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DETAILED ^{57}Fe CONTINUOUS WAVE NMR SPECTRA IN Fe-BASED DILUTE Fe-Co ALLOYS

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ABSTRACT

The hyperfine field distribution around cobalt impurities in iron has been measured by continuous wave nuclear magnetic resonance methods. Four distinct satellites are observable on the high frequency side of the central resonance and an unresolved structure on the low frequency side. The hyperfine field shifts are positive at the four nearest neighbours and probably negative at the fifth and sixth neighbours. The concentration dependence of the average hyperfine field suggests considerable conduction electron polarization contribution, the radial dependence reflects that of the moment perturbation. The temperature dependences of the satellites and main line frequencies follow the temperature dependence of the hyperfine field measured in pure iron.

KIVONAT

A kobalt szennyezések körüli hiperfinom téreloszlást mértük vasban állandó gerjesztésü mag-mágneses rezonancia módszerrel. Négy szatellit figyelhető meg a magas frekvencia felőli oldalon és egy nem elkülönült szerkezet az alacsony frekvenciás oldalon. A hiperfinom tér eltolódás pozitiv az első négy szomszéd helyén és valószinüleg negativ az ötödik és hatodik szomszédnál. Az átlagos hiperfinom tér koncentrációfüggése jelentős vezetési elektron polarizációs járulékra utal, a távolságfüggés a momentumeloszlást tükrözi. A szatellitek és fő-vonal hőmérsékletfüggése megegyezik a tiszta vasban mért hőmérsékletfüggéssel.

РЕЗЮМЕ

Было измерено распределение сверхтонкого поля вокруг примесей кобальта в железной матрице методом ядерного магнитного резонанса с постоянным возбуждением. Наблюдаются четыре дискретных сателлитов со стороны высокой частоты, а неразобщенная структура со стороны нижней частоты. Сдвиг сверхтонкого поля - положителен на месте первых четырёх соседов и наверное отрицателен при пятом и шестом соседях. Зависимость среднего сверхтонкого поля от концентрации позволяет сделать вывод, что поляризационный вклад от электронов проводимости является значительным, а зависимость от расстояния показывает распределение моментов. Температурные зависимости сателлитов и частот основных линий соответствуют температурной зависимости, измереннной в чистом железе.

1. INTRODUCTION

The electronic structure of impurities in ferromagnetic hosts has been the field of considerable activity in recent years. In spite of numerous theoretical and experimental work our understanding is far from being complete. The problem is complicated by the fact that the Fermi surface of the 3d- transition elements intersects both the narrow 3d-like band and the broad 4s-like band of electrons, and at present one can not tell from first principles the nature of electrons taking part in the screening of the excess potential of the impurity. On the other hand, both the 3d-and 4s-like bands contribute to the parameters measured in different experiments, and it is often difficult to separate the two contributions and the different behaviour they reflect.

Various nuclear methods, such as diffuse neutron scattering /DNS/ /Collins and Low, 1963/ Mössbauer effect /ME/ measurements /Steams, 1966/ Wertheim et.al 1964/ and different types of nuclear magnetic resonance /NMR/ /Mendis, 1967; Budnick et.al. 1970/ have been put forward to investigate both the perturbation on the impurity site and the radial dependence of the perturbation around impurities. By comparing the results of these methods, valuable information can be obtained on the nature of the perturbations: with DNS one measures the magnetic moment distribution, e.g. the perturbation of the 3d-like band, while the hyperfine field distribution - measured by ME and NMR methods - reflects the perturbation of both the 3d- and 4s-like bands. On the basis of such experiments recent publications /Campbell, 1969; Grüner G., 1968/ have led to the qualitative picture that in the case of non-transitional impurities the perturbation of the 4s-like band is dominant, whereas in the case of transitional impurities the impurity potential is screened by 3d-like electrons.

The iron-based dilute Fe-Co system is one of the most studied dilute transitional metal alloys. /For an extensive review of the ME and NMR results on Fe-Co alloys see Rubinstein, 1968./ But in spite of widespread investigation the detailed distribution of the hyperfine field around the cobalt impurities is still not clear. The origin of the disagreement between the different authors is in the assignment of the measured hyperfine field shifts to the different neighbour shells around

the impurities. Aside from reporting various satellites on the high frequency side of the nearly unshifted central resonance line, recent publications agree on the existence of a prominent satellite shifted by $\Delta H/H = 1,3\%$. Mendis and Anderson /1967/ and Budnick et. al. /1970/ attribute this line to the contribution of the third neighbours of the Co impurity, while on the basis of the splitting he observed in the ^{59}Co resonance spectra Rubinstein /1968/ suggests that it arises from a collapsing contribution of the first and second neighbours. On the basis of this assignment and the similarity of the ^{57}Fe and ^{59}Co resonance spectra, he also suggests that the contribution of the conduction electron polarization term to the measured hyperfine field distribution is negligible, and thus the radial distribution is determined mainly by the momentum perturbation around the Co impurities. In contrast the assignment of Mendis and Anderson /1967/ should yield a considerable conduction electron polarization change due to the introduction of Co atoms into the iron matrix.

The aim of this paper is to present detailed ^{57}Fe hyperfine field spectra measured in the 100-300°K temperature range by continuous wave nuclear magnetic resonance /cwNMR/ method and to discuss the results of the different NMR and ME measurements on the basis of our investigations. Taking the DNS data of Collins and Low /1963/ and the data on the concentration dependence of the average magnetic moment /Weiss and Forrer, 1929/, the contribution of the different components of the internal magnetic field to the measured hyperfine field distribution can be obtained.

