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Nuclear Magnetic Resonance Study of Al^{27} in NiAl*

J. A. SEITCHIK† AND R. H. WALMSLEY
Department of Physics, University of Pennsylvania, Philadelphia, Pennsylvania

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The NMR of Al^{27} has been studied in two samples of the bcc ordered alloy NiAl whose atomic compositions were 48% Ni, 52% Al and 50% Ni, 50% Al. The line intensity of the first sample is accounted for by assuming all satellite transitions wiped out by quadrupolar interactions with defects. The width of the observed $-\frac{1}{2}\rightleftarrows\frac{1}{2}$ transition was 4.5 ± 0.3 Oe, field- and temperature-independent, and can be almost entirely accounted for by Al^{27} dipole-dipole interactions. The second sample had an 11% greater width and 85% greater intensity, indicating contributions from satellite transitions. The Knight shift of both samples was $0.055\pm0.005\%$, which is only one-third that of pure aluminum, and was independent of magnetic field and temperature. The specific heat of NiAl was measured from 2.2 to 4.2°K, giving $\gamma_{el}=12.6\times10^{3}$ ergs $mole^{-1}$ $°K^{-2}$, which is 87% of the value for pure aluminum. We conclude that the low value of the Knight shift results from a small Fermi-contact interaction at the aluminum sites for electrons at the Fermi surface and is not due to a low density of states.

INTRODUCTION

THE usefulness of nuclear magnetic resonance (NMR) in studying the electronic structure of pure metals and alloys has been recognized for many years.[1,2] It has been exploited only recently in the study of metallic systems with high concentrations of transition elements.[3-8] While a few of these investigations have been carried out in disordered alloys,[7,8] generally, in order that the line should not be lost by inhomogeneous magnetization broadening and quadrupolar broadening, the system must be highly ordered. Specifically, the study of NMR on the nuclei of nonmagnetic elements in ordered binary alloys with transition elements[3-6] promises to shed light upon the role of the conduction electrons in the coupling between magnetic ions.[9-12] Until the present, the work on such systems of the first transition series includes CrB_2, VB_2, and V_3X where X is Sn, Si, Sb, or Ga.[4,5]

In this and in a forthcoming paper, we shall present the results of a study of ordered aluminum binary alloys with iron, cobalt, and nickel: FeAl, CoAl, and NiAl. These alloys are body-centered cubic structures, CoAl and FeAl being strongly paramagnetic and NiAl being essentially nonmagnetic ($\chi\approx12\times10^{-6}$ cgs per mole). The ordered alloy Fe_3Al is ferromagnetic at room temperature and is not included in the present work.

The purpose of this study of NiAl is twofold. First, it contributes to the general state of knowledge of a system which has been the object of continuing investigation and interest for a number of years.[13-18] Second, it is the nonmagnetic analog of CoAl andFeAl and, hence, provides the necessary reference for distinguishing between magnetic and nonmagnetic effects on the NMR of these systems.

CRYSTAL STRUCTURE

In the vicinity of equal atomic concentrations, the melting point of nickel-aluminum alloys rises very sharply to a maximum at the stoichiometric compound NiAl. This compound has the cesium chloride structure and is ordered at all temperatures in the solid phase with nickel atoms on body-center sites, say, and the aluminum atoms on the cube-corner sites. If an excess of aluminum is present, the aluminum atoms first replace nickel on the body-center sites until an excess of one atomic percent has been reached (49.5% Ni, 50.5% Al). Further deviations in stoichiometry are accomplished by simply leaving randomly distributed vacancies on body-center nickel sites.[13]

The valence electron to atom ratio of NiAl is thought to be 3:2.[16] This is consistent with the observed body-centered cubic structure favored by this ratio as well as the fact that aluminum is electropositive with respect to nickel. Thus, the nickel atom achieves a nonmagnetic $3d^{10}$ configuration and contributes no electrons to the conduction band, while the aluminum atom contributes

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[16] G. Raynor, in *Progress in Metal Physics*, edited by B. Chalmers (Interscience Publishers, Inc., New York, 1949), Vol. 1, p. 15.

