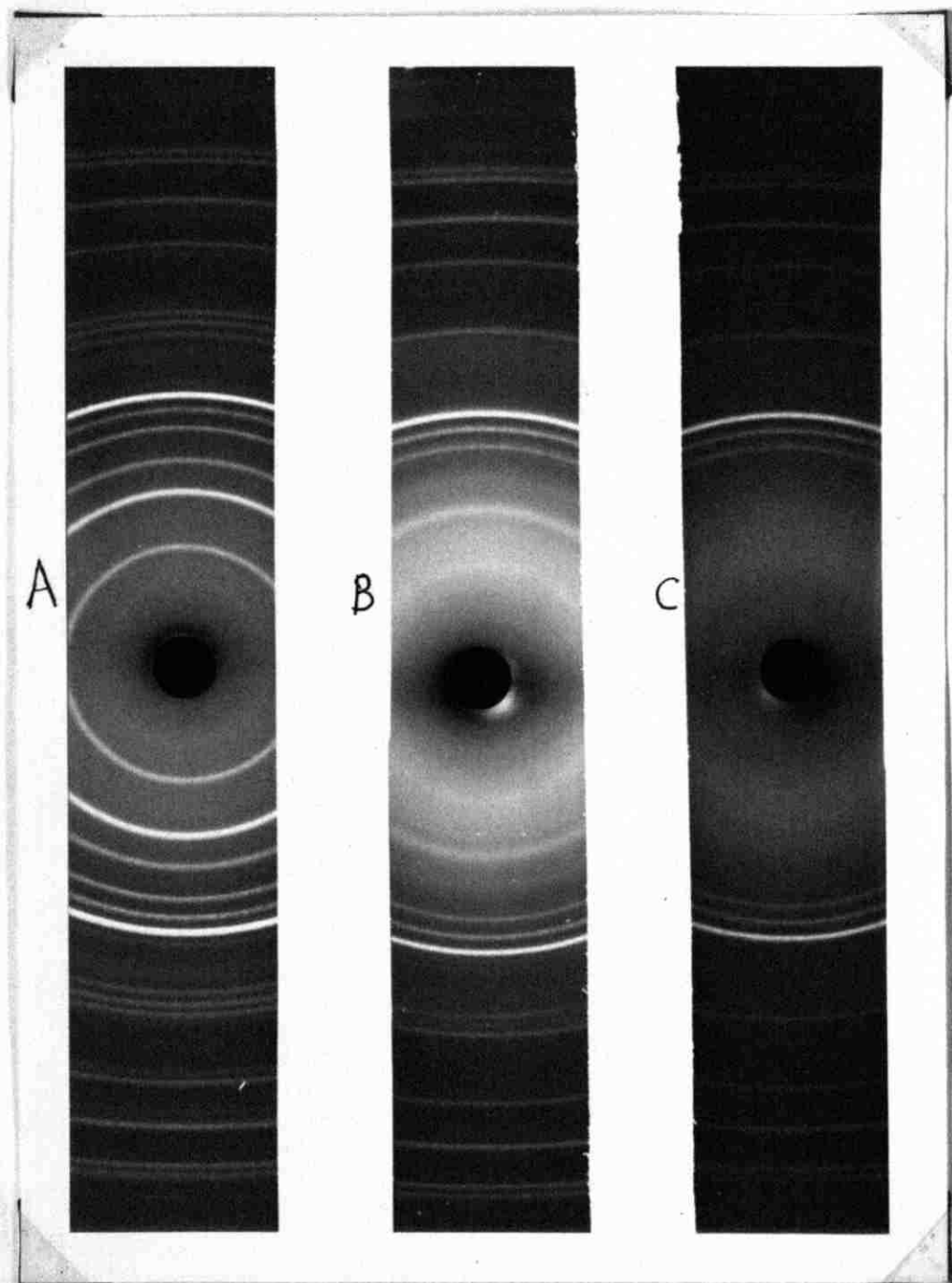Order-Disorder Transformations in $CdMg_3$
by
Sa'id Fares Mughabghab

Submitted in partial fulfillment for the requirements
of the degree Master of Science
in the Physics Department of the
American University of Beirut

Beirut, Lebanon

1959

Frontispiece: A: Perfectly Ordered
B: Partially Ordered
C: Completely Disordered

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Order in Alloys

Mughabghab

Abstract

The measurement of long range order by x-ray methods is described. The fundamental and superlattice reflections of a $CdMg_3$ alloy are determined both experimentally and theoretically. The ordering transformation of the alloy is studied. It is observed that ordering takes place even at room temperature. Ordering at $70^{\circ}C$ is investigated. The order of reaction at this temperature was found to be 3 and the rate constant was determined within the order of 1 min^{-1}.

