THE MAGNETIC STRUCTURE OF Mn_2Sb

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Abstract—Neutron diffraction investigations on single crystals and powdered samples of Mn_2Sb have determined the magnetic structure of this compound. The ferrimagnetic structure predicted by GUILLAUD was confirmed except for the exact values of the individual atomic moments of the manganese atoms. Values of $+2\cdot13\pm0\cdot20$ μ_B and $-3\cdot87\pm0\cdot40$ μ_B were obtained for the magnetic moments of the Mn atoms on the two sublattices from single crystal investigations, and these experimental values satisfactorily explain the scattering from powdered samples at temperatures from 78°K to about 800°K. Results from previous macroscopic magnetic experiments are also consistent with these values of the atomic magnetic moments.

INTRODUCTION

THE compound, Mn_2Sb, has been found to possess unusual magnetic properties which have been the subject of considerable investigation. The crystal structure of Mn_2Sb was determined by HALLA and NOWOTNY[1] to be tetragonal with two molecules in a unit cell of lattice constants $c = 6\cdot56$Å and $a = 4\cdot08$Å. The manganese atoms are located at positions with co-ordinates $(0, 0, 0)$; $(\frac{1}{2}, \frac{1}{2}, 0)$; $(0, \frac{1}{2}, Z_1)$; $(\frac{1}{2}, 0, \overline{Z}_1)$, while the antimony co-ordinates are $(0, \frac{1}{2}, Z_2)$ and $(\frac{1}{2}, 0, \overline{Z}_2)$. Values obtained for the parameters in these early measurements were $Z_1 = 0\cdot27$ and $Z_2 = -0\cdot30$, but neutron diffraction results[2] disclosed evidence that these values might be in error. Recent results by both neutron diffraction and X-ray diffraction have confirmed these errors and established new values for the parameters. An X-ray investigation by HEATON and GINGRICH[3] which included 17 orders of reflection from the $(00l)$ planes of a large single crystal found Z_1 to be $0\cdot295$ and Z_2 to be $-0\cdot280$, while neutron data taken for nine orders of $(00l)$ reflections gave values of $0\cdot293$ and $-0\cdot279$ for Z_1 and Z_2 in excellent agreement with the X-ray measurements. It should also be

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stated that weak anomalous reflections at or near the (100) and (300) positions which were reported in the X-ray investigation by HEATON and GINGRICH have been observed in the neutron scattering from the $(h00)$ planes of a single crystal. The origin of these reflections, which should be absent if the structure is described by the above two parameters, is not understood.

The first indication of the unusual magnetic properties of Mn_2Sb was found in the measurements of GUILLAUD,[4] who predicted that this material is *ferrimagnetic* below the Curie point, T_c, of 550°K. His measurements of the spontaneous magnetization at low temperatures determined the saturation atomic magnetic moment to be 0·936 Bohr magnetons per manganese atom, and this value decreased abnormally rapidly with increasing temperature. As an explanation of these results, GUILLAUD suggested that the two types of manganese atoms present in the unit cell would be in different magnetic states. One type of atom, Mn(II), located at co-ordinates $(0, \frac{1}{2}, Z_1)$ and $(\frac{1}{2}, 0, \overline{Z}_1)$ would be in the $3d^54s^2$ electronic state, while the other Mn co-ordinates would be filled with Mn(I) atoms consisting of a temperature-dependent mixture of manganese atoms in the $3d^7$ and $3d^54s^2$ electronic states. Both groups of magnetic ions would be spontaneously magnetized below T_c but in opposite directions. This model satisfactorily accounted for the

non-integral value of the saturation moment and also the temperature variation of the spontaneous magnetization. Furthermore, it explained qualitatively the unusual temperature behavior of the inverse magnetic susceptibility measured by SERRES.[5] However, in 1948, NÉEL developed the molecular field treatment of ferrimagnetism which predicted that the magnetization of the two sublattices of a ferrimagnetic material could have quite different variations with temperature. This theory satisfactorily explained the unusual temperature variation of the net spontaneous magnetization and the inverse susceptibility of Mn_2Sb without requiring a change in the population of the electronic states with temperature. In fact, good quantitative agreement with the magnetic measurements was obtained with the assumption that the manganese atoms in the two sublattices had spin atomic magnetic moments of 5 Bohr magnetons and 3 Bohr magnetons, respectively, corresponding to GUILLAUD's suggestion that the two kinds of Mn atoms were in $3d^5 4s^2$ and $3d^7$ electronic states. The variation of the measured value of the saturation moment from unity was presumed to be due to a slight contribution from orbital magnetic moments or to an inequality in the numbers of the two types of manganese atoms. Measurements on single crystals of Mn_2Sb by GUILLAUD, BERTRAND, and VAUTIER[7] have shown that there is a change in the direction of easy magnetization at 240°K. Between 240°K and T_c, the atomic moments are directed along the c-axis of the unit cell, while at temperatures below 240°K, the moments are located in the base plane of the unit cell.

