High-precision measurement of electrical resistivity of nickel near the ferromagnetic phase transition at high pressure

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High-precision measurement of the resistance of nickel near its ferromagnetic phase transition at high pressure was done in a large volume hydrostatic cubic multiple-anvil pressure cell between 25 and 45 kbar. A real-time computer-controlled measurement system was developed to measure the resistance vs temperature and pressure. The values of the critical exponent α and amplitude ratio of the leading term A/A' are in good agreement with the results published by Ahlers and Kornblit from specific-heat measurement and by Källbäck *et al.* in a resistance measurement at atmospheric pressure. They are found to be independent of pressure. The critical temperature T_c increases linearly with pressure at a rate of 0.193 ± 0.013 K/kbar. The amplitude ratio for the correction to scaling term D/D' has also been determined. There is considerable uncertainty in the measurement but the data may indicate a variation with pressure contrary to expectations of universality.

INTRODUCTION

The discontinuities in physical behavior which occur when a system undergoes a phase transition have claimed the attention of scientists for many years. Particular interest has been focused on phenomena associated with critical points. In recent years there has been considerable progress toward a greater understanding of phase transitions and critical phenomena, and the research literature has grown rapidly. One of the major theoretical advances is the development of the renormalization-group theory[1] of critical phenomena. This theory provides a method to calculate the critical parameters. The experimental literature is not as prevalent and often lacks the precision required to make a good comparison with the theory.

Theory predicts that if a field variable, for instance, pressure, does not alter the symmetry of the ordered state, all the quantities which characterize a given universality class remain unchanged by the variation of this field variable. Our motivation for this research is to test this postulate. It has been shown that the magnetic energy of a metallic ferromagnet in the vicinity of the critical temperature is proportional to the spin-dependent electrical resistivity.[2–4] In fact, the singular parts of the temperature dependence of the specific heat and of the temperature derivative of the resistivity are the same.[5,6] This implies that the specific-heat critical exponent, α, and amplitude ratios can be determined either from a specific-heat measurement or from a resistance measurement. Electrical resistance can be measured with a precision far better than that realized in specific-heat measurements. On the other hand, since the resistance is less singular at T_c than is the specific heat, a higher accuracy is required in a measurement of the resistance in order to obtain equally good estimates for the critical parameters. A specific-heat measurement is very difficult in a high pressure environment, where everything is in intimate thermal contact, so to test pressure effects on critical phenomena a high precision resistance experiment is preferred. The ferromagnetic phase transition in nickel is second order so one can readily approach the region where critical phenomena is dominant. Therefore, we have measured the resistance near the ferromagnetic phase transition in nickel to pressures of 45 kbar.

There are two recent electrical resistance measurements near the nickel ferromagnetic phase transition which have been published. A high precision measurement at atmospheric pressure was reported by Källbäck *et al.*[7] in 1981. Their data gave excellent experimental critical parameters for this Heisenberg ferromagnet but differed somewhat from the results of RG theory accepted at that time. Another measurement of nickel electrical resistance versus temperature over a range of pressure to 47 kbar was reported by Yousuf *et al.*[8] in 1986. A few years later they reanalyzed this data[9] to get critical exponents and amplitude ratios versus pressure. They claimed that their results were consistent with universality and found critical parameters that agreed exactly with early results of RG theory.

Any significant data analysis of such measurements, which can provide evidence to test inferences of the RG theory, must be based on a set of high precision experimental data such as that of Källbäck *et al.* The data and analysis of Yousuf and Kumar[8,9] cannot match the required precision because (1) of the nonhydrostatic environment of their pressure cell, (2) of the extremely small sample and associated difficulty of measuring a small resistance (1 mΩ), and (3) the temperature control was only $\pm 1\,°$C. The scatter of their resistance data versus temperature appears at least in the third digit. Stimulated by the attempt of Yousuf *et al.*, we decided to measure the resistance of pure nickel versus temperature and pressure in a hydrostatic environment and to provide high precision data for testing pressure dependence of the critical parameters. We have succeeded in achieving an accuracy in measured resistance versus temperature at high pressures which yields data good enough to serve as a basis for a nonlinear analysis near the second-order phase transition of nickel.