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INTRODUCTION

Chemical and physical evidences indicate that the atoms constituting an alloy arrange themselves in regular patterns just in the same way that sodium and chlorine ions arrange themselves in a crystal lattice. One of these evidences for the existence of order in an alloy is the appearance of the superstructure lines on an X-ray photographic film. To explain this, consider a two dimensional lattice occupied by two types, A and B, of atoms. As shown in Fig. 1, there are sites that are occupied by A atoms and others by B atoms. These are called the A sites and the B sites respectively. An A atom on an A site is described as a right atom. On the other hand, an A atom on a B site is said to be a wrong atom. A similar nomenclature applies to the B atoms. All throughout our subsequent discussion it will be assumed that the atomic scattering factors of the two types of atoms are different. Now examine what might happen if an X-ray beam is incident on the scattering centers at such an angle that the path length difference between the two reflected rays is half a wave length difference. Two limiting cases will merit special atttention.

1. The Perfectly Ordered Alloy.

By this is meant that all the A atoms are on A sites and all the B atoms are on B sites. Under such a circumstance, the two layers of atoms will scatter the radiation to varying amounts; and, in consequence, partial interference will result. Hence, an X-ray reflection will appear on the photograph.

11. The Completely Disordered Alloy.

On the other hand, suppose that the alloy is completely disordered. Under such conditions, the average scattering factors of the two sites are equal. Accordingly, the two layers will scatter the radiation equally well. This necessitates that there will be complete destructive interference. From these consideration, it is seen that when there is complete disorder, cer

tain reflections called superstructure reflections will dis-
appear from the photographic film. However, if the crystal
possesses perfect order these reflections will have maximum
intensity; for intermediate states of order, there will be a
gradation of intensity until at a particular temperature, de-
fined as the critical temperature, the intensity of the super-
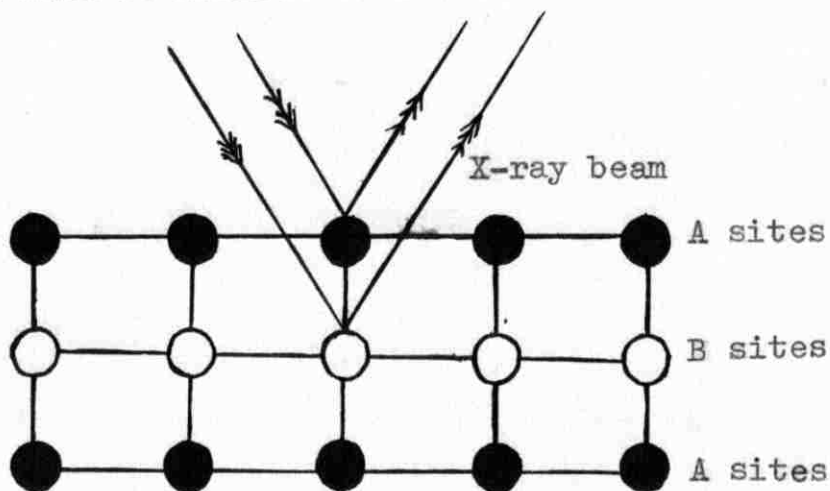lattice line will be reduced to zero.

Fig.1. - The perfectly ordered
crystal

To describe the state of order in a crystal, designate
the fraction of A atoms and of B atoms constituting an alloy by
F_A and F_B respectively. Since in our discussion, we shall ass-
ume that the atoms can be present only on the sites (and not in
the interstices) it directly follows that the fractions of A and
B sites are similarly represented by F_A and F_B respectively. In
addition, let r_A be the fraction of A sites rightly populated
by A atoms and w_A the fraction of A sites wrongly populated by
B atoms. Then clearly,

$$r_A + w_A = 1 \qquad\qquad 1$$

A corresponding relation holds for the B sites.