Fig. 1 represents the magnetic structure of Mn_2Sb which was predicted on the basis of the macroscopic magnetic measurements which have just been reviewed. It was of interest to examine this compound by neutron diffraction to determine if the predicted magnetic model was correct. Studies were made on both powdered samples and single crystals of the material, and the results of the two types of investigations are qualitatively compatible with the ferrimagnetic structure assumed by GUILLAUD.

EXPERIMENTAL TECHNIQUES

During the course of these experiments, it was necessary to study the scattering characteristics of Mn_2Sb at temperatures from 78°K to about 800°K. The low temperature measurements were made with the samples
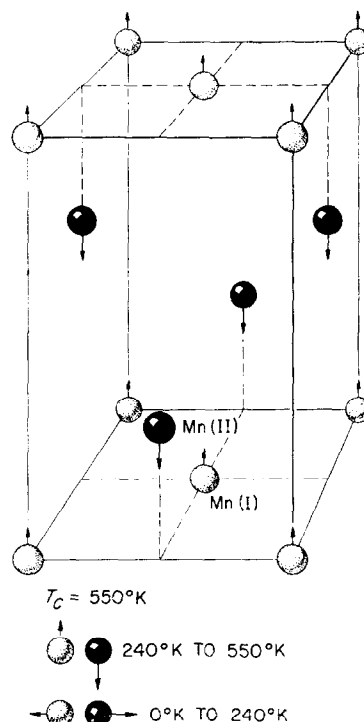

$T_C = 550°K$

240°K TO 550°K

0°K TO 240°K

FIG. 1. Magnetic structure proposed for Mn_2Sb by GUILLAUD from magnetic measurements. Suggested values of atomic magnetic moments were $+3$ μ_B for Mn(I) and -5 μ_B for Mn (II).

enclosed in a double-jacketed vacuum cryostat at pressures below 10^{-6} mm Hg, while the data at high temperatures were obtained in a vacuum furnace at pressures lower than 10^{-5} mm Hg. Effects of extraneous scattered radiation were kept to a minimum by containing the powdered samples in cells made from vanadium or aluminum and by passing the neutron radiation through windows in the radiation shields which were also of thin vanadium and aluminum.

The single crystal specimens were cut from two large crystals grown from different samples of manganese and antimony. One of these crystals (Crystal H) was obtained from Horizon's, Inc., Cleveland, Ohio. The other crystal (Crystal M) was grown at the University of Missouri by HEATON and GINGRICH and was the same specimen which they used in the X-ray examination[3] of the crystal structure of Mn_2Sb. Both large crystals were irregular in shape with dimensions of the order of two centimeters. Quantitative chemical analyses showed that the crystals were stoichiometric within 2 per cent, the accuracy of the analyses. The value of the net magnetic moment* at room temperature deter-

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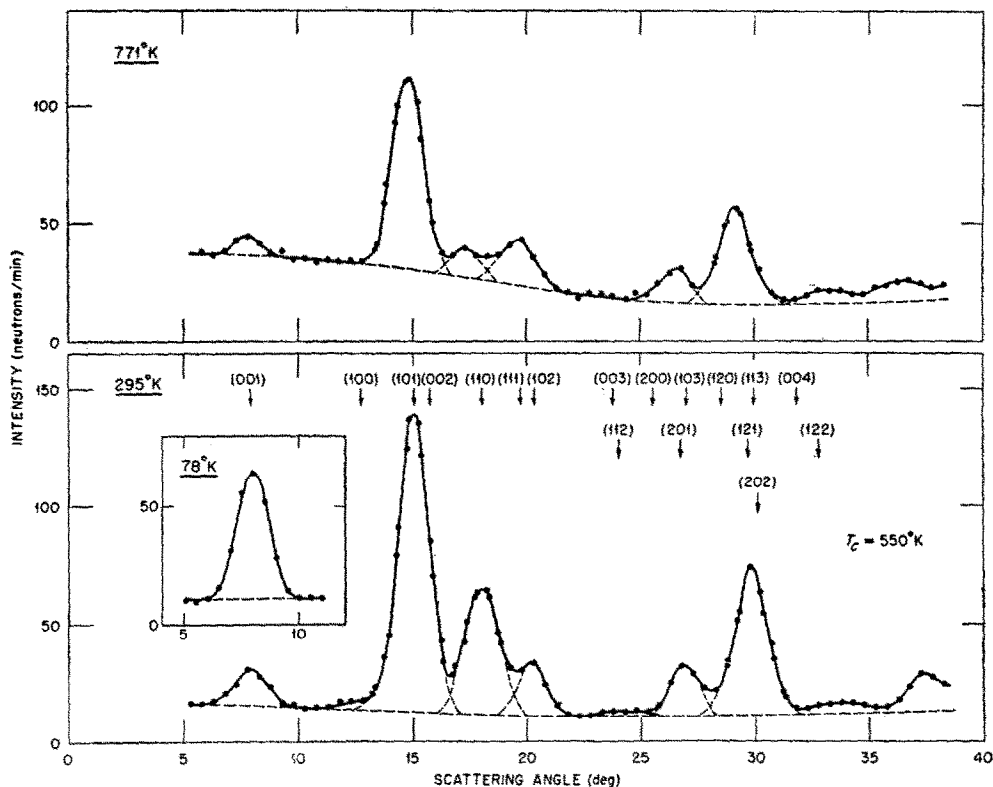
FIG. 2. Neutron diffraction patterns from powdered samples of Mn₂Sb at an effective neutron wave length of 0·900Å for three sample temperatures.

