Acta Cryst. (1961). **14**, 823

A Neutron-Diffraction Study of Very Pure Chromium*

BY G. E. BACON

Atomic Energy Research Establishment, Harwell, England

(*Received* 30 *October* 1960)

Single crystal and polycrystalline samples of pure chromium (0·01% oxygen, 0·001% nitrogen, 0·0002% metallic impurities by weight) lose the major portion of their antiferromagnetic ordering at 40 °C. but there remains some residual order in polycrystalline material and this increases with reduction of grain size. In all cases it is concluded that the magnetic moment on the chromium atom, well below the Néel temperature, is about $0{\cdot}45\mu_B$. With addition of 0·5% of iron and 0·16% of nickel the Néel temperature is lowered to 26 °C.

Introduction

Chromium was first studied by neutron diffraction by Shull & Wilkinson (1953) who observed a weak 100 magnetic reflection which disappeared on heating to 200 °C. They deduced an antiferromagnetic structure according to which the chromium atoms had a magnetic moment of $0{\cdot}40\mu_B$ and the moments of atoms at the corners and body-centre of the cubic unit cell were arranged antiparallel. It was realized that the apparent Néel temperature of 200 °C. was quite different from the position of the anomaly at 40 °C. in the electrical resistivity and other physical properties (see, for example, Powell & Tye, 1956). This early neutron-diffraction study was done with powdered samples.

Some years later single crystals were studied at the Brookhaven National Laboratory and this work was reported briefly by Corliss, Hastings & Weiss (1959). Not only did the single crystals show a Néel temperature at the much more acceptable value of 38 °C. but they also displayed satellite reflections in the diffraction pattern, which could be explained by assuming the existence of an antiphase domain structure, or, alternatively, an arrangement of spiral spins such as has been reported to occur in $MnAu_2$ (Herpin, Meriel & Villain, 1959). When explanations were sought for the different behaviour of powdered and single crystal material it was suggested that sample purity was particularly important, not only in the neutron-diffraction measurements but also for the measurements of other physical properties. Apart from the possible influence of metallic impurities, it is very difficult to produce chromium free from nitrogen, oxygen and hydrogen and it was pointed out, for example, that Cr_2O_3 is antiferromagnetic and has a Néel temperature not far from 40 °C. Accordingly, when we were able to obtain some samples of chromium of very high purity from the Aeronautical Research Laboratories, Melbourne, Australia, we took the opportunity to make independent measurements with both single crystals and polycrystalline samples. In these materials the metallic impurities are about 2 parts in 10^6 and the analyzed proportion of nitrogen was not greater than 0·001% by weight. The oxygen content ranged between 0·003 and 0·01% by weight in different samples.

Experimental data on Néel temperature

The single crystal which was examined first was cylindrical in shape, with a length of about 3 cm. and a diameter of 0·4 cm. and an oxygen content less than 0·003% by weight. The cylinder axis was very close to the [013] crystallographic axis, which was aligned vertically on the neutron spectrometer to permit examination of the $\{h00\}$ reflections in the equatorial plane. In order to allow measurements at various temperatures, the crystal, on its supporting goniometer, was mounted in a double-walled Dewar vessel, the interior of which could be filled with liquid nitrogen, or solid carbon dioxide, or could carry a heating coil. A very thin copper tube slightly larger in diameter than the crystal surrounded the latter, to act as a radiation shield, and a thermocouple was attached to each end of the crystal. The diffraction patterns were examined at the Harwell reactor DIDO, using neutrons of wavelength 1·04 Å, with the large spectrometer described by Bacon & Dyer (1959). The peak intensity of the 200 reflection, which was used for aligning the crystal, was about 100,000 neutrons per minute.

In order to examine the antiferromagnetic properties of the sample, the 100 reflection was studied. The form of this, as indicated by spectra obtained with counter and crystal rotating with angular velocities in the ratio 2:1, is illustrated in Fig. 1 which shows the experimental curves obtained at a variety of temperatures. Between about −120 °C. and 40 °C. the 100 reflection is 'split', as expected from the antiphase domain structure postulated by Corliss, Hastings & Weiss (1959), giving two reflections which at room

---

* Some of the results described in this paper were reported at the Neutron Diffraction Conference held at Gatlinburg, U.S.A., April 1960, and at the Cambridge meeting of the International Union of Crystallography, August 1960.