$$r_B + w_B = 1 \qquad\qquad 2$$

It is to be noticed that if by one means or another some of the
A atoms migrate to the B sites, they will drive out an equal num-

ber of B atoms that will in their turn occupy the A sites.
Hence, the number of wrongly occupied A sites is equal to the
number of wrongly occupied B sites. If we represent the total
number of atoms (also the total number of sites) by N, then
one can write the above statement as,

$$(NF_A)w_A = (NF_B)w_B$$

$$\text{Or,} \quad F_A w_A = F_B w_B \qquad\qquad 3$$

Let us apply this result to the case of an AB_3 alloy. Here,
we have $F_A = \frac{1}{4}$ and $F_B = \frac{3}{4}$. The substitution of these values
in the above expression will yield

$$w_A = 3w_B \qquad\qquad 4$$

And with the aid of Eqs. 1 and 2, one can get

$$r_A = 3r_B - 2 \qquad\qquad 5$$

Notice carefully that for the perfectly ordered alloy
we have,

for the A sites: $\quad r_A = 1, \quad w_A = 0$

for the B sites: $\quad r_B = 1, \quad w_B = 0$

But how can we describe the completely disordered state? Un-
der these conditions, any type of atom does not have a prefer-
to remain on any particular site. The probability of finding
an A (or B) atom on an A or B site is the same and is equal to
its atomic fraction. That is,

for A atoms: $\quad r_A = w_B = F_A$

for B atoms: $\quad r_B = w_A = F_B$

Bragg and Williams defined the long range order parame-
ter S in a such manner that it will take a value of unity for
perfect order, zero for complete disorder, and intermediate
values for various degrees of order. In the light of what has
been stated, the parameter S can subsequently be written down

in various equivalent forms:

$$S = \frac{r_A - F_A}{1 - F_A} = \frac{r_B - F_B}{1 - F_B} = 1 - \frac{w_A}{F_A} = 1 - \frac{w_B}{F_A} \qquad 6$$

For an AB_3 alloy, such as $CdMg_3$ or $AuCu_3$, we have

$$S = \frac{1}{3}(4r_A - 1) \qquad Or \qquad S = 4r_B - 3$$

and in terms of r_A and r_B, S could be written in the convenient form that we shall use in later calculations,

$$S = r_A + r_B - 1 \qquad 7$$

The Kinetics of Order Disorder Transformations

The formation of an ordered structure in an alloy is associated with the tendency of atoms to be surrounded by nearest unlike neighbors. This could be accounted for by the fact that such a configuration possesses minimum energy. To begin with, let us assume that we have a perfectly ordered, two-dimentional crystal (Fig.2.). Suppose now that the temperature of the crystal is increased. Kinetic theory tells us that the atoms will start vibrating more vigorously about their equilibrium positions. Furthermore, if the temperature is increased still more, the atoms may acquire enough energy of vibration to break away from their right sites and interchange places with the unlike neighboring atoms. Such a process will introduce disorder into the crystal. The natural question to ask now is: On what the rate of disorder depends? To answer the question, refer to Fig.2. First, let us assume that process No.1 and No.2 will occur. After each interchange, atom A is surrounded by three like nearest neighbors. Such an interchange requires a certain amount of energy. Next, let process No.3 follow. It is clear that atom A is now surrounded by two like nearest neighbors. And the energy requisite for an interchange is less than that of either process No.1 or process No.2. Hence, at higher temperatures there is not only greater degree of disorder but also greater . . . .

ease of disorder. Such a process could be conveniently described
by the following expression,

$$\frac{dD}{dt} = f(D) \qquad\qquad 8$$

where $D = 1-S$ is the disorder existing in the crystal and $f(D)$
is a function of D which is to be determined experimentally.
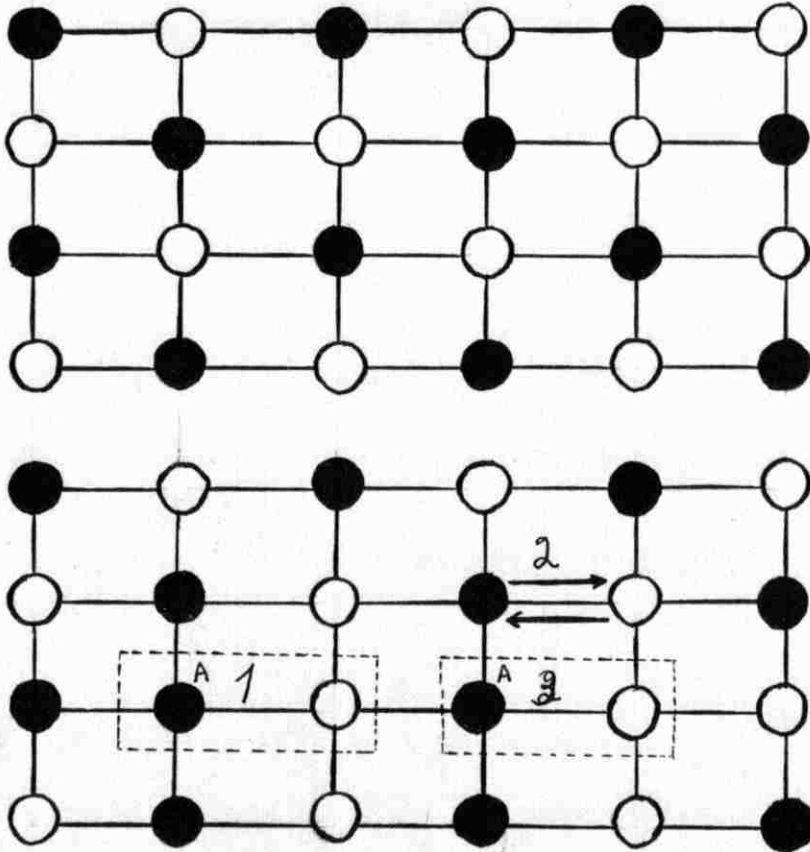
Fig.2. - Disorder in a crystal.