EXPERIMENTAL

The measurement was done in a large volume hydrostatic cubic multi-anvil pressure cell. The high pressure system[10] consisted of a six-anvil, 400-ton cubic press whose anvils compressed a pyrophyllite cube (3.4-cm edge length) as the pressure transmitting medium. Within the cube was a cylindrical thin-wall Inconel tube serving both as a furnace to attain the required temperature and also to contain the liquid (petroleum ether 30–60 °C b.p.) that provided the hydrostatic medium. Within the liquid was a 0.005-mm diameter 5-cm length of 99.997% purity Ni wire wound on a vespel form along with a Chromel-Alumel thermocouple to monitor the temperature. Tapered high density polyethylene plugs sealed the ends of the furnace to contain the liquid.

Temperature uniformity within the high pressure liquid chamber was achieved by placing thermally insulating vespel rings around the ends of the tube furnace to alter the heat flow.[11] The pyrophyllite cubic cell, tube furnace with minimized temperature gradients, thermocouple, and the nickel sample are assembled as shown in Fig. 1. An insert in this figure shows the winding of the nickel wire on the vespel form. The sample had a resistance of 1 Ω at room temperature and about 4 Ω at the critical temperature. All electrical leads pass out of the furnace area through the polyethylene plugs.

The resistance was measured by a four-lead technique. A constant current (4 mA) passes through a standard resistor in series with the nickel sample. The voltages across the standard resistor and the sample are measured by HP-3456A and HP-3457A multimeters, respectively, and resistance of the nickel is calculated. The emf of the thermocouple is monitored and converted to temperature in the computer. All procedures of the measurement are controlled by a HP-9816 computer. The temperature control and the determination of the temperature equilibrium in the measurement are accomplished by software written in Rocky Mountain BASIC. The communication between the computer, multimeters and digital-to-analog converters are via an IEEE-488 bus.

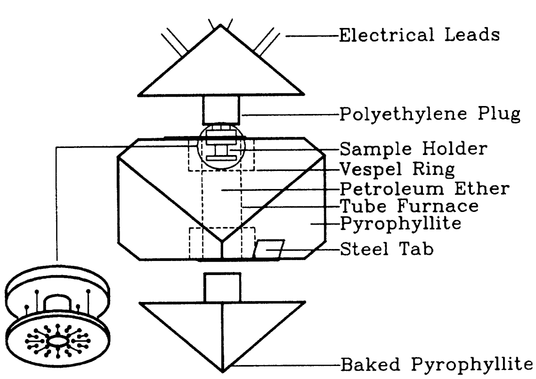

FIG. 1. The high pressure cubic cell showing the placement of the tube furnace, the vespel rings for reducing the temperature gradients, the thermocouple, and the nickel sample wound on a vespel coil.

The leads to the nickel sample were 0.05-mm diameter nickel wire to reduce the effects of thermal emf's. Using small diameter leads also helps to reduce heat flow out of the sample region. These nickel leads were attached to 0.25-mm diameter Chromel wire with poor thermal conductivity to pass through the polyethylene plug and out of the pressure cell. This stronger wire was necessary to keep from breaking the lead wires in the gasket area. For the purpose of keeping heat flow from the sample area small, very thin (0.076-mm diameter) thermocouple wires were used in the liquid region which were attached to 0.25-mm diameter thermocouple wires to exit from the pressure cell through the gasket.

The atmospheric pressure calibration of the thermocouples has an absolute temperature accuracy of ±1.2 °C in the temperature range of the experiment. There is a small correction to the temperature, between 0.7 and 1.1 °C in the range of interest to this experiment, due to a pressure effect on the thermocouple emf. We used an average of this correction measured by Hanneman, Strong, and Bundy[12] and Getting and Kennedy.[13] Due to the difference in their reported results this alters the absolute temperature accuracy to ±1.5 °C. Each run consisted of a measurement of resistance versus temperature at constant press load. The pressure increases by about 1 kbar and the relative temperature measurement may differ by as much as ±0.5 °C over the range of the temperature measured. The temperature error between consecutive points however is less than 0.01 °C. All measurements were made on the same pressure run so the relative temperature error over the pressure range, for the small range of T_c, comes only from the relative uncertainty in the pressure correction on the thermocouple emf. There could be a systematic error of about ±0.2 °C in this correction.