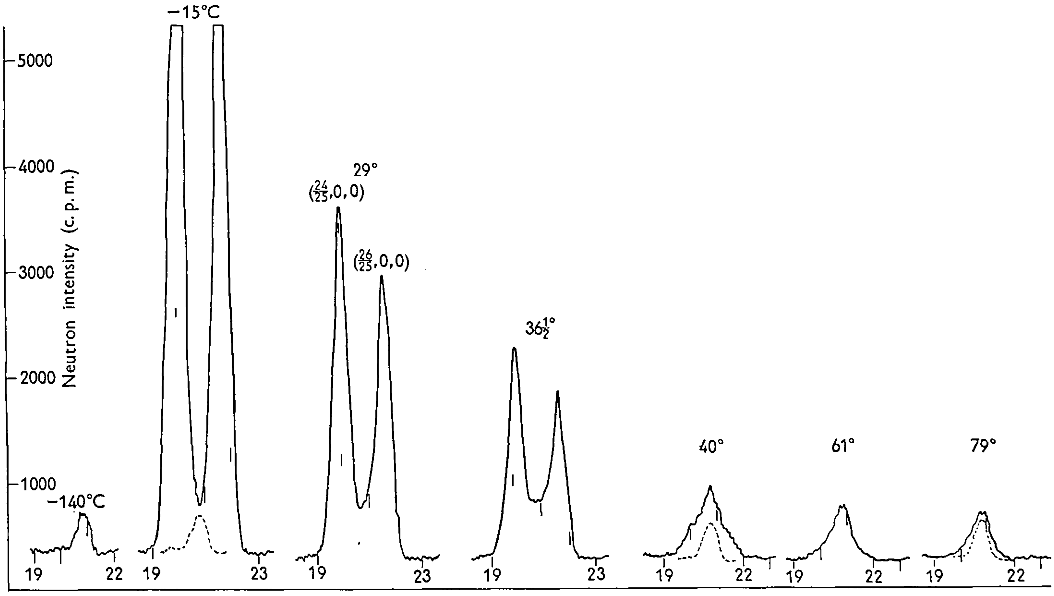

Fig. 1. The shape of the (100) reflection at various temperatures for a large single crystal, with counter and crystal moving in synchronism after alignment on the (200) nuclear reflection.

temperature index approximately as $\frac{24}{25}$,0,0 and $\frac{26}{25}$,0,0. We shall discuss the form and position of these reflections in more detail later. For the present we simply draw attention to the dependence of their intensity on temperature which Fig. 1 indicates. The two halves of the reflection are almost completely resolved, with a slight overlap at the centre with the $\lambda/2$ component of the 200 reflection. This second-order component has an intensity which is only about $\frac{1}{3}$% of the first-order reflection but this amounts to about 350 neutrons per minute for the very intense 200 reflection.

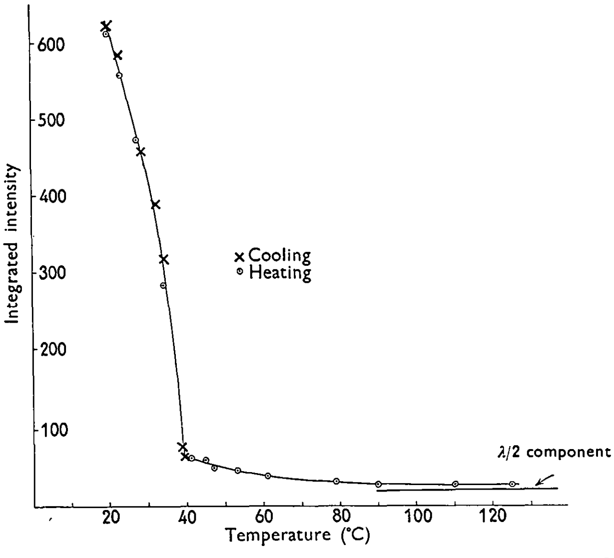

Fig. 2. The variation with temperature of the integrated intensity of the (100) reflection of a single crystal of pure chromium, near 40 °C. Points taken during cooling and heating, respectively, show no thermal hysteresis. The saturation value of the intensity, at low temperatures, is 1600 on the arbitrary scale used.