Several investigators searched for the dependence of the time
rate of disorder on the disorder. Among these, G.J. Diens (1)
showed that for the AuCu alloy, the following equation is sa-
tisfied,

$$\frac{ds}{dt} = K(1-S)^n \qquad\qquad 9$$

where n is determined to be 3 and K, the rate constant, is found
to be dependent on the obsolute temperature according to:

$$K = 4.46 \times 10^{10} \qquad e^{-\frac{14300}{T}} \qquad\qquad 10$$

It will be worthwhile to test the above relations to other types
of alloys.

The X Ray Method For the Measurement of S.

In the following discussion we will show how to determine
the long range order parameter by the use of the X-ray techniques.
Standard textbooks (2) on X-rays give the integrated intensity per
unit length of diffraction line for a powdered sample by,

$$I.E. = C \, m(L.P.)F^2 \, A(\theta) \, T(\theta) \qquad\qquad 11$$

where:

C is a constant for all reflections and which depends, among other
things, on the wavelength.

M is the number of planes that contribute to the same reflection.
It is known as the multiplicity factor.

L.P. is the Lorentz and polarization factor =

$$\frac{1 + \cos^2 2\theta}{\sin^2 \theta \, \cos\theta} \quad \text{for the Debye Sherrer method}$$

$A(\theta)$ is the absorption factor

$T(\theta)$ is the temperature factor

F is the structure factor of the crystal which
is given by the following general equation

$$F_{hkl} = \sum_{x,y,z} \sum_{n} f_n e^{2\pi i (h x + ky + lz)} \qquad\qquad 12$$

where h,k,l are the miller indices specifying the scattering
planes; x,y,z denotes the position coordinates of the atoms;
and finally fn is the atomic scattering factors of the atoms cons-
tituting the crystal. If the crystal possesses a center of symm-
etry such that for each atom at (x,y,z,) there is a corresponding
atom at (-x, -y, -z), then the sine terms will vanish. If this
is the case, the structure factor is simplified to,

$$F_{hkl} = \sum \sum f_n \cos 2\pi (h x + ky + lz) \qquad\qquad 13$$

Let us calculate the structure factor for $CdMg_3$. The coordinates
of the atoms of this alloy (3) are given by

2 Cd atoms at: $\pm(\frac{1}{3}, \frac{2}{3}, \frac{1}{4})$ Let these be referred

to as the A sites

6Mg atoms at: $\pm(2X, X, \frac{1}{4})$, $\pm(\bar{X}, X, \frac{1}{4})$ and $\pm(\bar{X}, 2X, \frac{1}{4})$ with

$$X = \frac{1}{6}$$

Let these be the sites.

Since such a type of crystal possesses a symmetry center, then Eq.13 is applicable. Represent the atomic scattering factors of the Cd and Mg atoms by f_A and f_B respectively. The scattering factor of the crystal when it is in any ordered (or disordered) state is

F = Scattering factor of A atoms on rightly occupied A sites + scattering factor of A atoms on wrongly occupied B sites + scattering factor of B atoms on rightly occupied B sites + scattering factor of B atoms on wrongly occupied A sites. In terms of our symbolism,

$$F_{hkl} = 2f_A r_A \cos 2\pi \left(\frac{h+2k}{3} + \frac{1}{4} \right)$$

$$+ 2f_A w_B \left[\cos 2\pi \left(\frac{2h+k}{6} + \frac{1}{4} \right) + \cos 2\pi \left(\frac{-h+k}{6} + \frac{1}{4} \right) \right. $$
$$\left. + \cos 2\pi \left(\frac{-h+2k}{6} + \frac{1}{4} \right) \right] + 2f_B r_B \left[\cos 2\pi \left(\frac{2h+k}{6} + \frac{1}{4} \right) \right.$$
$$\left. + \cos 2\pi \left(\frac{-h+k}{6} + \frac{1}{4} \right) \right] + \cos 2\pi \left(\frac{-h+2k}{6} + \frac{1}{4} \right)$$
$$+ 2f_B w_A \cos 2\pi \left(\frac{h+2k}{3} + \frac{l}{4} \right)$$

with the aid of Eqs. 1 and 2, this can be rearranged in the form.