mined from magnetization experiments on a specimen cut from Crystal M was found to agree within 2 per cent with GUILLAUD's value for the exact composition Mn₂Sb. According to GUILLAUD's measurements of the change in net magnetic moment with composition, this agreement would imply that the chemical composition of our sample and that of GUILLAUD were the same within $\frac{1}{2}$ per cent. The specimens which were used in the neutron diffraction experiments were cut in the form of small pillars about $\frac{1}{2}$ inch in length with approximately square cross-sections which varied from about 1 mm to 3 mm on a side.

In the experiments with single crystals it was necessary to examine the scattering from the samples when they were placed in an external magnetic field. An electromagnet was designed for these experiments in which the small pillars could be mounted either perpendicular or parallel to the plane of scattering, and a magnetic field could be applied along the axis of the pillar. The magnet was capable of producing a magnetic field of 9000 oersteds across a gap of $\frac{1}{8}$ inch, and this field was found to be sufficient to align the atomic magnetic moments along all of the crystallographic directions which were studied.

EXPERIMENTAL RESULTS

Results from Powdered Samples

The initial neutron diffraction investigations were performed on powdered samples of Mn₂Sb contained in thin-walled cells of aluminum or vanadium at various temperatures from 78°K to about 800°K. Fig. 2 shows powder patterns which were obtained with an effective neutron wave length of 0·900Å for sample temperatures of 295°K and 771°K, and the insert shows a portion of the diffraction pattern around the (001) reflection which was obtained at 78°K. Since the Curie transition in Mn₂Sb occurs at 550°K, the diffraction peaks at 771°K contained only a nuclear scattering contribution, while those at 295°K resulted from both nuclear and magnetic scattering. A comparison of the two patterns allowed a determination of the coherent magnetic scattering at 295°K, and this scattering was found to confirm

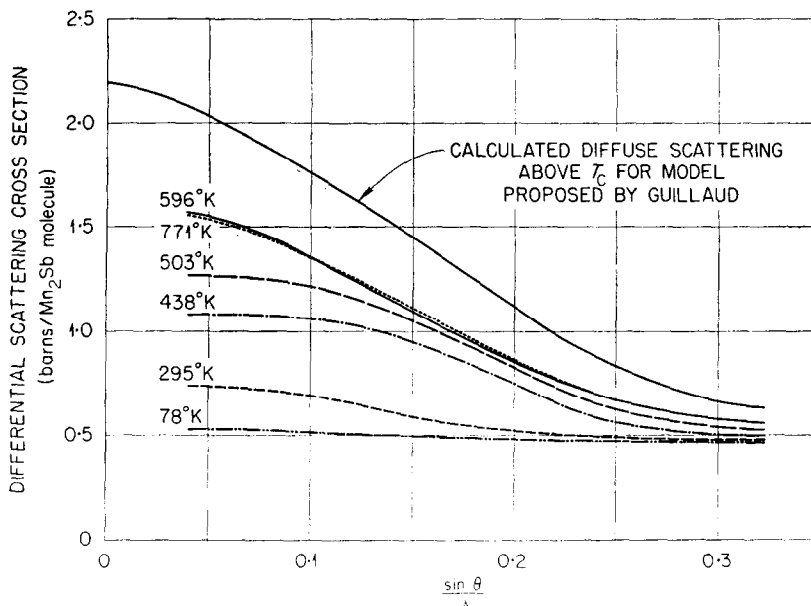FIG. 3. Diffuse scattering from Mn₂Sb powder at several sample temperatures. The calculated curve has been determined for atomic magnetic moment values of 3 μ_B and 5 μ_B as suggested by GUILLAUD.

qualitatively GUILLAUD's proposed magnetic model. Specifically, confirmation was obtained of the ferrimagnetic arrangement of the atomic magnetic moments located at the manganese positions, and the manganese atoms designated as Mn(II) were found to possess moments larger than those of the Mn(I) atoms. Furthermore, as shown by the intensity change in the (001) reflection at 78°K, the directions of the magnetic moments changed at some temperature between 295°K and 78°K from along the c-axis to within the base plane of the unit cell, without affecting the antiparallel orientation of the moments. However, while qualitative confirmation was obtained for GUILLAUD's model, the reflections did not confirm the suggested values of the atomic magnetic moments. The data at 295°K were not conclusive in suggesting other values, but the 78°K data required that both types of manganese atoms have magnetic moments which were smaller than those predicted.