A plot of the variation of integrated intensity with temperature from 20 °C. upwards is shown in Fig. 2. At lower temperatures, beyond the range covered by this figure, the integrated intensity rises to a value of about 1600 on the arbitrary scale used, so that the residue of about 45 units which remains above the effective Néel temperature of $39\frac{1}{2}$° is only 3%. Setting aside therefore, for the present, this relatively insignificant 'tail' to the intensity curve, we conclude that the Néel temperature of pure chromium is $39\frac{1}{2}$ °C., to an accuracy of about $\frac{1}{2}$ °C. It will be noticed that separate points are marked in Fig. 2 for measurements taken during heating and cooling. The agreement between these suggests that there is no significant thermal hysteresis in the alignment of the magnetic moments on the chromium atoms, in contrast with the conclusions of Bykov *et al.* (1959).

Before discussing these results in more detail we will present the results of our measurements with polycrystalline material. At the outset we emphasize that this has to be examined by powder diffraction methods, and the data, and the conclusions therefore, will be much less accurate because of the greatly reduced intensities. Thus, in a typical measurement with the limited quantities of pure material which were available, the peak intensity at room temperature was about 40 counts per minute for the polycrystalline sample, in contrast to 6000 counts per minute from the single crystal which we have just

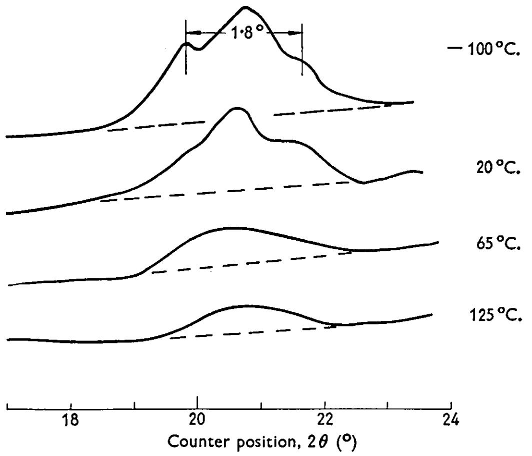

Fig. 3. The (100) powder reflection for coarse-grain polycrystalline chromium, indicating satellite structure below 40 °C.

discussed. Two samples of polycrystalline material, differing markedly in grain-size, were examined. As initially supplied these were in the form of slightly curved sheets but they were lightly broken up into fragments in order to randomise the grains in the sample as far as possible. They were enclosed in thin vanadium cans for the neutron-diffraction examination. Fig. 3 shows the 100 reflection at various temperatures for the first sample, which was a large-grain material with grains varying from about 0·2 to 2 mm. in size, with an average size in the region of 0·7 mm. There was also a well-defined subgrain structure with a very uniform subgrain dimension close to 0·05 mm. Although the angular resolution is much worse than for the single-crystal pattern, the influence of the antiphase domain structure in determining the shape of the diffraction lines can still be discerned. As we shall see later, the powder pattern will include the satellite reflections $1,\frac{1}{25},0$, $1,\frac{\bar{1}}{25},0$, $1,0,\frac{1}{25}$ and $1,0,\frac{\bar{1}}{25}$ in addition to $\frac{24}{25},0,0$ and $\frac{26}{25},0,0$ which we observed with the ($h00$) setting of the single crystal. These four additional lines have spacings which are very close to the 100 value and therefore appear centrally between the two $h00$ satellites to give the rather broad overall reflection which appears in Fig. 3. Correction has already been made in these curves for the scattering contributed by the sample holder and the walls of the Dewar vessel. It will be noticed that, in contrast to the results of Fig. 1 for the single crystal, a considerable fraction of the integrated intensity remains at 65 °C. and above. This conclusion is seen more clearly in the plot of intensity versus temperature which is shown in Fig. 4. With increase of temperature the intensity falls off as 40 °C. is approached but this initial decrease is succeeded by a substantial tail which persists up to about 200 °C. It is difficult to define precisely at what temperature this tail reaches the level of the background, set by the intensity of the $\lambda/2$ component for 200, but this temperature is certainly close to the value of T_N which was given by Shull & Wilkinson (1953).