2. EXPERIMENTAL TECHNIQUE, RESULTS

The ^{57}Fe resonance was measured with a frequency-swept, frequency-modulated nuclear magnetic resonance absorption circuit described in /Tóth F et.al to be published/, using automatic frequency control. Adiabatic fast passage /Cowan and Anderson, 1965/ with a modulation frequency $f_m = 280$ Hz was used to plot the hyperfine field distribution. Though all the details of the adiabatic fast passage could not be observed, due to some residual 0° signal, and the line width of pure iron was slightly dependent of the modulation amplitude, the Lorentzian shape of the resonance signal in pure iron showed that the resonance line measured on the alloy represents the distribution of the hyperfine field. For the measurements at low temperature the temperature control system described in /Balla et.al, 1968/ was used. Fe-Co samples were prepared

from 99.99% purity Fe and high purity Co in an induction vacuum furnace. The ingots were ground down to pass through a 54 μ sieve. The resulting powder was mixed with MgO and annealed at 700°C for 1 h to remove the lattice defects produced by grinding. The cobalt content of the alloys was determined with nitroso-Rm salt by spectrophotometric method.

In Fig. 1 we show the room-temperature cwNMR spectra for Fe-Co alloys of several concentrations. Aside from the marked broadening of the spectra with increasing concentration, several satellites are to be seen on the high frequency side of the central resonance, which is shifted slightly to lower frequencies. In the Fe-0,72 at% Co alloy four satellites, shifted by $\Delta H / H$ = 2,5%; 1,5%; 1,3% and 0,18% are observable which we designate 1 to 4 respectively. At the lowest concentration satellite 4 is nearly resolved from the central resonance, while at the largest concentration this satellite collapses with it, due to the considerable broadening of the components. The most pronounced satellite 3 is well resolved at all concentrations. We have observed a long unresolved tail on the low frequency side of the central resonance extending to about 45,2 Mc/s.

Fig. 3 shows the spectra of the Fe-0,72 at% Co and Fe-1,41 at% Co alloys at 100°K. A marked broadening, and the disappearance of the satellites 2 and 4 are the most pronounced differences from the room-temperature spectrum. In Fig. 3 the temperature dependence of the resonance frequencies of the different satellites /measured on the Fe-0,72 at% Co alloy/ and the line width measured at half the amplitude are presented together with the spin echo /SE/ results of Budnick et.al /1970/ taken at 1,35°K.

The connection between the latter results and ours will be discussed presently.

3. HYPERFINE FIELD DISTRIBUTION

Each impurity in the bcc iron lattice is surrounded by eight nearest neighbours, six next-nearest neighbours, and 12, 24, 8 and 6 third, fourth, fifth and sixth neighbours. The relative intensity of a satellite due to the given neighbours of the impurities is given by $nc/1-c/^{-1}$, where n the number of sites at the given shell around the impurity, c the impurity concentration. The method of assigning the different satellites observed in the hyperfine field distribution is to measure the intensities of these satellites and compare them with the

intensities calculated for the given concentration and shells. /Corrections of the form $(\omega/\omega_o)^3$ /Furley and Dean, 1968/ can be neglected in view of the small relative hyperfine field shifts./ Instead of the integrated intensity, the amplitude is often used to assign the satellites /Rubinstein, 1968/ but this procedure is established only in the case of satellites whose width is equal to the central resonance. In the following we consider the satellites separately in order to assign them to the different neighbour shells.

Satellite 4, at 45,46 Mc/s, is not quite resolved from the central resonance even in the lowest concentration alloy studied here. The resonance signal, however, can be graphically decomposed into two components of equal width with a relative amplitude $D^4_s/D_{CR} = 0{,}075 \pm 0{,}005$. This amplitude ratio corresponds to the fourth neighbours. The computed relative intensity for this concentration is 0,072. At higher concentrations this satellite is broadened more than that of the central resonance, because of the contributions coming from two, three or more Co atoms in the fourth neighbour shell, which give a relative hyperfine field shift about twice that caused by a single impurity.

Satellite 3, at 45,97 Mc/s, has a width similar to that of the central resonance, except in the spectrum at the Fe-1,41 at% Co alloys, where it collapses with satellite 4, which makes its width difficult to determine. The amplitude of this satellite indicates that it arises from the third neighbours. For example, in the Fe-0,72 at% Co alloy the measured value of D^3_s/C_{CR} is $0{,}1 \pm 0{,}01$; for this concentration the calculated value is 0,086.

Satellite 2, at 46,05 Mc/s, has a shift $\Delta H_2 = \Delta H_3 + \Delta H_4$ indicating that it arises from Fe nuclei having a Co neighbour in the third and one in the fourth neighbour shell. The intensity relative to the central resonance is $D^2_s/D_{CR} \sim 0{,}02$ in the case of the Fe-0,72 at% Co alloy; the above supposition yields $n_3 n_4 c^2 (1-c)^{-2} = 0{,}014$, in good agreement with the measured value.

Satellite 1, at 45,6 Mc/s, is much broader than the central resonance and any of the other satellites. Its shift is about twice that of satellite 3, so in principle it could be the contribution of Fe nuclei having two Co neighbours in the third neighbour shell. The calculated intensity corresponding to this supposition is $D^1_s/D_{CR} = 0{,}01$ for the largest concentration alloy which is much smaller than the estimated of $0{,}06 \pm 0{,}02$. The large width of this satellite suggests that it corresponds to iron nuclei close to the Co impurities, and to experiencing

large anisotropic interactions. On the basis of reported ME measurements /Wertheim and Jaccarino et.al, 1964; Grüner and Vincze, 1971; Wertheim, 1971/, we suggest that it corresponds to the second neighbours of the Co impurities. From the amplitude of this satellite we estimate a width of about 300 kc/s for the highest concentration alloy.