Analyses were also made of the diffuse scattering in the powder diffraction patterns. In addition to the decrease in coherent scattering at 771°K compared to that at 295°K, it is seen that there is a pronounced increase in the diffuse background

which was caused by the presence of paramagnetic scattering. Observations were made of this diffuse background as a function of temperature, and Fig. 3 shows the results at the various temperatures after they were converted to differential scattering cross-sections. These curves have been corrected for instrumental background and for thermal diffuse scattering using the Debye theory of independent oscillators for a characteristic temperature of 290°. The curve at 78°K indicates the presence of very little magnetic disorder scattering, a result that would be expected from GUILLAUD's magnetic measurements which show that at 78°K the net magnetization is over 98 per cent of its value at absolute zero. As the temperature of the sample was increased and the sublattices became less saturated magnetically, the intensity of the diffuse scattering increased with the disorder in the alignment of the atomic moments. It is interesting that the data at 596°K, less than 50 degrees above T_c, show no indication of short-range order. Instead, this curve has an angular dependence which is quite comparable with the magnetic form factor that has been observed previously[8, 9] for the divalent manganese

ion. Furthermore, the similarity of the data at 596°K and 771°K indicates that all of the paramagnetic scattering is present in the diffraction patterns at these temperatures.

For comparison with the experimental data, a curve showing the expected diffuse scattering in the paramagnetic region for the model proposed by GUILLAUD is also shown in Fig. 3. This calculated curve represents the sum of the S^2 term in the expression for the differential scattering cross-section for a paramagnetic atom[10] and the experimental data at 78°K. While this method of calculation is an approximation, it appeared to be the best method for obtaining a curve which could be compared with the diffuse scattering at high temperatures. It is immediately obvious that the calculated curve for the diffuse scattering above T_c is much higher than that actually obtained from these experiments. Hence, both the coherent scattering and the diffuse scattering in the powder diffraction patterns suggest values for the atomic magnetic moments of the manganese atoms which are lower than those of the proposed model. Attempts were made to extract the actual values of the magnetic scattering amplitudes and the corresponding atomic magnetic moments from the powder data, but the complexity of the patterns and the difficulties of the analysis did not permit an unambiguous determination.

Results from Single Crystals

Room temperature measurements in electromagnet

The single crystal specimens were cut in the form of pillars with the orientation of the pillars chosen with respect to various crystal planes so that scattering data could be obtained from those reflections which have important magnetic components. The specimens were mounted in the gap of the electromagnet and data at room temperature were obtained with the samples both magnetized and unmagnetized.

As derived by HALPERN and JOHNSON,[10] the diffracted intensity in a Bragg reflection is proportional to the differential scattering cross-section which for incident unpolarized neutrons is given by

$$F^2 = b^2 + q^2 p^2$$

in which b and p are the nuclear and magnetic

structure amplitudes, and

$$q^2 = 1 - (e \cdot k)^2$$

where e is the unit scattering vector and k is a unit vector parallel to the atomic magnetic moment. Hence, as shown experimentally by SHULL, WOLLAN, and KOEHLER,[11] the amount of magnetic scattering in reflections from a material with a net spontaneous magnetization can be varied by the application of an external magnetic field of sufficient strength to orient the magnetic domains. In the experiments with single crystals of Mn₂Sb, the magnetic field was always applied in the direction relative to the scattering vector which would give the largest change in intensity from that of the unmagnetized sample for the particular reflection under investigation.