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its three valence electrons. From this point of view, the vacancies occurring in aluminum-rich stoichiometries are thought to result from the high stability of the three electrons per unit cell concentration which is preserved by maintaining just one aluminum atom per unit cell. On the other hand, in nickel-rich stoichiometries the nickel atoms simply replace aluminum atoms and the electron concentration is reduced. A similar situation occurs in CoAl but not in FeAl.[18] Such behavior indicates a highly ordered structure; however, some disorder has been shown to exist in NiAl when quenched from high temperatures.[17]

THE SAMPLES

Two samples of NiAl were used for this study, each one taken from separately cast ingots very kindly supplied by the New Kensington Laboratories of the Aluminum Company of America. Since NiAl is extremely brittle, coarse granules were obtained from the ingots by shattering them. The granules were then ground to a powder with a porcelain mortar and pestle and sieved with a 325-mesh screen to yield the sample material. This powder was given a heat treatment to promote ordering and annealing by heating it *in vacuo* to 700°C and cooling to room temperature over a period of three days. Finally, it was magnetically cleaned, although almost no observable contamination was found. Some observations were made on unannealed powders and will be discussed separately. A portion of the final specimen was then x-ray photographed to establish the crystal structure and order, while a sample of the original ingot was sent to a commercial laboratory for chemical and spectrographic analysis. Table I gives the stoichiometry of the two samples together with the concentrations of their principal impurities. Whereas Sample II was close to the NiAl stoichiometry, Sample I showed a 4 at.% excess of aluminum over nickel.

TABLE I. Composition of samples.

Element	Sample I (at.%)	Sample II (at.%)
Nickel	48.0	50.1
Aluminum	51.8	49.7
Chromium	<0.004	<0.004
Cobalt	0.047	0.042
Copper	0.005	0.005
Iron	0.072	0.088
Lead	<0.001	<0.001
Magnesium	0.040	0.026
Manganese	0.003	0.002

EXPERIMENTAL DETAILS

The NMR spectrometer used is of the Pound-Watkins type employing 400-cps field modulation and field derivative output on a Brown potentiometer pen recorder. The magnet is a 12-in. Varian with a 3-in. gap, tapered pole caps, and a field inhomogeneity over our specimen volume (≈ 0.6 cm^3) of $\pm\frac{1}{4}$ Oe. To make the Knight-shift measurements at all temperatures, a small amount of copper powder was mixed with the sample and the Cu^{63} resonance used as a reference. A separate measurement of the aluminum resonance in a solution of $AlCl_3$ with respect to Cu^{63} provided the sample Knight shifts by subtraction. The Dewar assembly and cryogenics apparatus were conventional, liquid nitrogen and helium being the refrigerants.

LINE SHAPES AND INTENSITIES

The derivative of the absorption curve of each sample was observed at 293, 77, and 4.2°K in magnetic fields of 6000 and 10 000 Oe. In each case the line shape and width were found to be independent of temperature and magnetic field. In linewidth measurements the sample sizes were reduced to eliminate inhomogeneous magnetic-field broadening.

Sample I

The absorption curve for Sample I was symmetric and very nearly Gaussian in shape. The peak-to-peak derivative width δH was 4.5 ± 0.3 Oe.

A. Dipolar Broadening

The dipolar contribution to the linewidth from the simple cubic array of aluminum nuclei is given by the Van Vleck second-moment formula[19] as

$$M_2=\frac{5.1\gamma^4\hbar^2 I(I+1)}{d^6}, \qquad (1)$$

where M_2 is the second moment of the line in frequency units, γ and I are the aluminum nuclear gyromagnetic ratio and spin, and d is the aluminum internuclear distance. For a Gaussian line shape Eq. (1) gives $\delta H = 4.1$ Oe, which accounts for almost the entire linewidth. Although there is at present some question as to whether the nickel moment is 0.3 nm or 0.9 nm,[20] neither value is sufficiently large to make an observable contribution to the aluminum NMR linewidth.