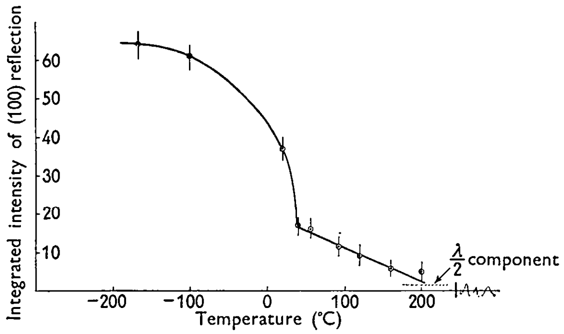

Fig. 4. The variation with temperature of the integrated intensity of the (100) powder reflection from a sample of large-grain polycrystalline chromium.

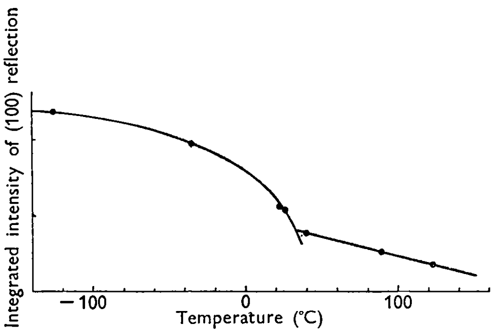

Fig. 5. The temperature dependence of the (100) reflection for fine-grain polycrystalline chromium.

The second polycrystalline sample was a fine-grain material for which the average grain size was 0·004 mm., and the largest grains were about 0·01 mm. The corresponding variation of the intensity of the 100 reflection with temperature is shown in Fig. 5. The general form of the curve is very similar to that in Fig. 4 but the proportion of material which contributes to the tail of the curve is higher than for the large-grain material.

The antiphase domain structure

Before considering further the implications of the different curves of intensity versus temperature for the single crystal and polycrystalline samples, we shall discuss in greater detail the antiphase-domain structure, as postulated by Corliss, Hastings & Weiss (1959). Fig. 6 illustrates in diagrammatic form the domain structure of a single-crystal cube of chromium. There can be three different orientations of antiphase domains parallel to the cube edges, as indicated by A, B and C. Each small cubic unit cell has chromium atoms at its corners and body-centre with their magnetic moments oppositely directed: in the figure only the corner atoms have their polarity marked. For simplicity the antiphase domains have been shown much smaller than they really are and only 10 unit cells have been drawn in each positive–negative unit compared with an experimentally determined value of about 25. If we consider the reflection of neutrons from the group of cells shown at C there will be zero intensity at the position of the 100 magnetic reflection because of the mutual cancellation of the sections PQ, QR. There will, however, be partial reinforcement at slightly larger and slightly smaller values of the Bragg angle $\theta \pm \delta\theta$, if $\delta\theta$ is such as to give an additional change of path length of $\pm\lambda$ between the contributions from P, R. It can be shown that the distance PR will be equal to $\lambda/2\delta\theta \cos\theta$ and at room temperature the angular separation of the satellites demonstrates that PR contains about 25 unit cells, amounting to approximately 70 Å. So far, therefore, as the domain PR is concerned, the 100 reflection will be replaced by two satellites which occur at the positions $\frac{24}{25},0,0$ and $\frac{26}{25},0,0$. The first of these is the more intense on account of the slightly different geometrical factor in the expression for the intensity and also because of the larger value of the scattering factor f. For the domain B, on the other hand, the directions close to [100] at which reinforcement can take place will be those for reflections by the planes which are shown dotted, thus giving reinforcement between atoms M and N. These are the planes which index as $1,\frac{1}{25},0$ and $1,\frac{\bar{1}}{25},0$. In the same way domain A will give the pair of reflections $1,0,\frac{1}{25}$ and $1,0,\frac{\bar{1}}{25}$. The net result, therefore, is that the 100 spot in reciprocal space is replaced by the grouping of six closely spaced spots which is shown at (iii) at Fig. 6. The relative intensities of these spots depend on the orientation of the magnetic moments relative to the domain wall. In fact it is found that at room temperature two of the spots are twice as intense as the other four, which is consistent with the belief that the magnetic moments are *parallel* to the domain wall, as indicated in the sketch at (v) in Fig. 6. In our single crystal measurements the vertical axis of rotation is the [013] axis so that the pattern of spots shown at (iii) has in fact to be tilted about $0x$ through $\tan^{-1}(\frac{1}{3})$ (approximately 18°) to correspond to the geometry of the spectrometer. The angular resolution of the system is sufficiently good to exclude the four smaller spots entirely when counter and crystal are synchronized in the $h00$ setting. As a result a traverse of the 100 region shows simply the $\frac{24}{25},0,0$ and $\frac{26}{25},0,0$ reflections and it is these which appear in Fig. 1. By tilting and rotating the crystal through the appropriate angle, and at the same time setting the counter at the correct value of 2θ it was found possible to observe individually the other four reflections i.e. $1,\frac{1}{25},0$, $1,\frac{\bar{1}}{25},0$, $1,0,\frac{1}{25}$ and $1,0,\frac{\bar{1}}{25}$, and to measure their intensities, which confirmed in detail that very pure chromium shows satellite reflections of this type. We have avoided saying that chromium possesses a particular type of antiphase-domain structure, since only a detailed demonstration of the presence of secondary satellites could distinguish the essentially 'discontinuous' model of antiphase domains from the alternative model of continuously spiralling spins. Overhauser & Arrott (1960) have discussed the different possible models. We have not found any convincing evidence of secondary satellites.