The diffraction patterns shown in Figs. 4 and 5 are typical of those which were obtained in these experiments. For the $(00l)$ reflections shown in Fig. 4, there was no magnetic contribution in the reflections of the unmagnetized sample, since at 295°K the atomic moments are aligned along the c-axis of the unit cell. However, when the crystal was magnetized perpendicular to the c-axis and perpendicular to the scattering vector, the entire magnetic scattering allowed by the structure factors was present in the diffraction peaks. Conversely, as shown in Fig. 5, for the $(hh0)$ reflections, the entire magnetic scattering amplitude was present in the coherent scattering from the unmagnetized sample, and this magnetic scattering was reduced to zero when the crystal was magnetized parallel to the scattering vector. Similar data were taken for all the important magnetic reflections, and a comparison of the patterns taken with the samples magnetized and unmagnetized allowed a direct determination of the magnetic scattering in each reflection. It was necessary to know if an external magnetic field of 9000 oersteds was sufficient to align the atomic moments in the direction of the field, and, consequently, the intensities of the reflections were studied as a function of the magnetic field produced in the gap of the electromagnetic. The data shown in Fig. 6 for the (110) reflection represent typical results for this investigation. While the exact field required to swing the magnetic moments may have been slightly different for the various
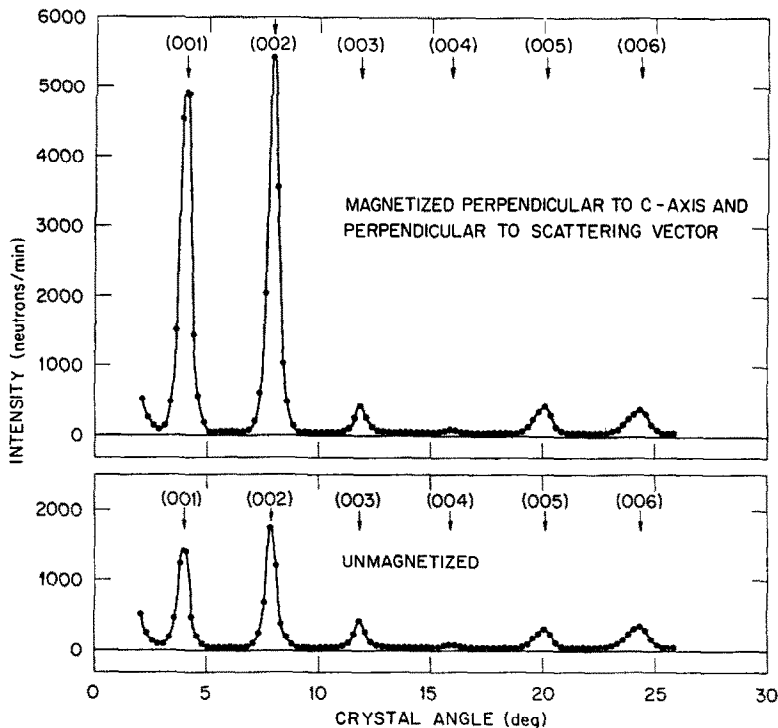
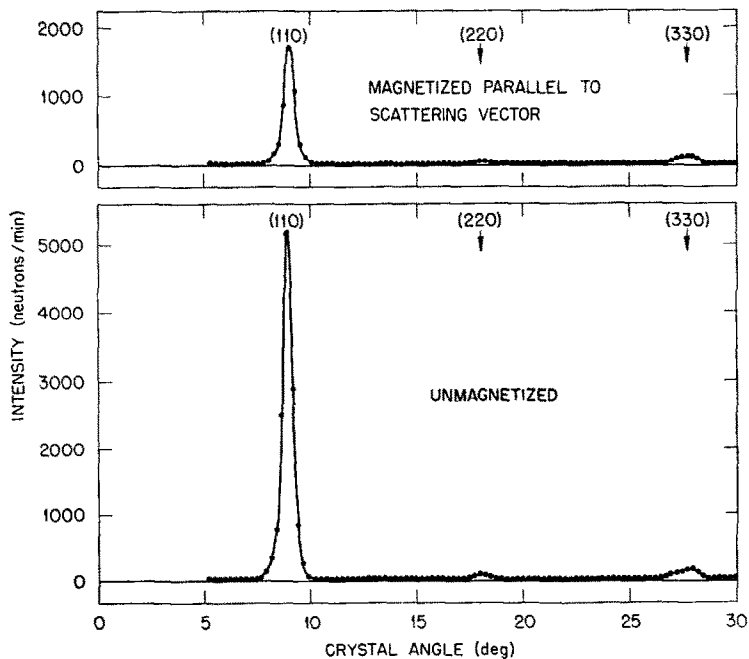FIG. 4. Neutron diffraction patterns of the (00l) single-crystal reflections from Mn$_2$Sb.

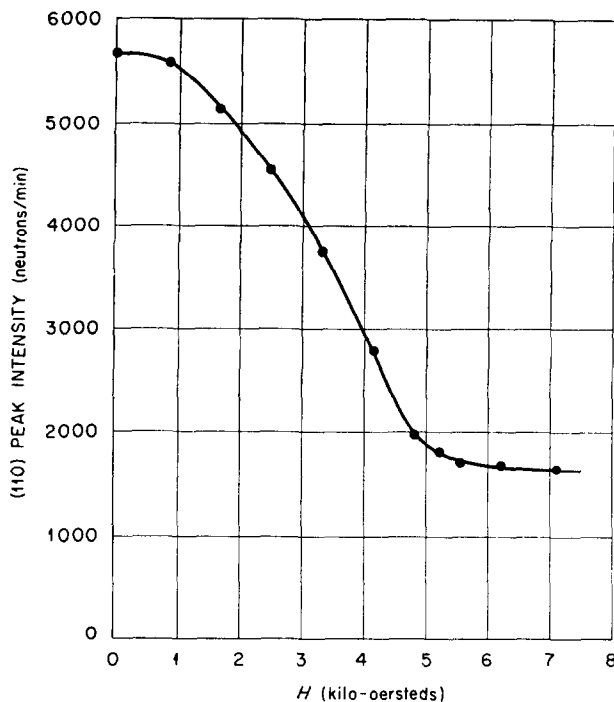FIG. 5. Neutron diffraction patterns of the (hh0) single-crystal reflections from Mn$_2$Sb.

FIG. 6. Magnetic structure amplitudes of the magnetic reflections from Mn₂Sb plotted as a function of the magnetic scattering amplitudes of the two manganese atoms. Solid lines represent data on specimens cut from Crystal *H*, and dotted lines represent data on specimens cut from Crystal *M*.

reflections, a field of about 5000 oersteds was required to reach approximate saturation in each case.