The assignments of the above satellites and the satellites observed by other authors are summarized in Table 1 together with the results of the decompositions of Mössbauer spectra /Wertheim et.al, 1964; Grüner and Vincze, 1971, Wertheim, 1971/. It can be seen that as regards the appearance of the satellites our spectra are in agreement with the earlier cwNMR measurements of Mendis and Anderson /1967/, the SE measurements of Budnick et.al /1970/ and Rubinstein /1968/, and with the results of the ME measurements.

The assignments of the spectra as regards satellites 4 and 3 support the interpretation of Mendis and Anderson /1967/ and are in agreement with the measurements of Budnick et.al. /1970/, taking into account that due to the spectrum broadening at low temperatures satellite 4 is not observable. The splitting found by Rubinstein /1968/ finds its natural explanation as the contribution of the 3N and 3N+4N neighbours. Satellite 1 observed by all the above authors we interpret, on the basis of the great width of this satellite, as the contribution of the next-nearest neighbours, instead of as the contributions of two third neighbours.

This assignment is supported by the ME experiments, where by decomposition the shifts due to the first and second neighbours can be obtained. The second column of Table 1 also shows the relative hyperfine field shifts due to the firts neighbours, as determined by ME; these seem to correspond to the other satellite observed by Budnick et.al /1970/.

In concluding this analysis, we suggest that the hyperfine field around Co impurities, as measured by a number of authors with different methods of different resolution, can be interpreted as a positive polarization near to the impurities which decreases the further away from the impurities one goes yielding the relative hyperfine field shifts 4% at the first, 2,5% at the second, 1,3% at the third and 0,18 at the fourth neighbours of the impurities, while at distances of about 6 Å the perturbation is negative. The broadening of the main line suggests an oscillatory hyperfine field distribution at larger distances from the Co impurities.

Fig. 5 shows the hyperfine field distribution based on the present analysis. From the measured hyperfine field shifts we can determine the concentration dependence of the average hyperfine field. At low impurity concentrations $\sum n_i \Delta H_i = -200 \pm 10$ kG is obtained. From the shifting of the central resonance to lower frequencies we estimate an additive contribution of $+15 \pm 5$ kG, yielding the concentration dependence of the average hyperfine field $\frac{\partial H_{av}}{\partial c} = 185 \pm 15$ kG, in good agreement with the value of -180 kG determined by ME experiments /Grüner and Vincze, 1971/.

4. DISCUSSION

The hyperfine field in ferromagnetic metals has two main contributions: the core polarization term H_{CP} due to the moment localized on the atomic site and interacting with the inner s shells; and the conduction electron polarization term H_{CEP} due to the polarization of 4s-like electrons by 3d-like electrons.

The radial distribution of the hyperfine field can be expressed /Rubinstein, 1968/ as

$$\Delta H(r) = a\, \Delta\mu(r) + b\Delta\sigma(r) \qquad /1/$$

where $\Delta\mu$ is the change of moment of the matrix atoms, and σ is the 4s-band polarization. The first term arises from the momentum change in the neighbourhood of the impurity, the second from the change in the polarization of the 4s-like band. In the case of transition impurities in iron the screening of the excess charge of the impurity is performed by the 3d-like electrons in view of their large density of states, and the concentration dependence of the average moment, as well as the moment localized on the impurity site can be well described in terms of rather simplified models /Campbell, 1969/. However, no attempt has been made yet to describe this screening problem where 3d- and 4s-like bands are present and to determine the radial distribution of the hyperfine field. Using the DNS data of Collins and Low /1963/ and the average magnetization data /Weiss and Forrer, 1929/, it is possible, however, to determine the concentration dependence of the average hyperfine field in the framework of the Campbell-Daniel-Friedel /Campbell, 1969; Daniel and Friedel, 1963/ /CDF/ model. In this model the change of the average magnetic field has two contributions. One coming from the average momentum change and can

be expressed as

$$\frac{\partial H_{CP}}{\partial C} = h_{CP}\left[\frac{d\mu}{dc} - (\mu_{Co} - \mu_{Fe})\right] \qquad /2/$$

where $h_{CP} = 50$ kOe/μ_B is the core polarization hyperfine field constant, $d\mu/dc$ the concentration dependence of the magnetic moments of the Co and Fe atoms in iron. Assuming that the average polarization of the 4s-like band is proportional to the average moment, the CDF theory yields for the conduction electron polarization /Campbell, 1969/

$$\frac{\partial H_{CEP}}{\partial C} = h_{CEP}\left[\frac{d\mu}{dc} - 0.4(\mu_{Co} - \mu_{Fe})\right] \qquad /3/$$

where the conduction electron polarization hyperfine field constant $h_{CEP} = -100$ kOe/μ_B. The moment localized on the impurity site can be determined from the DNS data /Collins and Low, 1963/ and from the hyperfine field measured at the Co impurity /Shirley et.al 1968/. The former gives a value $\mu_{Co} = 1{,}9\ \mu_B$, the latter $\mu_{Co} = 1{,}7\mu_B$