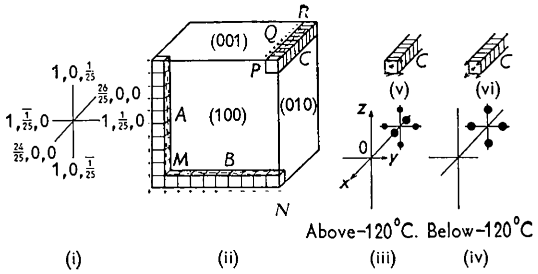

Fig. 6. Diagrammatic representation of the antiphase domains in chromium and the distribution of scattering density in reciprocal space.

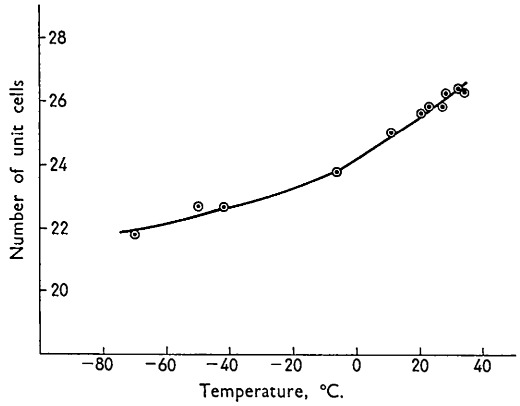

Fig. 7. The variation with temperature of the overall size (PR in Fig. 6) of the antiphase domains, measured in unit cells.

In terms of a model with antiphase domains, it is of interest to deduce the change of domain size with temperature from the variation in the angular separation of the $\frac{24}{25},0,0$ and $\frac{26}{25},0,0$ reflections. The data are presented in Fig. 7 in the form of a variation with temperature of the number of unit cells which form a complete domain unit, such as the assembly PR in Fig. 6(ii). The number of cells increases continuously with temperature, and more rapidly as the Néel temperature is approached. Clearly it is the average number which we determine, though the sharpness of the reflection precludes much variation. There is no justification for concluding that this 'parameter' is an integer. From Fig. 7 we form a picture of antiphase domains which expand in size until they become unstable so that, in the case of the single crystal, both they and the underlying antiferromagnetic arrangement between neighbouring chromium atoms break up practically completely at 40 °C. It may be significant that as the temperature increases the neutron intensity falls away more quickly than would be expected for a Brillouin type of dependence. We might imagine that the instability in the long-range magnetic order could be prevented if domains could become locked in position at strains and crystallite boundaries such as will occur in polycrystalline material. In the latter the underlying antiferromagnetic arrangement might then be preserved up to some high temperature. There would not be any well-defined domain size but basic antiferromagnetism between nearest neighbour atoms would persist over distances of the order of a few hundred Ångström units: by contrast, below 40 °C. unit blocks of cells of 70 Å in length succeed one another in completely ordered $+-+-+-$ fashion over even greater distances. On the alternative picture of spiralling spins we assume that in place of the steadily rotating screw which occurs below 40 °C. we can preserve a screw with random defects, pinned in position, at higher temperatures in polycrystalline material. This picture is very similar to that followed by Overhauser & Arrott who regard the defects and terminating effects in the polycrystalline material as the means of preventing the nodes in their spin density wave from falling near lattice sites. New domains are then permitted to start at the defects, so as to have a more favourable phase relation to their spin-density wave. Overhauser & Arrott suggest that the Néel temperature could be doubled in this way. Any detailed domain picture and distribution could be tested in principle by a study of the line shapes of the 100 reflection above and below 40 °C. However, the intrinsic weakness of the powder diffraction lines and the fact that there is only a very weak tail to the single-crystal curve means that no accurate conclusions can be obtained. Nevertheless, such measurements as can be made do lend some plausibility to the explanation. The width of the 100 powder reflections gets less with increase of temperature and at 90 °C. is only 70% of the value at room temperature, and there is certainly no evidence for the existence of any satellite structure above 40 °C. We have examined carefully both the rocking curve and the spectrum shape for the remnant of the 100 single crystal reflection which persists above 40 °C. In the curve for 40 °C. in Fig. 1 there are no discrete satellite peaks, although the remaining intensity is sufficiently large for these to have been distinguished if they appeared in defined positions. If the contribution from the $\lambda/2$ component, which is shown dotted in Fig. 1, is subtracted, then the resulting spectrum has a width of about $1\frac{1}{2}^\circ$ in 2θ. The rocking-curve width, however, is much greater, about 5° in θ, suggesting that the tail for the single crystal might be due to a residue of antiphase units with a wide distribution of size, contributing to reflections of the type $1,0,\frac{1}{5}$ or $1,\frac{1}{25},0$ which have spacings close to the 100 value.