$$F_{hkl} = 2 \left[f_A r_A + f_B(1-r_A) \right] \cos 2\pi \left(\frac{h+2k}{3} + \frac{1}{4} \right)$$

$$+ 2 \left[f_A(1-r_A) + f_B r_B \right] \left[\cos 2\pi \left(\frac{2h+k}{6} + \frac{1}{4} \right) \right.$$
$$\left. + \cos 2\pi \left(\frac{-h+k}{6} + \frac{1}{4} \right) + \cos 2\pi \left(\frac{-h+2k}{6} + \frac{1}{4} \right) \right] \qquad 14$$

An inspection of this equation reveals that F = 0 if h+2k (or h-k) = 3n and l is odd. As a result, there will be no reflections from planes with such miller indices. For example, the 001, 111, 003, 221, etc. reflections will not be present. As an application of the above formula, let us calculate F_{001}. Substituting these values of (h,k,l) in the above, one obtains,

$$F_{100} = (r_A + r_B - 1)(f_B - f_A)$$

But from Eq. 7, $S = r_A + r_B - 1$. Hence,

$$F_{100} = S (f_B - f_A)$$

Clearly, such a line depends on the order existing in the crystal, through the S term and will vanish as S approaches zero. On the other hand, a similar calculation for the 200 line will give

$$F_{200} = (f_A + 3f_B)$$

The integrated intensity of such a line does not depend in any manner on the order existing in the crystal. Lines similar to this are called fundamental lines (F lines). The values of the structure factors for other reflections are included in Table 1.

The structure of $CdMg_3$ is closed packed hexagonal (c.p.h.) with the following fimensions for the unit cell(4): a = b = 6.26, c = 5.07 and hence a/c = 1.24. The interplanar spacing d between hkl planes in a c.p.h. crystal is

$$d = \frac{a_0}{\sqrt{\frac{4}{3}(h^2 + hk + k^2) + (\frac{a}{c}l)^2}}$$

The Bragg rule states that $2d \sin \Theta = n\lambda$ and hence for reflections of the first order,

$$\sin \Theta = \frac{\lambda}{2a_0} \sqrt{\frac{4}{3}(h^2 + hk + k^2) + (\frac{a}{c}l)^2}$$

Substituting the values of a, b, c, and (Cu radiation of wavelength 1.54 angstroms is used) one gets,

$$\sin \Theta = \frac{1.54}{2 \times 6.26} \sqrt{\frac{4}{3}(h^2 + hk + k^2) + (1.241)^2} \qquad 15$$

By allowing the miller indices to assume their permissible values, the corresponding angles are calculated. (Refer to Table 1.)

Applying Eq. 11 to an F and an S line and solving for S^2, one gets,

$$S^2 = \frac{(I.E.)ss}{(I.E)F} \frac{\left[m(L.P.)F^2 A(\Theta) T(\Theta) \right]_F}{\left[m(L.P)F^2 A(\Theta) T(\Theta) \right]_{ss}}$$

Now, the area under the peak of an intensity trace is proportional to the integrated intensity:

$$\frac{(I.E.)ss}{(I.E.)F} = \frac{A_{ss}}{A_F}$$

Table 1

Indices	Calculated angles	Structure factor	Multiplicity
100	$8°10'$	$S(f_B-f_A)$	2
011	$12° 0'$	$\sqrt{3}S(f_B-f_A)$	12
110	$14° 16'$	$2S(f_B-f_A)$	6
200	$16° 30'$	$f_A + 3f_B$	6
002	$17°40'$	$2(f_A+3f_B)$	2
201	$18° 48'$	$\sqrt{3}(f_A+3f_B)$	12

Furthermore, we shall make the assumption that the temperature and absorption factors are the same for two near reflections. The reasonableness of such an assumption will be tested experimentally later on. In view of these considerations, the final expression for S^2 is reduced to

$$S^2 = \frac{A_{ss}}{A_F} \frac{\left[m(L.P.)F^2\right].F}{\left[m(L.P.)F^2\right]_{s.s.}}$$

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Intensity Calibration Curve For Kodak Industrial X ray Film, Type KK.