Two digital-to-analog converters, one for coarse and the other for fine control, are used for controlling the power supplies generating the temperature. When the desired temperature is reached the coarse control remains constant and the fine control is varied to regulate the power supply output with a temperature resolution of 0.002 °C. Two Kepko power supplies operating in parallel provide about 130 amperes for the tube furnace to attain the temperature of the measurement. The noise level of the temperature control system is in the range ±0.005 °C around the set temperature. This allows a precision in resistance measurement of the order of 1 part in 10^5. The temperature is stepped in 0.5° intervals and a resistance measurement is made after the temperature has been stable to ±0.01 °C for a time interval of greater than 1 min.

The temperatures of interest in the measurement are in the range of 335–390 °C. A Manganin pressure sensor cannot be used in this temperature range, therefore the press load was used to calculate the pressure according to a pressure versus load calibration curve.[14] Because of thermal expansion of the internally heated pressure cell the actual pressure in the cell increases by about 0.016 kbar per degree[15] above the room-temperature calibration. The accuracy of the pressure measurement is about ± 0.7 kbar near 25 kbar to ± 1.1 kbar near 45 kbar. The lowest data point is at 25 kbar because one must be at a sufficiently high pressure to seal the gaskets against internal expansion as the temperature is raised. No zero pressure measurement was made because we have no good temperature control system at atmospheric pressure.

RESULTS AND DATA ANALYSIS

We measured the nickel resistance versus temperature at several fixed pressures chosen between 25 and 45 kbar with a 5-kbar increment. The temperature range scanned was approximately from 335 °C to 390 °C with an increment of 0.5 °C. The resistance versus temperature corresponding to different pressures is shown in Fig. 2. The resistance decreases and the critical temperature T_c increases versus pressure. The data is first fitted by nonlinear least-squares techniques to the following function[7] with $z=0.57$ and R_0, R', A', A, α, D', D, and T_c as variable parameters:

$$R(t)=R_0+R't+\begin{cases} A'|t|^{1-\alpha}(1+D'|t|^z), & (t<0)\ ; \\ At^{1-\alpha}(1+Dt^z), & (t\geq 0)\ . \end{cases} \tag{1}$$

Expression (1) gives rise to a function with confluent singularities when the reduced temperature $t=T/T_c-1$ approaches zero. The parameters R_0 and R' are the magnitude and slope of the background (noncritical or nonmagnetic part) of the electrical resistance. The parameter α is the critical exponent with A and A' as the critical amplitudes for the leading critical term. The parameter z is the exponent and D/D' the amplitude ratio for the second-order correction to scaling or the confluent singularity. We used primed notations to refer to parameters below T_c, unprimed for parameters above. The difference

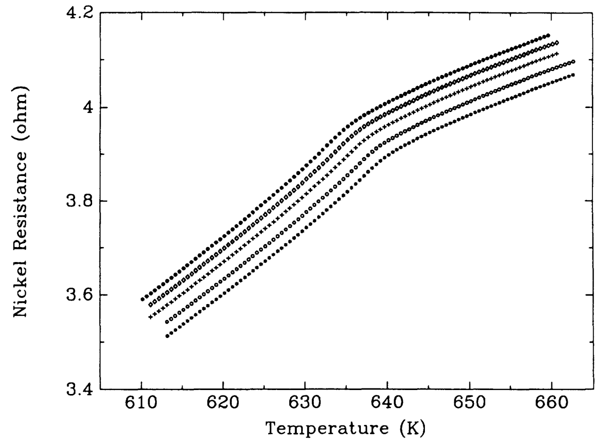

FIG. 2. Nickel resistance vs temperature corresponding to different pressures (dot—25.4 kbar, diamond—30.4 kbar, plus—35.5 kbar, circle—40.5 kbar, asterisk—45.5 kbar).

between the theoretical best fit curve to Eq. (1) and the experimental data for the measurement at 25 kbar is shown in Fig. 3(a). For this least-squares fit the 7 data points nearest T_c were not included in the calculation. The peak near T_c indicates a broadening of the phase transition. The broadening likely is due to a temperature variation along the sample or possibly to inhomogeneities in sample composition. Thus we proceed by using a convolution of the resistance of Eq. (1) and a Gaussian distribution in temperature or homogeneity.