Taking the value $\mu_{Co} = 1{,}8\ \mu_B$ and the average magnetization $\frac{d\mu}{dc} = +1{,}1\ _B$ /Weiss and Forrer, 1929/ measured at low Co concentration we get from eq. /2/ the average hyperfine field change contribution due to the momentum perturbation $\frac{\partial H_{CP}}{\partial c} = -70$ kG, while /3/ yields a conduction electron polarization term of $\frac{\partial H_{CEP}}{\partial c} = -120$ kG. The joint contribution of the two terms of -190 kG agrees well with the experimental value -185 ± 15 kG. This analysis leads to the conclusion that conduction electron polarization plays an important role in the average hyperfine field even in the case of a 3d-transition metal impurity in iron. The good agreement seems to be fortuitous, owing to the drastic approximations of the CDF model, and other authors suggest a somewhat different hyperfine field coupling contants /Shirley et.al, 1968; Stearns, 1971/, but large CEP contribution is obtained by using other reasonable H_{CP} and H_{CEP} parameters. Having established the role of the two contributions, we can ask whether the radial dependence of the conduction electron polarization is different from the radial dependence of the momentum change, or whether the CEP term is mainly determined by the momentum localized on the same matrix atom. In principle this can be done by comparing the measured radial dependence with that of the momentum change. Unfortunately, due to

the poor resolution of the neutron scattering experiments only a few qualitative remarks can be given. Fig. 4. shows the radial distribution of the hyperfine field and momentum change around the Co impurities. Due to the low resolution, Collins and Low /1963/ were able to determine the general behaviour of the momentum perturbation around transition impurities to the right of iron, which show a very similar structure. We have normalized this radial dependence /shown in Fig. 6 of their paper/ to get the concentration dependence of the matrix magnetization $d\mu/dc - (\mu_{Co} - \mu_{Fe}) = + 1.5\mu_B$. The similar behaviour of the two perturbations suggests that the moment localized on the iron atom in question is mainly responsible for the conduction electron polarization. There are, however, some essential differences between the two perturbations: the hyperfine field distribution is negative around 6 Å, in contrast to the moment perturbation, which is definitely positive over the whole range. This difference shows that no expression of the form $\Delta H = a\Delta\mu + b \sum_i n_i \Delta\mu_i$ /where the sum is over the neighbours/ can possibly fit the data with negative values of a and b, as stated by Bernas and Campbell /1966/ and by Rubinstein /1968/. We mention, however, that the RPA solution of the screening of the impurity potential in a polarized electron gas /Kim et.al, 1970/ reproduces this behaviour of the hyperfine field surprisingly well.

The cwNMR measurements of Mendis and Anderson /1967/ and the preliminary ME results of Vincze /1971/ show that the same situation arises in the case of Fe-Ni alloys. The hyperfine field shifts are positive at the first four coordination shells, while at the fifth the shift is negative. The temperature dependences of the satellites and central resonance frequencies show that the magnitude of the hyperfine field perturbation follows the average magnetization. A strong departure from this behaviour is expected only where the temperature dependence of the impurity hyperfine field is anomalous. /A notable example is Fe-Mn /Koi et.al 1964; Jaccarino et.al, 1964/./ No anomalous temperature dependence was found in the Co hyperfine field by Koi /1962/, in accordance with the present measurements.

5. CONCLUSIONS

By performing detailed cwNMR investigations of Fe-Co alloys and examining the results of other authors we have determined the radial distribution of the hyperfine field around Co impurities. We have dis-

cussed the average hyperfine field in the framework of the Campbell-Daniel-Friedel theory and demonstrated the role of the conduction electron polarization. The good agreement between the radial dependences of the hyperfine field and momentum change leads to the conclusion that the CEP term is mainly determined by the moment localised on the same atom. The temperature dependences of the satellite frequencies follow the temperature dependence of the central resonance.

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Table 1

Positions of satellite lines and corresponding configurations of atoms around a cobalt impurity

ASSIGNMENT	-	1	2	3	4	5	6
Neighbours Configuration	1N	2N	3N+4N	3N	4N	5N	6N
Budnick et al [1]	4%	2,5%	-	1,3%	-	-	-
Rubinstein [2]	4,2%	2,8%	1,5%	1,3%	0,25%		
Mendis, Anderson [3]	-	2,5%	-	1,2%	0,19%	-	-
Wertheim et al [4]	4,3%	2,1%	-	-	-	-	-
Grüner, Vincze [5]	4%	2,1%	-	-	-	< 0	<0
Wertheim et al [6]	3,5%	2,5%	-	-	-	-	-
Present results	-	2,5%	1,5%	1,3%	0,18%	-	-

[1] Budnick J.I. et al. /1970/ Phys.Rev.Lett. 24, 511-4
[2] Rubinstein M. /1968/ Phys.Rev. 172, 277-83
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[4] Wertheim G.K. et.al /1964/ Phys.Rev.Lett. 12, 24-7
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FIGURE CAPTIONS

Fig. 1. ^{57}Fe cwNMR spectra of Fe-Co alloys at room temperature.

Fig. 2. ^{57}cwNMR spectra of Fe-0,72 at% Co and Fe-1,41 at% Co alloys at T = 100° K

Fig. 3. Temperature dependence of satellite and central resonance frequencies. The inset shows the temperature dependence of the line width.

Fig. 4. Radial dependence of the hyperfine field around Co impurities in iron.

Fig. 5. Momentum and hyperfine field distribution around Co impurities in iron.