Our first problem is to search for a relationship between the density of blackening on a photographic film and the exposure E defined by

$$E = It$$

17

where I is the beam intensity and t is the exposure time. In our case, t is the same for all reflections. Consequently, the den-

sity is varying because of a change of the intensity. However, an examination reveals that a change of I keeping t constant amounts to the same thing as a variation of t keeping I constant. Accordingly, the problem is reduced to the finding of a relationship between density and exposure time. To accomplish such a result, the experimental arrangement is shown schematically in Fig.3. An aluminum absorber **A** of absorption coefficient 1265 mg/cm^2 is utilized to cut down the intensity of the X-rays. Such a procedure will enable one to obtain exposures of appreciable length. In front of the absorber is suspended a copper shutter of thickness 6mm that can be operated manually. To obtain uniform intensity, the film wrapped in black paper, is supported behind an aluminum absorber at a distance of 27cm from the X-ray window.
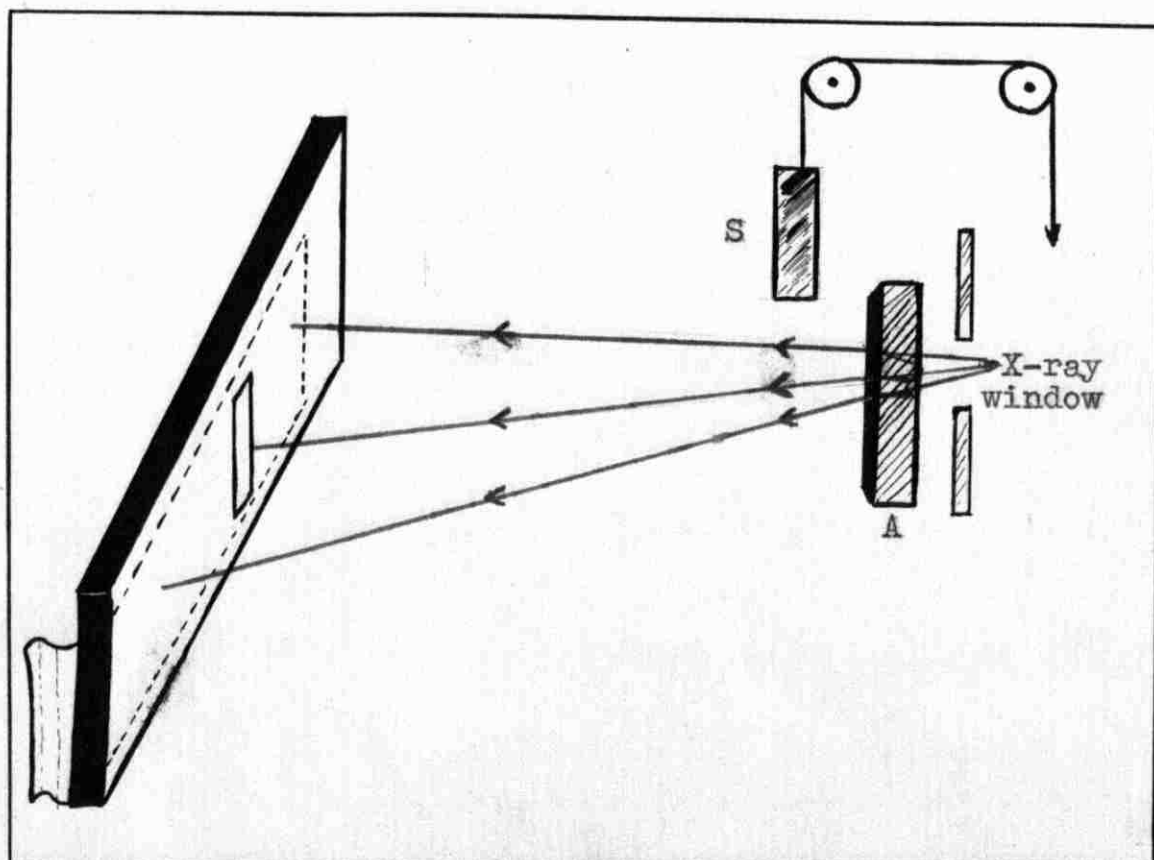

Fig.3. - Experimental set-up.