Provided the critical terms in Eq. (1) are good representations of the singular part of the temperature dependence in a completely homogeneous material in a uniform temperature, we can correct for temperature variation over the sample and/or inhomogeneities in the material, leading to different parts of the sample passing through T_c at different times, by using a convolution of the form

$$R^*(T,T_c,\sigma)=\int R(T,T_c-x)g_\sigma(x)dx \quad \text{or} \int R(T+x,T_c)g_\sigma(x)dx\ , \tag{2}$$

where $g_\sigma(x)$ is a Gaussian in x of width σ and x is the temperature variance of each part of the sample from some average T or the difference in T_c from the average T_c of the sample. The variable R in the integrand takes the functional form of Eq. (1). The integral in Eq. (2) was done numerically, using 20-point Hermite integration, and the width of the Gaussian was included as another parameter in the nonlinear least-squares fit. Figure 3(b) shows the variance between the data and the fit to Eq. (2) with no data points excluded. This is over the range $5\times 10^{-4}<|t|<4\times 10^{-2}$.

Because of the high correlation with other parameters one cannot include z as a free variable so it is fixed at a chosen value while the other parameters are varied. Källbäck *et al.* found a good fit with the parameter $z=0.57$, but the minimum is so flat as to be virtually without statistical significance. The value for $z=0.55$ is predicted by the RG theory.[16] The value of 0.55 for z gave the smaller goodness of fit (root mean-square deviation per degree of freedom) for our data but the parameters in the fit with $z=0.57$ do not differ significantly from their values found with $z=0.55$.

The fit with the nine variable parameters R_0, R', α, A, A', D, D', T_c, and σ still displays considerable correlation between the parameters leading to large uncertainties in the results. See Table I. From these results we plotted α as a function of pressure, Fig. 4. It is noted that α does not vary with pressure to within the uncertainty of the measurement. We therefore conclude that α is constant and choose it to have the average of the indi-

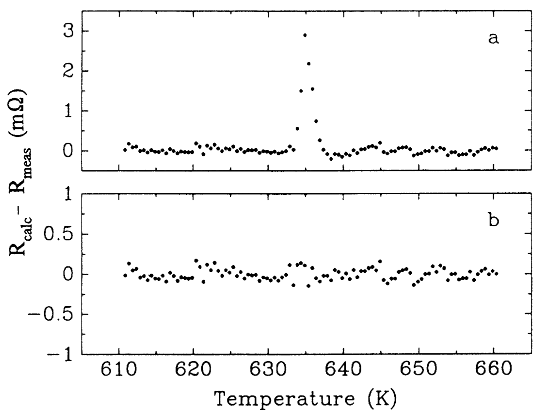

FIG. 3. (a) Residuals for the fit to Eq. (1) with 7 points nearest T_c not used in the fitting and (b) residuals for the fit to Eq. (2) with all data considered. Data points taken from measurement at 25.4 kbar.

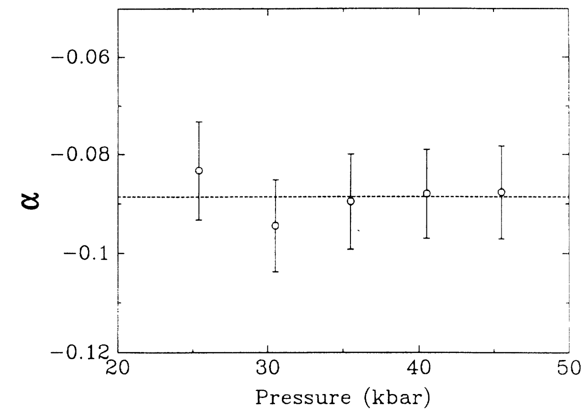

FIG. 4. The critical exponent α vs pressure. One standard deviation shown in data. The dashed line is the average value.

vidual measurements at the various pressures, i.e., $\alpha=-0.089\pm0.002$. The least-square fit of all the data is now repeated with α fixed at this value. With these results we plot the ratio A/A' vs pressure in Fig. 5. The results again are, to within the accuracy of the measurement, independent of pressure. Similarly, A/A' was replaced by an average calculated from these results at different pressures, $A/A'=1.466\pm0.017$ and a final least-squares fit was made to the data with the remaining seven variable parameters. All the results are summarized in Table I. The listed uncertainties represent 1 SD.