The single crystal measurements were consistent with the powder diffraction data in a qualitative confirmation of GUILLAUD's model. Furthermore, an examination of a series of (00*l*) reflections showed that the direction of the atomic moments at room temperature could not be more than a few degrees from the *c*-axis. With confirmation of the model, it was then possible to use the single crystal reflectivities to refine the moment values. For this magnetic model, an expression was known for the magnetic structure amplitude, p, for each reflection in terms of the two magnetic scattering amplitudes, $p_{Mn(I)}$ and $p_{Mn(II)}$, for the two types of manganese atoms. Furthermore, the nuclear structure amplitude for each reflection was readily calculable from known nuclear scattering data of manganese and antimony. The magnetic neutron

diffraction data from the single crystals were therefore effectively normalized by a direct comparison of the ratio of magnetic scattering to nuclear scattering experimentally determined for each reflection to that which was calculated assuming that the intensities were proportional to the squares of the structure amplitudes. This comparison gave an expression for the magnetic structure amplitude of each reflection as a function of $p_{Mn(I)}$ and $p_{Mn(II)}$ by a method which eliminated geometrical considerations of the shapes of the pillars. Most of the reflections were studied with at least two pillars which varied in thickness by a factor of three, and the expressions obtained for the magnetic structure amplitudes of these reflections were indistinguishable. These results show that the mosaic spread in the crystals was sufficiently large to make extinction effects negligible. In fact, a calculation of possible extinction effects for reasonable values of the
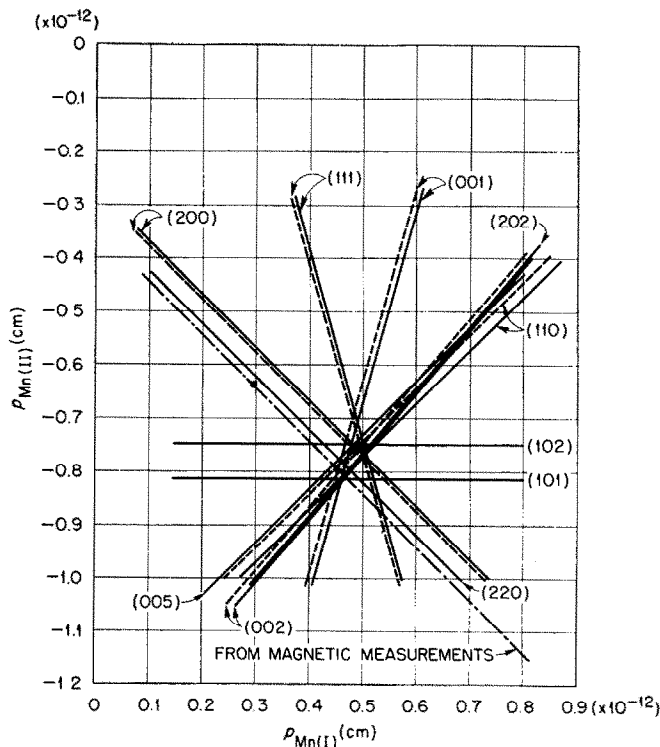
FIG. 7

mosaic spread shows that they are negligibly small because of the low absolute reflectivities of the reflections which have been studied.

Since the magnetic structure amplitudes for the various reflections contained a form factor variation, it was necessary to refer them to zero scattering angle before a determination could be made of the individual values for $p_{Mn(I)}$ and $p_{Mn(II)}$. This required a knowledge of the form factor for Mn_2Sb, and since this was also in doubt, an iterative process was required. For the first approximation, the magnetic form factor suggested by ERICKSON[9] for the divalent manganese ion was used, and only the (001), (002), (110), and (111) reflections were considered. These reflections had reasonably large magnetic contributions and occurred in a range of $(\sin\theta)/\lambda$ where the various experimental and theoretical form factors for scattering from the $3d$ electrons were in satisfactory agreement. The values of $p_{Mn(I)}$ and $p_{Mn(II)}$ obtained from this analysis were then

used to calculate the magnetic form factor of each of the magnetic reflections of Mn_2Sb that was measured. A smooth curve through these form factor values then represented a new average form factor from which new values of $p_{Mn(I)}$ and $p_{Mn(II)}$ could be determined using all the magnetic reflections. This procedure was repeated until the values converged, and this convergence was found to be quite rapid.

Fig. 7 is a graphical representation showing the final expressions which were obtained for the relationship between $p_{Mn(I)}$ and $p_{Mn(II)}$ for each of the reflections which was measured. The final magnetic amplitude form factor curve obtained for the magnetic manganese atoms in Mn_2Sb is shown in Fig. 8. The data shown in these two figures are representative of many pillars cut from the original two large crystals. In Fig. 7, the solid lines represent data on specimens cut from Crystal H, and dotted lines represent data on specimens cut from Crystal M, while this same distinction is made in