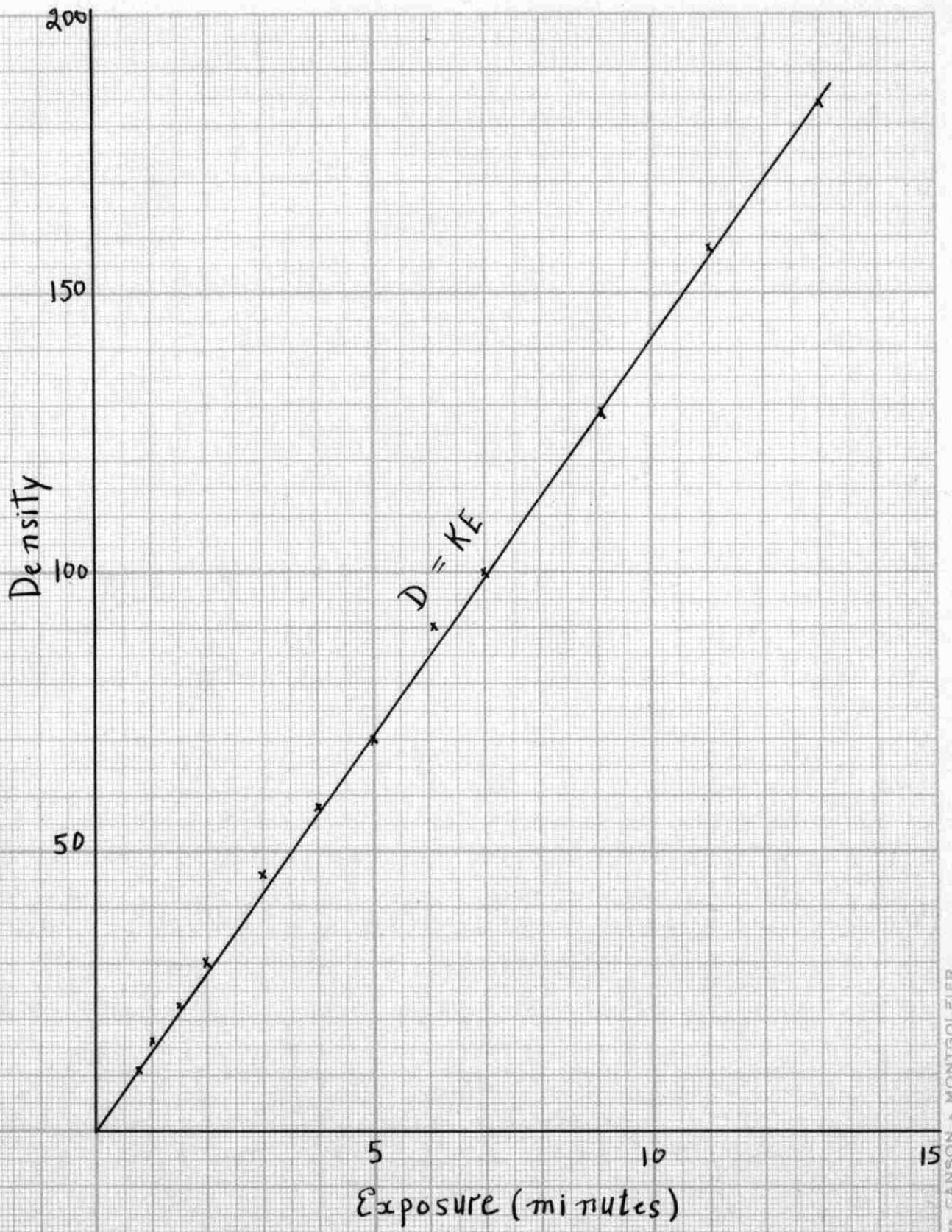Unresolved Cu K radiation passing through nickel filters falls

Fig. 4 – Graph of Density Versus Exposure.

on a rectangular slit, whose shape is similar to the profile of
the reflections on the film. In order to keep the voltage as
constant as possible a Sola Constant Voltage Transformer is emp-
loyed. The values of the exposures and the corresponding densi-

Table 2

Exposure minutes	Density
.75	11
1.0	16
1.50	22
2.00	30
3.00	46
4.00	58

Exposure minutes	Density
5.00	70
6.05	90
7.00	100
9.10	129
11.00	158
13.00	184

ties (above background) at 31kv and 31ma are listed in Table 2.
These values are plotted in Fig. 4 which shows that there is a
linear relationship between density and exposure (5). Such a
result is convenient since the ratio of the integrated intensi-
ties of two lines will then be equal to their integrated densi-
ties. That is,

$$\frac{\int_{hkl} D dx}{\int_{h'k'l'} D dx} \qquad \frac{\int_{hkl} KE dx}{\int_{h'k'l'} KE dx} \qquad \frac{\int_{hkl} E dx}{\int_{h'k'l'} E dx}$$

Study on Cu$_3$Au Alloy

A sample of Cu$_3$Au in the form of a wire is brought to
a high degree of order by the following heat-treatment: 46hrs.
at 346°C, 87hrs. at 332°C, 70hrs. at 324°C, and 70hrs. at 300°C.