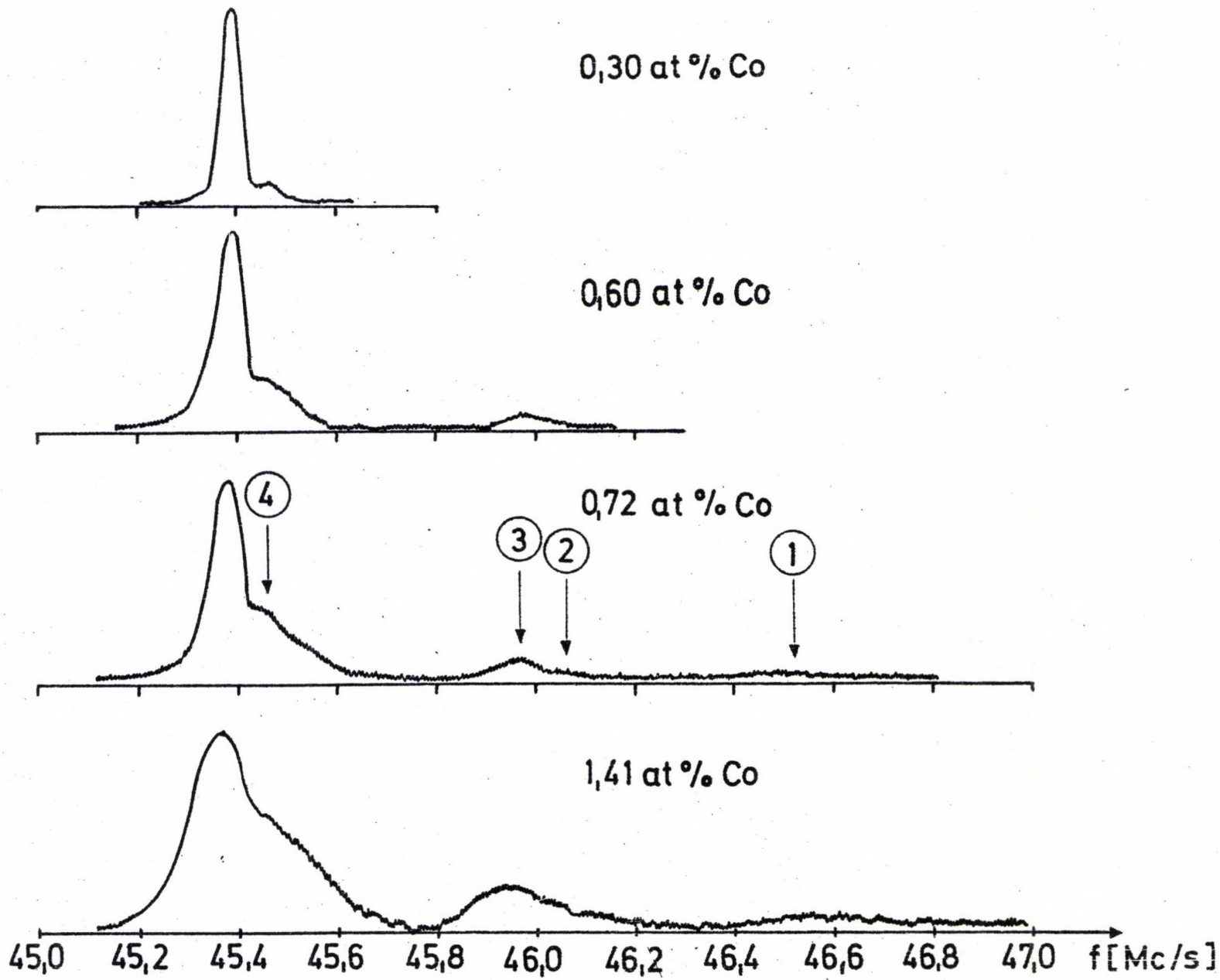

0,30 at % Co
0,60 at % Co
0,72 at % Co
4
3
2
1
1,41 at % Co
45,0
45,2
45,4
45,6
45,8
46,0
46,2
46,4
46,6
46,8
47,0
f[Mc/s]