The critical temperature T_c versus pressure is a linear function over the range of pressure measured as illustrated in Fig. 6. A linear relation to express the critical temperature versus pressure is given by standard least-square analysis as

$$T_c(P)=630.65(\pm0.10)+0.193(\pm0.003)P\ , \tag{3}$$

where P is pressure in kbar and the critical temperature is in Kelvin. The critical temperature extrapolated to atmospheric pressure is 630.65 K in excellent agreement with 630.28 K reported by Källbäck and Humble.[7] This is better than expected considered the $\pm1.5°$ uncertainty in the thermocouple calibration. The measured slope of T_c versus pressure of 0.193 K/kbar is only half the value

TABLE I. Results of least-squares analysis of resistance versus temperature across the ferromagnetic phase transition at several pressures. Initial results with all the variable parameters free, results with α fixed at the average of the measurements at all pressures in the initial analysis, and final results with the variables α and A/A' fixed at average values over pressure in the initial and second analyses, respectively. See text. rms is the root-mean-square deviation per point.

P (kbar)	T_c(K)	α	A/A'	D/D'	σ(K)	rms ($\mu\Omega$)
			All parameters free			
25.4	635.55±0.03	−0.083±0.010	−1.45±0.06	−1.05±0.27	1.28±0.03	6.92
30.4	636.62±0.02	−0.094±0.009	−1.54±0.06	−0.97±0.19	1.17±0.03	6.66
35.4	637.50±0.02	−0.089±0.010	−1.45±0.05	−1.61±0.39	1.17±0.03	6.34
40.5	638.47±0.02	−0.088±0.009	−1.46±0.05	−1.56±0.32	1.16±0.03	6.11
45.5	639.34±0.02	−0.088±0.009	−1.44±0.05	−2.80±0.80	1.12±0.03	6.03
		α fixed				
25.4	635.54±0.02	−.0887±0.0018	−1.478±0.016	−1.16±0.21	1.26±0.02	6.93
30.4	636.62±0.02	−.0887±0.0018	−1.503±0.016	−0.89±0.13	1.19±0.02	6.68
35.4	637.50±0.02	−.0887±0.0018	−1.454±0.015	−1.62±0.27	1.17±0.02	6.34
40.5	638.47±0.02	−.0887±0.0018	−1.466±0.013	−1.58±0.24	1.15±0.02	6.11
45.5	639.34±0.02	−.0887±0.0018	−1.446±0.013	−2.85±0.60	1.12±0.02	6.04
			α and A/A' fixed			
25.4	635.526±0.020	−.0887±0.0018	−1.466±0.017	−1.36±0.23	1.27±0.02	6.97
30.4	636.564±0.019	−.0887±0.0018	−1.466±0.017	−1.44±0.28	1.20±0.02	7.05
35.4	637.526±0.020	−.0887±0.0018	−1.466±0.017	−1.31±0.21	1.16±0.02	6.38
40.5	638.475±0.018	−.0887±0.0018	−1.466±0.017	−1.55±0.28	1.15±0.02	6.12
45.5	639.379±0.019	−.0887±0.0018	−1.466±0.017	−1.79±0.35	1.11±0.02	6.22

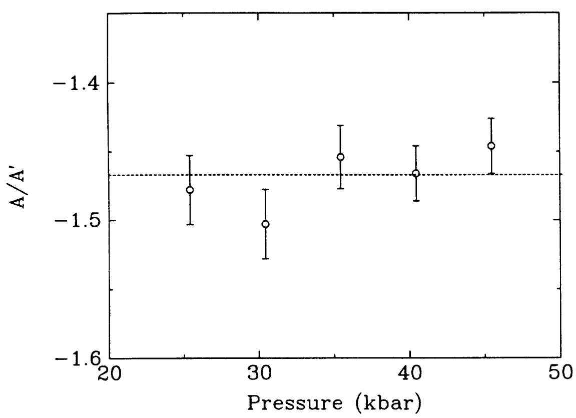

FIG. 5. The amplitude ratio A/A' vs pressure. One standard deviation shown in data. The dashed line is the average value.

