES, soft X-ray appearance potential spectroscopy (SXAPS), and EELS are all shown, in the above publication, to be related in a sensible manner.

Figure 2b: Comparison of the temperature dependence of σ^2 for the first three coordination shells of NiAl calculated from the force-constant model (continuous line) and obtained from the EXAFS spectra (points).

Figure 2 Comparison of the temperature dependence of σ^2 for the first three coordination shells of FeAl and NiAl. σ^2 of NiAl is calculated from the force-constant model (continuous line) and σ^2 of FeAl is obtained from the EXAFS spectra (points).

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