Fig. 1

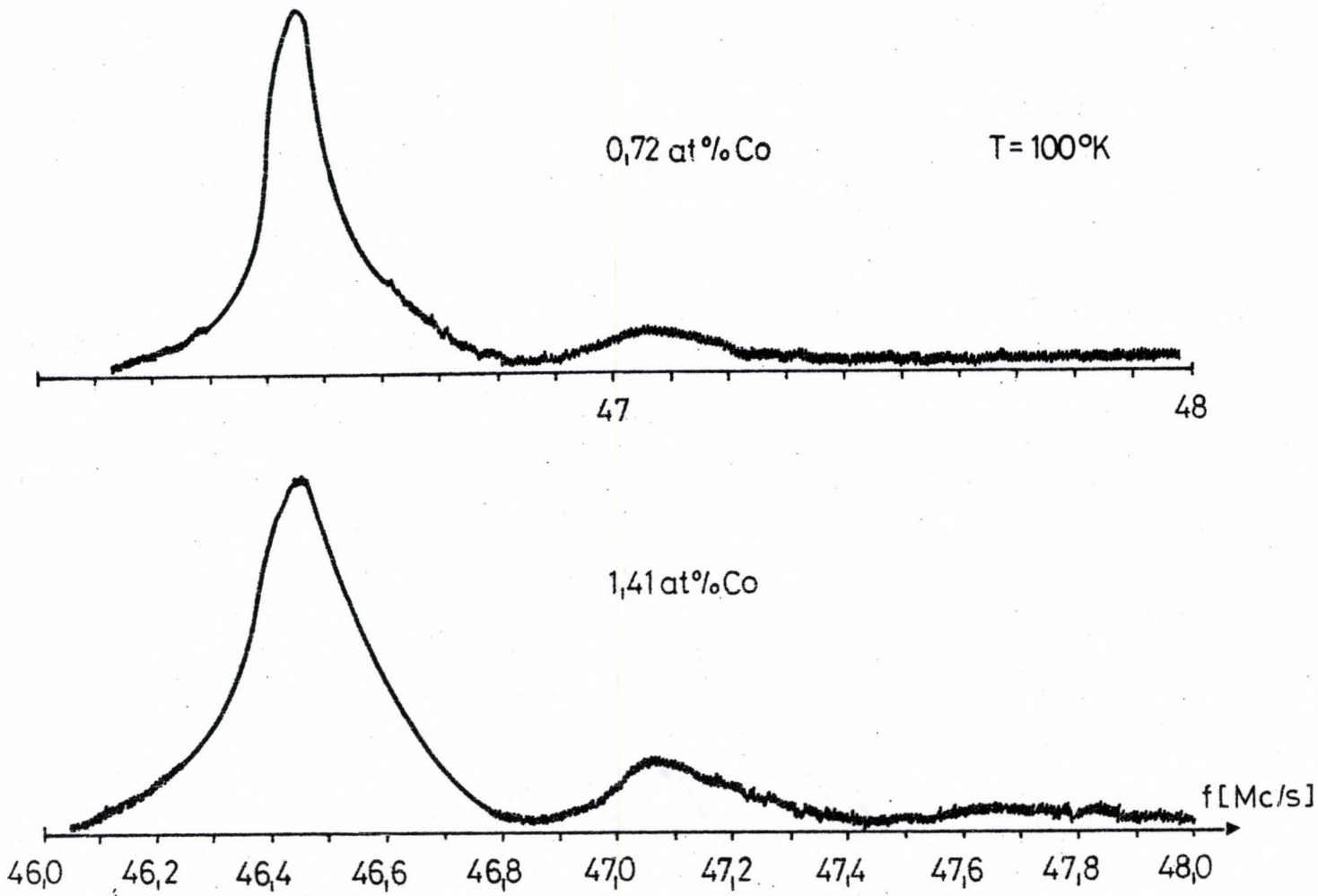

0,72 at% Co
T = 100°K
47
48
1,41 at% Co
f [Mc/s]
46,0
46,2
46,4
46,6
46,8
47,0
47,2
47,4
47,6
47,8
48,0