B. Relaxation Broadening

The Korringa relation[21] between the spin lattice relaxation time T_1 and the measured Knight shift $\Delta H/H$ is given by

$$T_1\left(\frac{\Delta H}{H}\right)^2=\frac{\mu_B^2}{h\pi kT\gamma^2}, \qquad (2)$$

where μ_B is the Bohr magneton, k is Boltzmann's constant, and T is the absolute temperature. Using our

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measured value for the Knight shift, we find $T_1=4.4\times10^{-2}$ sec at 293°K. Hence, spin-lattice relaxation is not expected to contribute to the linewidth at room temperatures or below.

C. Quadrupolar Interactions

Since Sample I was aluminum rich and not all aluminum nuclei were in sites of cubic symmetry, quadrupolar interactions could be expected to affect the line intensity. The "all-or-nothing" model of quadrupolar induced-frequency shifts,[22,23] which is commonly used in treating dilute alloys, assumes that the resonant frequency of an aluminum nucleus is shifted into regions where the line intensity is below noise if any one of its N nearest neighbors is a defect. If, on the other hand, none of the N sites is a defect, the frequency is taken to be essentially unshifted, and there is little broadening. Using this model the dependence of the line intensity on the defect concentration can be computed in the following way. Let C_1 and C_2 be the defect concentrations on the cube corner and cube-center sites, respectively. Considering an aluminum atom on a cube corner site, the probability that none of the nearest N_1 cube-corner sites and that none of the nearest N_2 cube-center sites be a defect is given by

$$(1-C_1)^{N_1}(1-C_2)^{N_2}. \tag{3}$$

In aluminum-rich NiAl, C_1 is zero. Accordingly, the line intensity I, as a function of the total defect concentration C, should vary as[24]

$$I=I_0(1-2C)^N, \tag{4}$$

where N refers to the cube-center sites only (cube-corner sites only, in nickel-rich NiAl).

In first order, quadrupolar interactions affect transitions between all nuclear-energy levels except the $-\frac{1}{2}\rightleftarrows\frac{1}{2}$ transition. The $-\frac{1}{2}\rightleftarrows\frac{1}{2}$ transition is affected in second order, and it is clear that N need not be the same for this transition as for the others, usually called satellite transitions. For example, manganese substituted into pure aluminum results in $N=130$ for the satellite transitions and $N=12$ for the $-\frac{1}{2}\rightleftarrows\frac{1}{2}$ transition.[23] Since the relative intensities of the aluminum transitions ($I=\frac{5}{2}$) are in the ratio of 5:8:9:8:5, 9/35 of the intensity contribution of a given nucleus comes from the central transition, and 25/35 comes from the satellites. Consequently, in the presence of defects the "all-or-nothing" model predicts the intensity of the resonance relative to the intensity for the same number of nuclei in the absence of defects to be given by (assuming identical shapes for the central and satellite lines)

$$I=I_0\left[\frac{9}{35}(1-2C)^N+\frac{26}{35}(1-2C)^{N'}\right], \tag{5}$$

where N characterizes the central transition and N' the satellites. For a Gaussian or Lorentzian line, the peak-to-peak intensity of the derivative curve is proportional to the number of nuclei and inversely proportional to the square of the peak-to-peak derivative line width δH. If the peak-derivative intensity I_1 for a sample of NiAl containing a known number n_1 of aluminum atoms is compared to the intensity I_2 of a sample of pure aluminum containing n_2 atoms, then for identical line shapes we have the relation

$$\left[\frac{9}{35}(1-2C)^N+\frac{26}{35}(1-2C)^{N'}\right]=\frac{\delta H_1{}^2I_1n_2}{\delta H_2{}^2I_2n_1}. \tag{6}$$

(It has been shown that both central and satellite transitions contribute to the observed line in pure aluminum.[23])

Before attempting to determine N and N' in Eq. (6), we must determine the proper value of the defect concentration. Since Sample I is aluminum rich, all cube corners are occupied by aluminum atoms, and the defects are the aluminum atoms and vacancies located on the body-center sites. In this sample, then, for each 104 aluminum atoms and 96 nickel atoms, there are 103 unit cells whose corners are occupied by 103 aluminum atoms and whose body-center sites are occupied at random by 96 nickel atoms, one aluminum atom, and six vacancies. Therefore, the defect concentration is $C=3\frac{1}{2}\%$.