The low-temperature transition

In a recent paper, Bykov *et al.* (1959) have observed a low-temperature transition in chromium which was revealed by the disappearance of the 100 single-crystal reflection at -115 °C. The experimental observation is repeated in our Fig. 1 which shows, in a measurement at -140 °C., the disappearance of the two satellite reflections, leaving only the $\lambda/2$ component of the 200 nuclear reflection. Such an apparently drastic disappearance of antiferromagnetism is not observed in polycrystalline patterns, nor in fact in single crystal observations with poor angular resolution. Hastings (1960) has shown that it is due simply to a spin-flip of the magnetic moments from positions parallel to the antiphase-domain wall into positions perpendicular to the wall. Since there is no magnetic scattering from a magnetic moment which is parallel to the scattering vector the reflections $\frac{24}{25},0,0$ and $\frac{26}{25},0,0$ become of zero intensity whereas the other satellites increase in intensity, to give the modified distribution in reciprocal space as indicated in Fig. 6(iv). Hastings' explanation has been confirmed directly with our crystal by demonstrating that the other reflections such as $1,\frac{1}{25},0$ still remain, with doubled intensity as expected, and they can be brought into the reflecting position by rotating the crystal through the calculated angle of about 2°, which is the angle between the true 100 plane and the dotted planes in Fig. 6(ii). Our measurements of the temperature at which this change of spin direction occurs for pure chromium lie between -118 and -120 °C. which is in satisfactory agreement with the value given in the Russian paper.

We have explored the possibility that the disappearance of the single-crystal antiferromagnetic reflection around 40 °C. might be due to some similar kind of change of moment orientation—though undoubtedly it is not of the 'instantaneous' nature which is found at -120 °C. However, in spite of a careful search for other reflections about 40 °C. we have obtained no evidence (other than the small tail in Fig. 2) of any remaining magnetic reflections. Nevertheless we must acknowledge that random searching with a counter detector is not a very powerful means of detecting unexpected reflections.

Determination of μ_B

The value of the magnetic moment of chromium in the metal is of particular interest in the theoretical interpretation of the electronic configurations of metals and alloys. In the original neutron-diffraction experiments of Shull & Wilkinson it was concluded that the moment was 0·4 Bohr magnetons and we have made a number of measurements with our pure metal to test this value.

Apart from the experimental measurement of the diffraction intensities, several other factors are involved in the determination—in particular the extrapolation of the scattering factor curve to the value at $\theta = 0°$ and the extrapolation of the low-temperature data to 0 °K. to give complete alignment of the magnetic moments. For the former we have used the scattering-factor curve determined experimentally for Mn^{2+}. There is some evidence (Weiss, 1959) that the intensities of higher-angle reflections of chromium such as 111 and 210, fall off as expected according to this curve, which approximates to $f = \exp(-0{\cdot}075k^2)$, where $k = 4\pi \sin\theta/\lambda$. At the present time therefore this Mn^{++} curve would seem to represent the most plausible assumption: for the 100 reflection the value of f is 0·69. Our low-temperature measurements were made at temperatures T between 125° and 147 °K., for which the values of T/T_N, where T_N is the Néel temperature, are between 0·40 and 0·47. We have applied the appropriate small correction factor to the intensities on the assumption that these show a Brillouin type of temperature dependence in this region. The correction varies from 2 to 6% between these limits.