Fig. 2

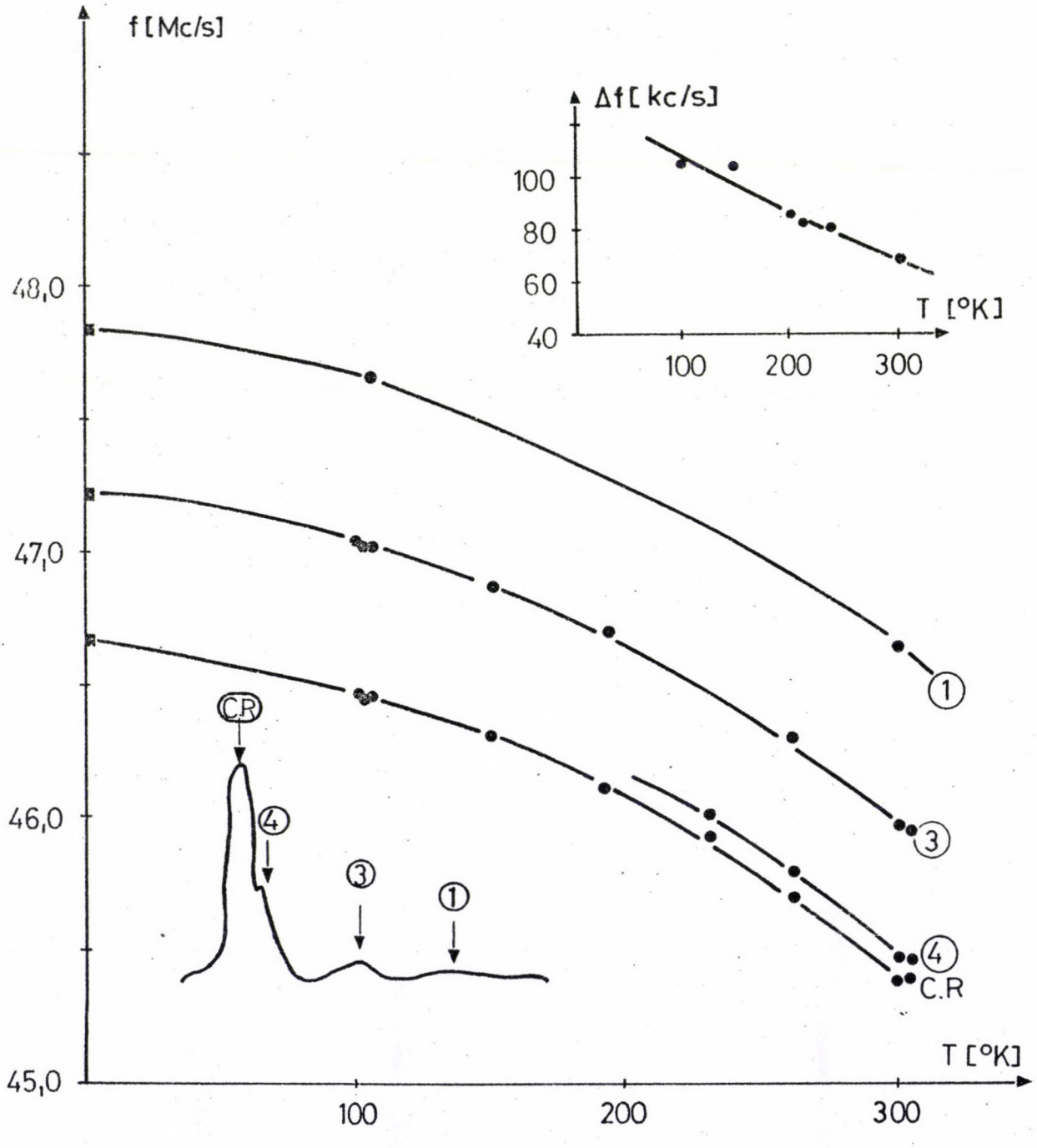

f [Mc/s]
48,0
47,0
46,0
45,0
100
200
300
T [°K]
Δf [kc/s]
100
80
60
40
100
200
300
T [°K]
C.R
④
③
①
①
③
④
C.R

Fig. 3

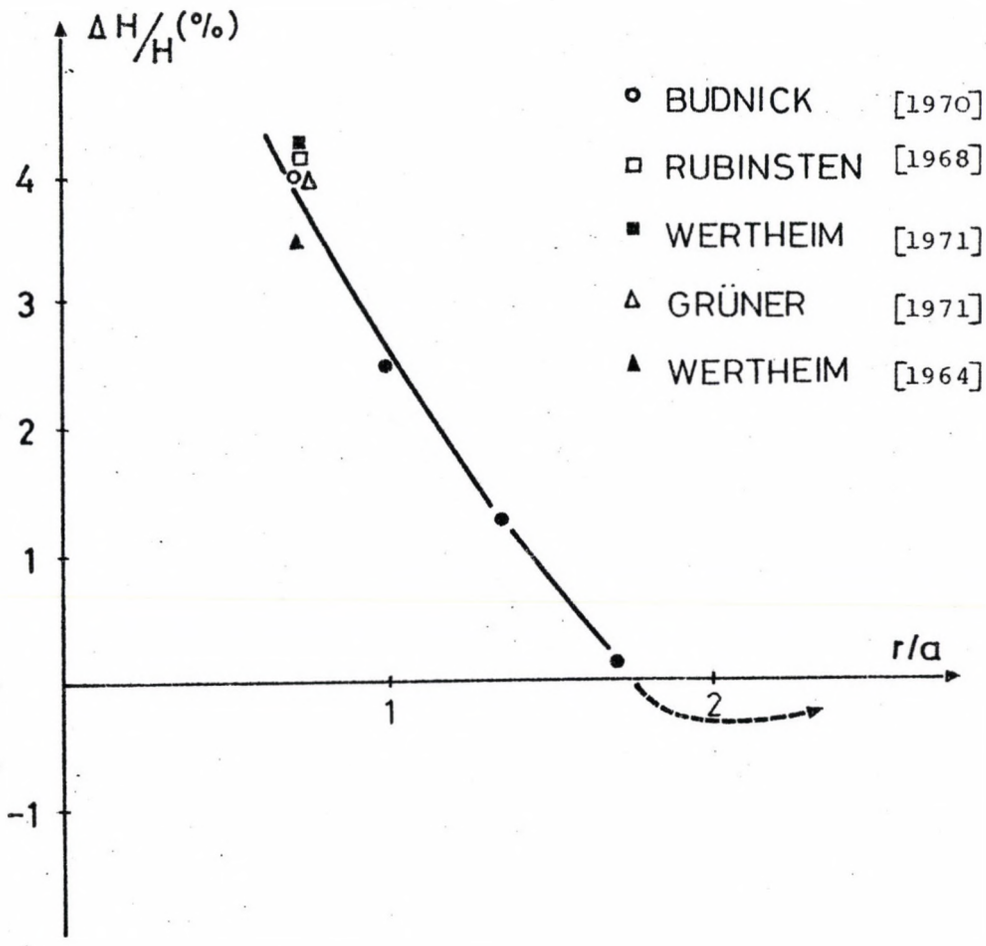

ΔH/H (%)
4
3
2
1
-1
r/a
1
2
BUDNICK [1970]
RUBINSTEN [1968]
WERTHEIM [1971]
GRÜNER [1971]
WERTHEIM [1964]