The right-hand side of Eq. (6) was experimentally determined to be 0.23 ± 0.04, where the estimate of error does not include the uncertainty due to the fact that the line shapes of NiAl and pure aluminum are not exactly identical. Since $9/35=0.257$, one is led to take $N=0$ and N' very large. If we were to take $N=8$ (first nearest neighbors), the value for N' resulting from Eq. (6) is from three to four times smaller than the values found in experiments on dilute alloys.[23] Furthermore, with $C=3\frac{1}{2}\%$, $n=8$, and assuming all satellites wiped out, the resulting intensity would be far less than that which is observed. We conclude then that the assumption of $N=0$ is correct. With $N=0$, the experimental accuracy is not sufficient to permit a meaningful calculation of N'.

Since those aluminum nuclei on the body-center sites have eight aluminum atoms as first nearest neighbors, at least their central transitions will contribute to a resonance at some frequency as yet unknown. This leads to a slight ambiguity in the proper value to be used for n_1 in Eq. (6) since these nuclei might or might not be contributing to the observed line. In either case the ambiguity is only 1%, which is less than the accuracy of the intensity measurements at room temperature. At 4.2°K, however, the signal-to-noise ratio was sufficiently high to detect the resonance of those nuclei in principle. Since no separate line was observed, we conclude that it was masked by the main line.

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[22] N. Bloembergen and T. Rowland, Acta Met. **1**, 731 (1953).

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[24] For large concentrations, configurations of defects that do not produce a quadrupolar field at an atomic site become important; see D. Weinberg, J. Phys. Chem. Solids **15**, 249 (1960).

Sample II

The line shape for Sample II was symmetric but was 11% wider than that of Sample I and 85% greater in peak intensity. These differences are clearly due to the contributions to the line of first-order quadrupolar broadened satellite transitions since this sample had a relatively low concentration of defects.

Cold Working

The effects of cold working on the resonance lines of the two samples were observed in runs made on the sample powders before annealing. For the unannealed Sample I powder, the resonance line was found to be broadened considerably and to be less intense than the annealed powder resonance line. On the other hand, the unannealed Sample II powder exhibited a comparatively slight broadening and intensity decrease.

Similar intensity decreases found in pure metals are attributed to quadrupolar effects arising from dislocations introduced by cold working. Copper, for example, is very sensitive to cold working, whereas aluminum is not, and x rays show a difference in dislocation density of two orders of magnitude. However, if 0.64% magnesium is added to aluminum, cold working leads to a pronounced intensity decrease.[23] This is thought to result from the impurity atoms pinning down the dislocations, which in the pure metal would be free to diffuse out.

By analogy it appears that 50-50 NiAl has a relatively low dislocation density at room temperature after cold working, and that the defects due to departures from that stoichiometry serve as effective dislocation trapping sites.

Further Discussion

The line shapes for the two samples were different, Sample II having greater area in the wings. A convenient indication of the line shape is provided by forming the ratio of the peak-to-peak derivative width δH to the half-intensity width $\Delta H_{1/2}$ of the absorption curve. Table II gives this ratio for both samples as well as for Gaussian and Lorentzian curves. The line shape of Sample II appears to result from a mixture of a Gaussian line from the central transition and a Lorentzian or nearly Lorentzian line from the quadrupolar broadened satellite transitions. Values for $\Delta H_{1/2}$ were obtained by integrating the derivative of the absorption curve with a planimeter.

TABLE II. Ratio of the peak-to-peak derivative width to the half-intensity width of absorption curves.