Fig. 8 by the solid points and open circles. It is significant that the data for specimens cut from two different crystals are in good agreement and give essentially the same values of the form factor and magnetic scattering amplitudes. The final
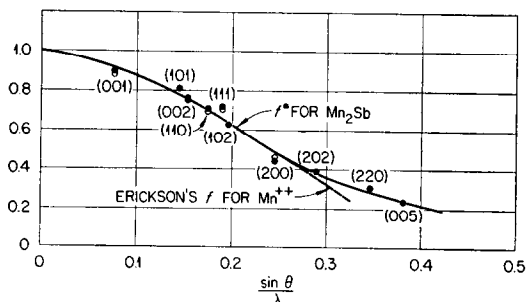

FIG. 8. Magnetic amplitude form factor for manganese atoms in Mn₂Sb. The solid points represent data on specimens cut from Crystal H, and open circles represent data on specimens cut from Crystal M.

form factor which best represents the Mn₂Sb single crystal reflectivities can not be distinguished from that suggested by ERICKSON at values of $(\sin \theta)/\lambda$ up to 0.26, while at higher angles, the Mn₂Sb data predict larger values than those indicated by the previous experiments for divalent manganese. The values of the magnetic scattering amplitudes at room temperature were determined to be $p_{Mn(I)} = +0.485 \pm 0.030 \cdot 10^{-12}$ cm and $p_{Mn(II)} = -0.780 \pm 0.060 \cdot 10^{-12}$ cm. These values agree within experimental error with those which would be predicted on the results of the magnetization measurements represented by the dashed line in Fig. 7.

Low temperature measurements

In order to determine the saturation atomic magnetic moments, it was necessary to know the degrees of magnetic saturation for the individual sublattices, since according to the NÉEL theory of
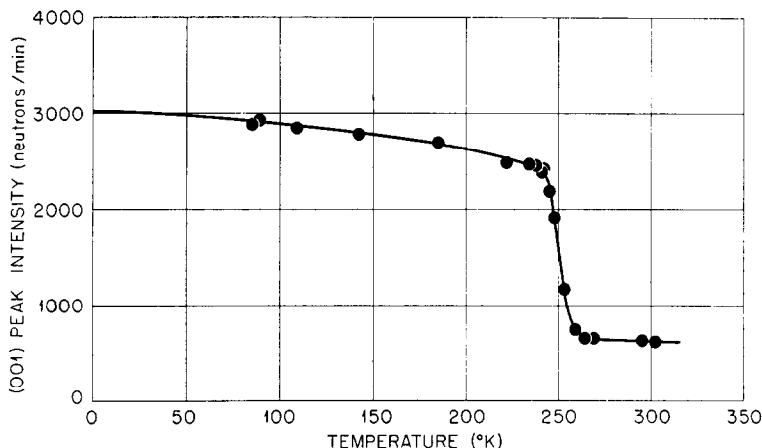

FIG. 9. Variation of the (001) peak intensity from Mn₂Sb with temperature. The absolute values of the temperature measurements are accurate only for the determinations near the two ends of the curve.

ferrimagnetism, these might be different. This determination has been made experimentally by measurements of the change in intensity of the (001) and (002) reflections between room temperature and low temperatures. Fig. 9 shows the results which were obtained for the (001) reflection, and those for the (002) reflection were quite similar. The intensities at room temperature were

representative of only the nuclear contributions to the reflections (since $q^2 = 0$), whereas data at 78°K included the magnetic contributions at that temperature. This allowed a determination of the magnetic component of these reflections at 78°K, and a small extrapolation was made to 0°K. The magnetic component at room temperature was also known, since it had been measured in

experiments with an external magnetic field. These two experiments, therefore, gave experimentally the ratio of the magnetic intensities at 295°K to those at 0°K. It was found that the degree of magnetic saturation at room temperature for the (001) and (002) reflections was slightly but significantly different. Since these two reflections have structure amplitudes which depend differently on the magnetic scattering amplitudes, $p_{Mn(I)}$ and $p_{Mn(II)}$, this difference was a definite indication of Néel's prediction that the two magnetic sublattices do not have the same temperature dependence of spontaneous magnetization. Specifically, these results showed that $p_{Mn(I)}$ at room temperature was 84 per cent of its value at 0°K, while $p_{Mn(II)}$ was only 75 per cent of its saturation value. The saturation values of $p_{Mn(I)}$ and $p_{Mn(II)}$ were calculated on this basis, and the corresponding values of the saturation magnetic moments of the manganese atoms, with estimated limits of error, were determined to be $+2\cdot13\pm0\cdot20$ and $-3\cdot87\pm0\cdot40$ Bohr magnetons.

In addition to studying the (001) and (002) reflections at low temperatures, data were obtained for the (110) reflection at 78°K. A comparison of the (110) intensity at 78°K to that at 295°K allowed a determination of the direction of the atomic magnetic moments below 240°K. These data indicated that in the low temperature region the moments are aligned parallel to the axes of the base plane of the unit cell.