Fig. 4

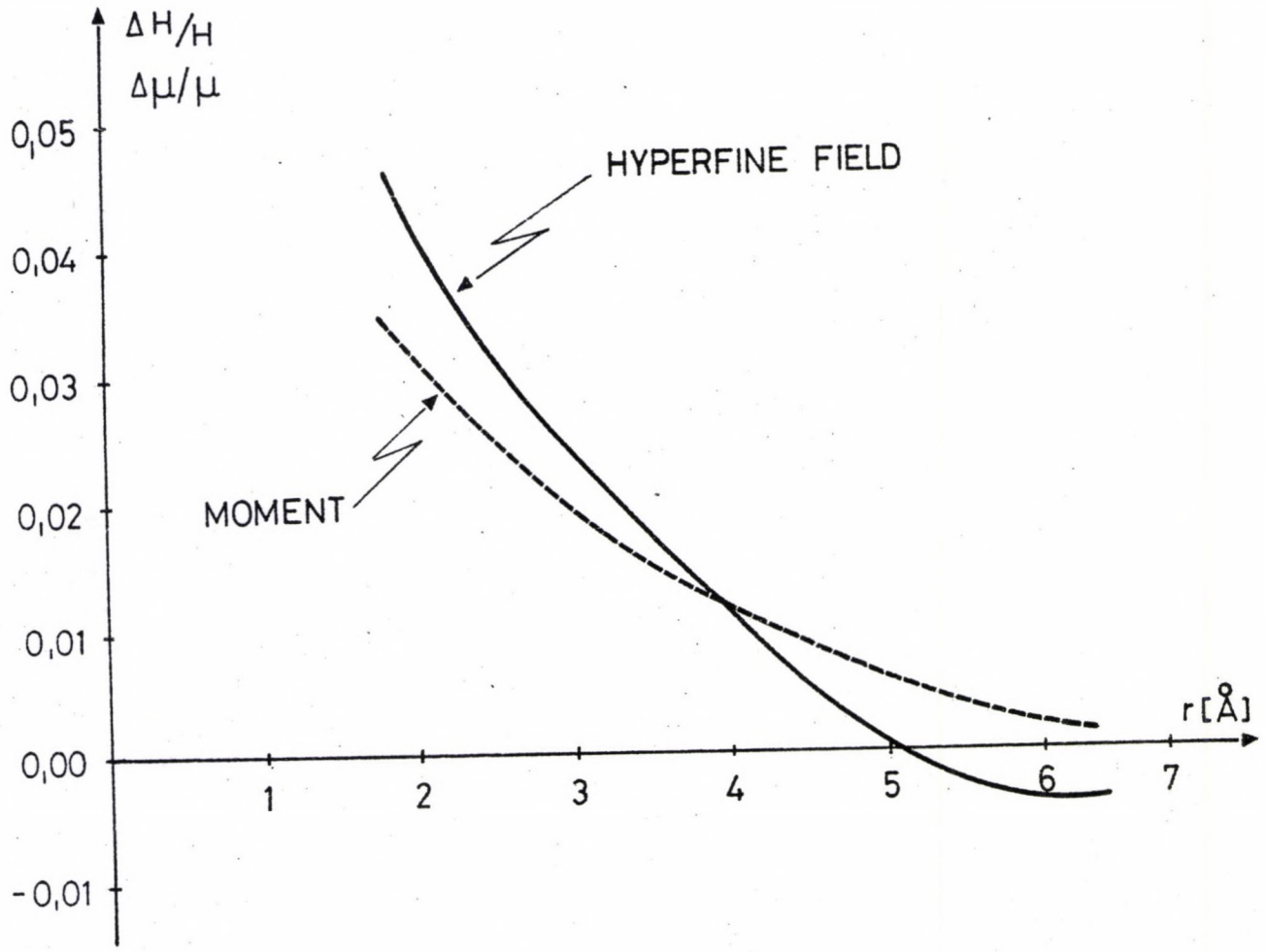
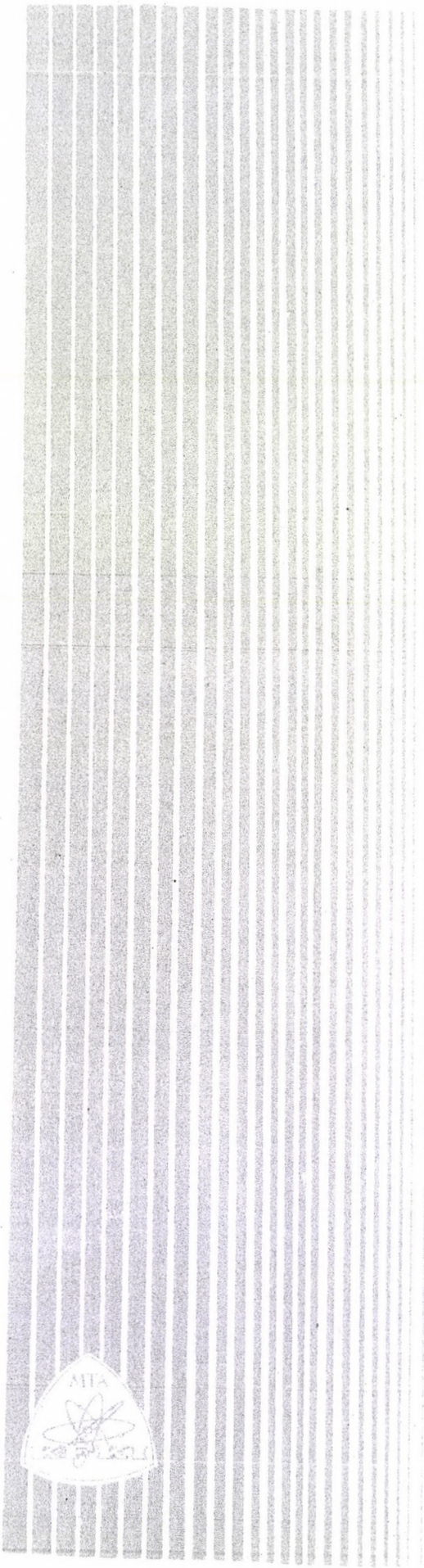

ΔH/H
Δμ/μ
0,05
0,04
0,03
0,02
0,01
0,00
-0,01
HYPERFINE FIELD
MOMENT
r[Å]
1
2
3
4
5
6
7

Fig. 5



Kiadja a Központi Fizikai Kutató Intézet
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