Absorption curve	$\delta H/\Delta H_{1/2}$
Gaussian	0.85
Lorentzian	0.58
Sample I	0.82±0.08
Sample II	0.65±0.08

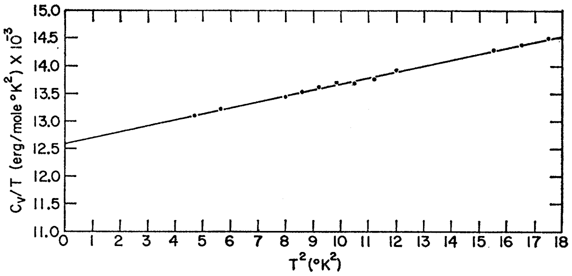

FIG. 1. A plot of C_v/T versus T^2 for NiAl between 2.2 and 4.2°K.

KNIGHT SHIFT AND ELECTRONIC SPECIFIC HEAT

The Knight shifts of the two samples were measured at 293, 77, and 4.2°K in magnetic fields of 6500 and 10 000 Oe. The Knight shift in each case was found to be field and temperature independent. The value of the shift was the same for both samples and equal to 0.055±0.005%, which is unusually low. (The value in pure aluminum is 0.162%.[25]) Since the Knight shift is proportional to both the density of states at the Fermi level and the fractional *s* character on the aluminum sites of the states averaged over the Fermi surface, an unusually small value of either can lead to the observed small Knight shift. Accordingly, the specific heat of NiAl was measured from 2.2 to 4.2°K in order to obtain the electronic contribution which is proportional to the density of states. We are indebted to Brooks Low of this Laboratory for making these measurements for us. A plot of C_v/T versus T^2 is shown in Fig. 1, yielding a coefficient of the term linear in T of $\gamma_{\mathrm{el}}=12.6\times10^3$ ergs mole^{-1} °K^{-2}. This is 87% of the value of 14.5×10^3 for pure aluminum,[26] and so the low shift is attributable to lack of *s* character on the aluminum sites in the wave functions.

In the tight-binding approximation the electronic wave functions may be written

$$\psi_{\mathbf{k}}(\mathbf{r})=\frac{1}{\sqrt{N}}\sum_{m,j}e^{i\mathbf{k}\cdot\mathbf{R}_m}a_j\varphi_j(\mathbf{r}-\mathbf{R}_m)\,,\quad \sum_j|a_j|^2=1\,,\tag{7}$$

where the φ_j are normalized and localized functions centered on the jth atom in the mth cell, and N is the number of unit cells. The Knight shift on l-type sites is then given by[27]

$$K_l=\frac{(H_{\mathrm{hf}})_l}{\mu_B}\chi_a n|a_l|^2\xi_l\,,\tag{8}$$

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[25] W. Knight, in *Solid State Physics*, edited by F. Seitz and D. Turnbull (Academic Press Inc., New York, 1956), Vol. 2, p. 93.

[26] J. A. Kok and W. H. Keesom, Physica 4, 835 (1937).

[27] See, for example, A. M. Clogston and V. Jaccarino, Phys. Rev. **121**, 1357 (1961).

where $(H_{\mathrm{hf}})_l$ is the hyperfine field per electron of the free atom, μ_B is the Bohr magneton, χ_a is the magnetic susceptibility per atom, n is the number of atoms per unit cell, and ξ_l is the ratio of $|\varphi_j(0)|^2$ in the metal to its value in the free atom. The appropriate formula for the band approximation can be obtained from Eq. (7) by putting $n|a_l|^2=1$ and considering ξ_l to be averaged over all states at the Fermi surface. The ratio of the Knight shift of the aluminum nuclei in NiAl, K_1, to its value in pure aluminum, K_2, is

$$K_1/K_2=(\chi_a^{(1)}/\chi_a^{(2)})(2|a_1|^2\xi_1/\xi_2). \tag{9}$$

Substituting $K_1=0.055\%$, $K_2=0.162\%$, $\chi_a^{(1)}/\chi_a^{(2)}=\gamma_{\mathrm{el}}^{(1)}/\gamma_{\mathrm{el}}^{(2)}=0.87$, and $\xi_2=0.36$, we obtain[25]

$$2|a_1|^2\xi_1=0.13 \tag{10}$$

and find that indeed there is little s character in the wave functions at the Fermi level to be associated with the aluminum sites.