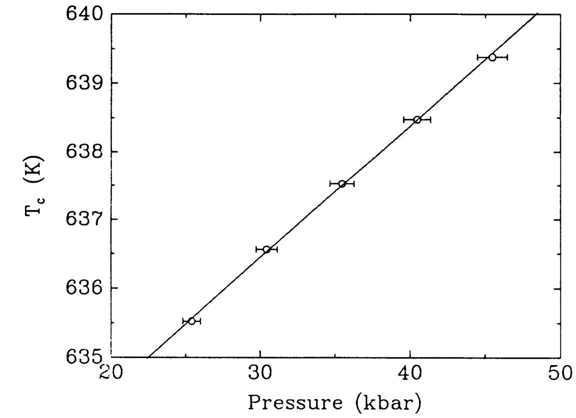

FIG. 6. Pressure dependence of the critical temperature T_c. The linear line is a least-squares fit. Only statistical error bars are shown. No systematic error in T_c is shown in the curve although it is considered in the text and analysis.

reported by Yousuf and Kuman.[9] Specific heat versus pressure measurements by Leger *et al.*[17] have a slope of T_c versus pressure ranging from 0.34 at zero pressure to 0.17 at 50 kbar. There is considerable scatter in the data, T_c at zero pressure was extrapolated to 627 K and the pressure was quasihydrostatic. The reported curvature may thus be suspect. A quadratic fit to our data would extrapolate to 629.63(15) K for T_c at zero pressure with an initial slope of 0.26 kbar K and a slope at 50 kbar of 0.17 kbar/K.

These results confirm the expectation that the values of the critical exponents, and amplitude ratio are invariant along the critical line at least for the leading term. Thus the investigations quantitatively verify the inference of RG theory that a field variable which does not alter the symmetry of the ordered state should not change the universality class to which the system belongs. The magnitude of our results for α and A/A' are in agreement with the reanalysis of Ahlers and Kornblit[18] of the specific-heat measurement by Connelly *et al.*[19] and with the resistance measurement of Källbäck *et al.* at atmospheric pressure. See Table II.

The ratio of the correlation to scaling term D/D' agrees with the results measured by Källbäck *et al.* at zero pressure but appears to increase in magnitude with pressure as shown in Fig. 7. There still exists a small temperature gradient across the sample that gives rise to broadening of the results near the critical temperature T_c and contributes to a large uncertainty in D/D'. It is difficult to compare this term with RG theory because the theory is developed only to a short series second order in $\epsilon=4-d$ where $d=3$ is the dimensionality of the system. The [2,0], [1,1], and [0,2] Padé approximates of the series[20] are -1.77, $+1.13$, and $+0.23$, respectively. With this great discrepancy the theoretical value of D/D' is essentially unknown.[21]

The major reason for the broadening comes from the small temperature variation across the sample. This is difficult to improve further because the internal structure of the pressure cell may distort slightly with changing pressure and temperature thus altering the temperature gradient. This is implied by the change in σ versus pressure shown in Table I. The best attainable temperature control is limited by the precision in the emf measurement of the thermocouple and stray thermal emf's.

The convolution analysis is limited because the actual temperature distribution is probably not accurately

TABLE II. A comparison of results at zero pressure for the different experiments and RG theory. Errors are one mean deviation.

T_c(K)[a]	α	$\lvert A/A'\rvert$	D/D'	Reference
	-0.115 ± 0.009	1.521 ± 0.002	1.13[b]	16,21,20 (RG theory)
631.415 ± 0.010	-0.091 ± 0.002	1.396 ± 0.010		18 (Specific heat meas.)
630.284 ± 0.003	-0.095 ± 0.002	1.51 ± 0.02	-0.8 ± 0.1	7 (Källbäck)
630.284	-0.115 ± 0.005	1.13 ± 0.07	-1.2 ± 0.1	9 (Yousuf)
630.65 ± 0.45[c]	-0.089 ± 0.003	1.48 ± 0.03[d]	-1.2 ± 0.2[d]	This work

[a]Does not include systematic errors in the temperature measurement.