Because of the antiphase-domain structure, or some equivalent structure, there is no complete reinforcement of the scattered contributions from all the atoms at the peak of the reflections such as $\frac{24}{25}$,0,0 and 1,$\frac{1}{25}$,0. Calculation of the structure factors for these reflections shows that each satellite reflection has an intensity which is 0·41 of the intensity which a normal continuously-phased antiferromagnetic domain would contribute to the (100) magnetic reflection.

With the above assumption we have determined the value of μ_B from our powder-diffraction intensities, using the accompanying nuclear reflections as a standard and making a very small correction for thermal vibrations appropriate to a Debye temperature of 520 °K. For the large-grain polycrystalline sample we conclude that $\mu_B = 0{\cdot}42$. For the small grain sample we get $\mu_B = 0{\cdot}47$. We do not believe that there is any significant difference between these two results, though we should expect the latter to be the more accurate as there will be a better statistical distribution of the small grains. In order to make a proper comparison with Shull & Wilkinson's original value of $0{\cdot}4\mu_B$ for a powder we have to increase the latter by the reciprocal of $\surd(0{\cdot}82)$ to allow for the reduction of structure factor in the satellite reflections, resulting in a value of $0{\cdot}44\mu_B$ which is entirely consistent with the present measurement with pure material.

We have also deduced a value of μ_B from our single crystal data. A direct comparison of the intensities of the 100 group of satellites with the 200 nuclear reflection leads to a value of 1·3 for μ_B but there is reason to believe that this value is quite false because of the very large effect of secondary extinction which heavily reduces the value of the intense nuclear reflections. This would not be unexpected with the large crystal in which the neutron path length is 4 mm. In principle we could demonstrate the correctness of this interpretation by cutting off a small fragment of the single crystal and repeating the measurements. However, not wishing to mutilate the large crystal in this way at the present stage, we have established the point indirectly. We have shown, by comparison with other crystals of smaller size, that the intensity of the 200 nuclear reflection is 5–10 times smaller than would be expected from a crystal of its measured size. Further, we have confirmed the conclusion more precisely in measurements with a small, less pure single crystal of chromium to which we shall refer in the next section.

Metallic impurities in chromium

In measurements of electrical resistance and Hall effect, de Vries (1959) has shown that the anomalies which occur near 38 °C. for pure material can be shifted to very different temperatures by alloying 1 at.% of other transition elements with the chromium. For example 1% of manganese moves the transition temperature up to 150 °C., whereas 1% of iron and nickel move it down to about 25 °C. and −50 °C. respectively. We have studied a single crystal containing 0·5% of iron and 0·16% of nickel (but not more than 0·01% of gaseous impurities), examining in particular the temperature dependence of the intensity of the 100 magnetic reflection. The results are expressed in Fig. 8 from which it is clear that the Néel temperature is well below the value of 39 °C. found for the pure crystal and amounts indeed to about

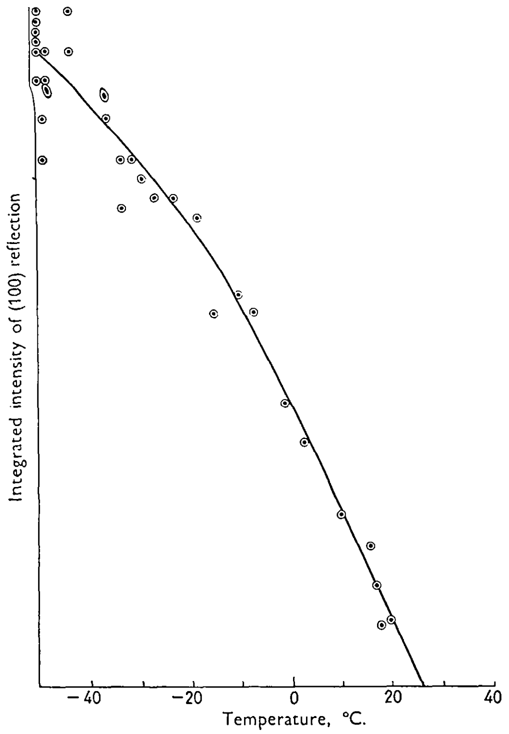

Fig. 8. Determination of the Néel temperature of a single crystal of chromium containing 0·50% Fe and 0·16% Ni.