Further Discussion

The closeness in value of γ_{el} for NiAl and pure aluminum suggests a band approximation for the states at the Fermi level. To check this we can compute the magnetic susceptibility from the specific-heat data and compare with the susceptibility measurements made by Höhl.[15] The total susceptibility per mole is (cgs units are used throughout)

$$\chi=\chi_{\mathrm{spin}}+\chi_{\mathrm{orbit}}+\chi_{\mathrm{ions}}. \tag{11}$$

In the one-electron picture

$$\chi_{\mathrm{spin}}=3\mu_B^2\gamma_{\mathrm{el}}/\pi^2k^2=17.3\times10^{-6}, \tag{12}$$

where k is Boltzmann's constant. In the effective-mass approximation

$$\chi_{\mathrm{orbit}}=-\tfrac{1}{3}(m/m^*)^2\chi_{\mathrm{spin}}. \tag{13}$$

The effective-mass ratio is related to the specific heat by

$$m^*/m=3\hbar^2\gamma_{\mathrm{el}}/mk^2(3\pi^2N)^{1/3}, \tag{14}$$

where N is the number of electrons per unit volume to be accommodated by the band. The effective-mass ratio is insensitive to N, and we shall choose three electrons per unit cell corresponding to the three valence electrons of aluminum and a closed nickel d shell. This yields a value of $m^*/m=2.2$ and $\chi_{\mathrm{spin}}+\chi_{\mathrm{orbit}}=16.1\times10^{-6}$.

The diamagnetic contribution of the aluminum and nickel ions is somewhat uncertain since it depends upon the effective radial distribution of the nickel $3d$ shell, which may be substantially different in the metal than in the free atom.[28] To make this estimation, we shall use a value for the copper ion in copper metal which is obtained by subtracting $\chi_{\mathrm{spin}}+\chi_{\mathrm{orbit}}$ from the measured total χ. This seems reasonable since the atomic volume is the same in copper and NiAl. The spin and orbit susceptibilities for copper are calculated as above using the measured values for copper of $\gamma_{\mathrm{el}}=6.88\times10^3$ erg mole^{-1} °K^{-2} and $m^*/m=1.5$.[29] Taking $\chi=-5.27\times10^{-6}$,[30] we obtain $\chi_{\mathrm{ion}}=-13\times10^{-6}$. Using half this value per mole of NiAl (the aluminum ion contribution is negligible) we find a total susceptibility for NiAl of $\chi=10\times10^{-6}$. This agrees well with Höhl's result of 11×10^{-6} to 13×10^{-6}[15] in the relatively temperature-independent region above room temperature.

SUMMARY AND CONCLUSIONS

Intensity studies of the Al^{27} resonance in NiAl show that at the stoichiometric composition 50 at.% Ni, 50 at.% Al, both the central and satellite transitions contribute to the observed resonance in the well-annealed material. In cold-worked specimens, the satellite contribution is reduced while the central transition remains unaffected. In contrast to this, for NiAl with the composition 48 at.% Ni, 52 at.% Al the satellite transitions are wiped out even in the annealed material, leaving only the full-strength central transition. Cold working this material reduces the intensity of the central transition.

The Al^{27} Knight shift is $0.055\pm0.005\%$ for both compositions, which is only one third the value for pure aluminum metal. However, specific-heat measurements in NiAl show that at the Fermi level, the electron density of states per atom is 87% of the pure aluminum value. Thus, we conclude that the conduction electrons at the Fermi surface have limited s character on the aluminum sites.

The specific-heat measurements yield $\gamma_{\mathrm{el}}=12.6\times10^3$ ergs mole^{-1} °K^{-2}. This is consistent with the value deduced from Höhl's magnetic susceptibility measurements when corrections are made for the diamagnetism of the conduction and nickel core electrons.

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[30] R. Bowers, Phys. Rev. **102**, 1486 (1956).