[b]This value is very uncertain. See text.

[c]Most of the error is from extrapolation to zero pressure including possible systematic error in the pressure correction to the thermocouple emf.

[d]Average, weighted by goodness of fit, of extrapolations to zero pressure from fitting to polynomials of zeroth and first order in pressure.

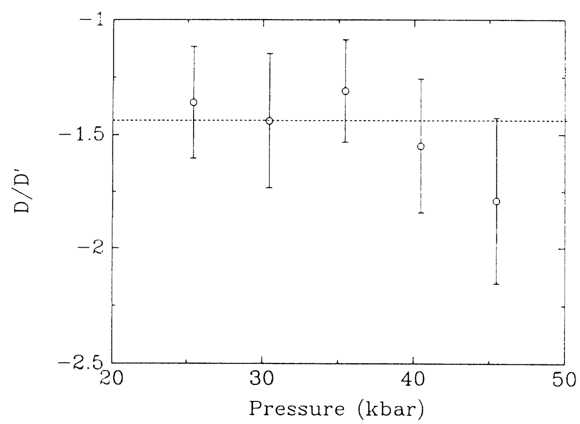

FIG. 7. The amplitude ratio of the confluent singularity vs pressure.

represented by a Gaussian. Considering that the precision of the experimental data in this measurement is much better than the data obtained by Yousuf at high pressure, the results of the data analysis should be more reliable. A comparison of the results obtained from the different experiments and RG theory is shown in Table II.

CONCLUSIONS

In summary, our experimental data and analysis of nickel electrical resistance versus pressure near the ferromagnetic phase transition give the following conclusions. The values of the critical exponents α and amplitude ratio of the leading term A/A' are in good agreement with the results published by Ahlers and Kornblit[17] in a specific-heat measurement and by Källbäck *et al.*[7] in a resistance measurement at atmospheric pressure. The critical exponent is smaller than the RG theory value for a Heisenberg ferromagnet with short-range coupling. The measured values of α and A/A' are independent of pressure which gives experimental evidence of universality. A linear least-squares fit of the data in Fig. 5 for A/A' as a function of pressure however does yield a slight slope of 0.0020(12) kbar^{-1} and a zero pressure intercept of 1.54(4). The uncertainty in the data does not give a significant preference to this interpretation over that of a pressure independence interpretation.

The pressure derivative of the resistance is negative at all pressures and shows no hint of a change at the ferromagnetic transition as argued by Yousuf *et al.*[8] The critical temperature T_c increases linearly with pressure at a rate of 0.193(13) K/kbar including possible systematic error in the pressure correction to the thermocouple calibration. This slope is half of that reported by Yousuf and Kumar[9] and also smaller than the result from the high pressure specific-heat measurement of Leger.[17] The nonhydrostatic pressure environment is probably the problem with Yousuf and Kumar's results and the quasihydrostatic solid medium may have affected Leger's results. Leger extrapolated his measured T_c to 627 K rather than 630 K which led him to postulate a curvature in the critical temperature versus pressure and a larger slope at zero pressure.

To within the accuracy of the data the magnitude of the amplitude ratios could be considered to be independent of pressure. However the data as viewed in Figs. 5 and 7 do not preclude the possibility of A/A' and D/D' slowly changing with pressure. There is a switch in sign of A/A' between the specific heat and resistance measurement but D/D' must have the same sign in both measurements. (Yousuf and Kumar made an error in their argument that this sign would differ between the two types of measurements.) This term has not however been measured in any specific-heat experiment. If these amplitudes do vary with pressure one might ask what implication that would have in terms of universality. It has been shown theoretically that there is a difference in the critical parameters as one considers the short-range interaction versus a long-range dipolar interaction[22] and that the exponents and amplitude ratio for nickel are characteristic of materials with some admixture of dipolar interaction. This is consistent with our experimental results. Ahlers and Kornblit[18] show that α and A/A' are not universal across all Heisenberg ferromagnetic and antiferromagnetic materials. They argue that the observed differences arise from differing amounts of long-range dipolar interaction in comparison to isotropic short-range coupling. It therefore could be argued that small changes in the critical parameters versus pressure might be due to a changing amount of dipolar versus short-range interaction as pressure is increased.

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