26 °C. The reduction is of the order expected from de Vries's results and serves to confirm that the anomalies in electrical resistance and Hall effect do occur at the Néel temperature where the antiferromagnetic arrangement breaks up. Once again the reduction of neutron intensity as the Néel temperature is approached is rather faster than expected for a Brillouin function.

This 'alloy' crystal had a mass of only 0·08 g. Its length was roughly parallel to the [110] axis and in the two possible positions for reflection from the 200 planes the neutron path lengths through the crystal were about 0·5 mm. and 2·0 mm. There was no significant difference in the reflection intensities in the two cases and this suggests that there was no appreciable effect due to secondary extinction. It was noteworthy that although the 100 magnetic reflection was reduced by a factor of 25 relative to the large pure crystal, yet the 200 reflection was only reduced by 3·7, confirming our previous deduction of heavy extinction of the 200 in the large crystal. From two independent sets of measurements, which involved remounting and realigning the crystal, we obtained μ_B values of 0·49 and 0·45 which we can compare with the values of 0·42 and 0·47 for our polycrystalline samples.

We conclude therefore that the value of μ_B for chromium at temperatures well below the Néel temperature is $0{\cdot}45\mu_B$ and that this value is the same in polycrystalline and single-crystal material and is not affected (at least in our particular example) by a small amount of metallic impurity. It is not possible to give a rigid standard deviation on this value, but from the general agreement of our various values it would appear to be correct to $0{\cdot}04\mu_B$, assuming that the form factor for Mn^{++} is an adequate approximation for the extrapolation to $\theta = 0°$.

One or two further observations made with the small impure crystal are of interest. The crystal showed the same type of satellite structure on cooling down below 26 °C. and the angular separation of the satellites indicated that the antiphase domains were of approximately the same size as for the large pure crystal. Since the crystal axis was [110], instead of [130], the reflections $1,\frac{1}{25},0$ and $1,0,\frac{1}{25}$, for example, come in at the same angular setting of the crystal. This position lay at the expected angular displacement from the $h00$ setting. Finally we have not been able to observe the spin-flip transition at low temperature. So far we have only been able to cool our crystal to −150 °C., which is 30° below the transition temperature found for the pure crystal, but down to this temperature we have seen no change in the details of the satellite reflection. We hope to investigate the behaviour at lower temperatures in due course.

We are particularly grateful to Mr A. R. Edwards and his colleagues of the Aeronautical Research Laboratories, Melbourne, for the supply of the pure chromium and for information on its grain size and chemical analysis; and to Messrs R. F. Dyer and W. Lowe of A.E.R.E. Harwell for much assistance with the experimental measurements.

References

Bacon, G. E. & Dyer, R. F. (1959). *J. Sci. Instrum.* **36**, 419.

Bykov, V. N., Golovkin, V. S., Ageef, N. V., Levdik, V.A. & Vinogradov, S.I. (1959). *Doklady Akad. Nauk. SSSR.* **128**, 1153.

Corliss, L. M., Hastings, J. M. & Weiss, R. J. (1959). *Phys. Rev. Letters*, **3**, 211.

Hastings, J. M. (1960). Reported at the conference on Neutron Diffraction at Gatlinburg, U.S.A., April 1960.

Herpin, A., Meriel, P. & Villain, J. (1959). *C. R. Acad. Sci., Paris*, **249**, 1334.

Overhauser, A. W. & Arrott, A. (1960). *Phys. Rev. Letters*, **4**, 226.

Powell, R. W. & Tye, R. P. (1956). *J. Inst. Met.*, **85**, 185.

Shull, C. G. & Wilkinson, M. K. (1953). *Rev. Mod. Phys.* **25**, 100.

Vries, G. de (1959). *J. Phys. Radium*, **20**, 438.

Weiss, R. J. (1959). Private communication.