DISCUSSION

The neutron diffraction measurements made on both powdered samples and single crystals of Mn_2Sb confirm the general characteristics of the ferrimagnetic structure which was first suggested by Guillaud. The measured values of $+2\cdot13\pm0\cdot20$ and $-3\cdot87\pm0\cdot40$ Bohr magnetons obtained from the single crystal measurements for the two types of manganese atoms are smaller than those of the suggested model, but they give a net saturation moment of $0\cdot87\pm0\cdot22$ μ_B per manganese atom which agrees satisfactorily with the value of $0\cdot936$ μ_B which Guillaud measured. In all of the analyses, the values obtained for the magnetic moments represent the average effective magnetic moments located at those particular atomic positions. The neutron data could not

distinguish between definite moments at each Mn position or disordered distributions of moments giving average values representative of the two types of atoms.

Since there is no apparent reason for assigning a magnetic moment to the Sb atoms, the value of the Mn moments were calculated on the assumption of no Sb moment. The fairly small area of intersection of the linear plots in Fig. 7 is evidence that this assumption is reasonable. However, the analysis was repeated without this assumption in order to determine what value of magnetic moment at the Sb positions would be consistent with the neutron diffraction results. A least-squares analysis was performed on the data from all of the magnetic reflections from the various single crystal pillars, and moment values were determined for the three types of atoms. The values of the moments for both types of manganese atoms were unchanged within 1 per cent of the values obtained by the previous analysis, and the value of the Sb moment, with an estimated limit of error, was found to be $+0\cdot05\pm0\cdot10$ Bohr magnetons. These results are therefore indicative that the Sb atoms do not possess a magnetic moment.

In an earlier section it was shown that the paramagnetic diffuse scattering from powdered samples of Mb_2Sb at temperatures well above the Curie transition did not agree with the scattering which would be expected if the atomic magnetic moments were 3 and 5 Bohr magnetons. The calculations were repeated with the values obtained from the single crystal experiments, and Fig. 10 represents a comparison of the calculated and observed differential scattering cross-sections. The curve for 78°K shows the diffuse level with the magnetic lattice about 98 per cent ordered, and the 771°K curve shows the scattering which is believed to be representative of the true experimental paramagnetic scattering. The calculated curve was obtained by the same method used previously, and the agreement between calculated and experimental curves is within experimental accuracy. Magnetic intensities in the diffraction peaks of the powder data were also compared to those calculated with the experimental values of the moments, and although there were small discrepancies, the agreement was satisfactory. Therefore, the results from the powder diffraction
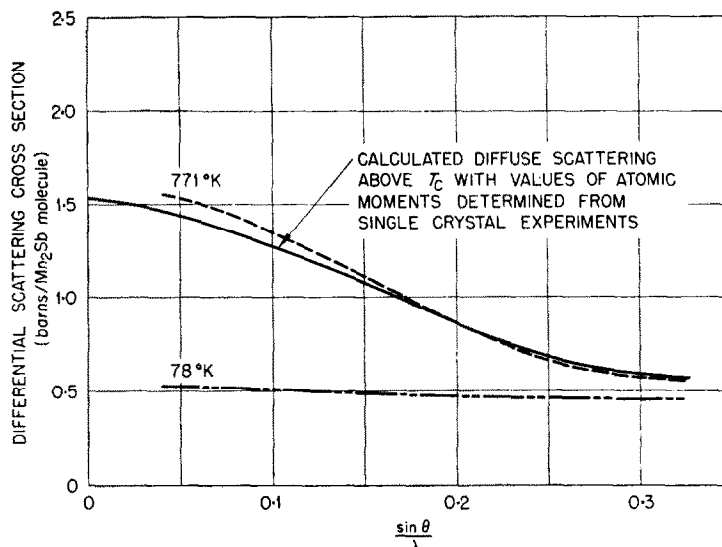
FIG. 10. Comparison of the experimental diffuse scattering from Mn₂Sb powder above T_C to that calculated for the values of atomic magnetic moments determined from single crystal measurements.

patterns are consistent with the moment values obtained from the single crystal measurements.

Since the molecular field calculations of NÉEL had so successfully accounted for the temperature variation of the inverse susceptibility and spontaneous magnetization with the moment values which GUILLAUD had suggested, it was of interest to repeat these calculations for the values determined in this investigation. The calculations were made for values of 2 and −4 Bohr magnetons,* and these moment values were found to fit the previous experimental magnetic data equally as well as the values of 3 and −5 Bohr magnetons which NÉEL has used. Hence, it appears that for this particular structure, molecular field calculations are not sensitive in selecting the values of the individual atomic magnetic moments. Furthermore, there seems to be no discrepancy between the moment values determined in this investigation and those required to explain previous experimental results. It is perhaps worth while mentioning that the temperature variation of the

spontaneous magnetization of the two sublattices as indicated in the experimental results seems to be somewhat more rapid than that which would be predicted from the molecular field calculations for either model. This difference is believed to be outside of experimental error and consequently suggests the inadequacy of the molecular field treatment when applied to this particular compound. This is not surprising since the molecular field theory is based on a Heisenberg exchange interaction between spins, and it is not clear how the presence of a small orbital contribution to the atomic magnetic moments might affect the theory. Furthermore, if there were a small Sb moment or a distribution of moments on the Mn lattices, the molecular field calculations would have to be modified.

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