Then the sample was cooled slowly in 11 hours. The S lines
appeared but were very faint. This could be accounted for by
the fact that a great deal of absorption of the Cu radiation
has taken place in the sample. As a result, long exposure
times are required or else a more intense radiation, such as
from a molybdenum source should be employed. However, the first
alternative will introduce complications because the density of
the F lines will then be greater than 2, in which case we cannot
have a microdensitometer trace of them. Such a situation forced
us to abandon work on the Cu_3Au alloy and to choose the $CdMg_3$
alloy as the next sample for our investigations. This was promp-
ted by the fact that little work was done on such an alloy.

Study on $CdMg_3$ Alloy

A powdered sample of $CdMg_3$ is centered in a Debye-Sherrer
camera of internal diameter 114.83mm. Such a choice of diameter
ensures that 2mm measured on the film will be equivalent to
1^oBragg, allowance being made for average film shrinkage. Dis-
tances on the film are measured to withing .005mm by determining
the separation of the reflection peaks with the aid of a micro-
photometer furnished with a vernier scale. The following angles
are calculated for the first six reflections: $8^o16'$, $12^o8'$,
$14^o23'$, $16^o37'$, $17^o52'$, $18^o48'$. To find out which reflections
are of the F or S type, a sample of $CdMg_3$ is heated to above its
critical temperature, 168^oC, (6) and subsequently quenched in wa-
ter at 0^oC. It is observed that the first three reflections on
the film disappeared indicating their S nature. On the other
hand, the next three reflections persisted showing that they
are F reflections. Such a result is consistent with information
obtained from the structure factors. (Refer to Table 3).

It is our purpose now to obtain the numerical values of
the structure factors and the Lorentz-polarization factors. First-
ly, plots of the Thomas-Fermi scattering factors for the cadmium
and the magnesium atoms as a function of $\sin\theta/\lambda$ are drawn. With

the aid of these, along with the values of F from Table 1, the structure factors for the $CdMg_3$ crystal for various reflections are determined. Secondly, the Lorentz-polarization factors for the Debye-Sherrer method are calculated by the equation

$$L.P. = \frac{1 + \cos^2 2\theta}{\sin^2 \theta \cos \theta}$$

The numerical values are included in Table 3.

For the evaluation of S, the 201 fundamental and the 011 superstructure reflections are chosen. The reason for such a choice is that these reflections are the most intense. Under such conditions, the expression for S^2 is reduced to the simple form,

$$S^2 = 1.53 \frac{A_{SS}}{A_F}$$

Before proceeding to study the kinetics of order disorder transformations, we have to subject the above equation to experimental test. Such a procedure is especially important in view of the assumptions made regarding the temperature and the absorption factors. To that effect, a sample is brought to a high degree of order. On calculating the value of S, it is found to be 1.0. Furthermore, the integrated intensities (also integrated densities) of the 201, and 200 fundamental reflections are compared both experimentally and theoretically. The results are quite satisfactory. Theory predicted $A_{201}/A_{200} = 4.3$ while experiment yielded a value of 4.2.

Table 3

Indices	Angles	$\frac{\sin\theta \times 10^8}{\lambda}$	F_{Mg}	F_{Cd}	F	L.P.
100	$8^\circ 6'$	.0921	10.1	44.0	33.9S	97.6
011	$12^\circ 0'$	.135	9.5	41.2	3x31.7S	43.4
110	$14^\circ 8'$	.159	9.1	40.0	61.8S	31.1
200	$16^\circ 23'$	.184	8.8	33.4	64.8	22.4
002	$17^\circ 42'$	.197	8.6	37.8	2x63.6	18.9
201	$18^\circ 42'$	.208	8.5	37.0	3x62.5	16.8

Preparation of the CdMg$_3$ Sample.

An ingot of CdMg$_3$, atomic composition 25% cadmium and
75% magnesium, is obtained from Johnson Matthey & Co. The sam-
ple is powdered with a file in the atmosphere and then passed
through 100 mesh screen. It is to be pointed out that if more
accurate work is desired, such a process must be accomplished
in an inert atmosphere to eliminate possible oxidation of the
sample. The powder is then stacked uniformly in Lindmann-glass
capillaries of internal diameter.5mm. After such a preliminary
preparation, similar samples are enclosed in pyrex tubes which
are evacuated to a pressure of 20-40 microns and then scaled off.
To remove the strains produced in the crystal during filing, it
is important that the sample be annealed above its critical tem-
perature.

Ordering at 25°C.

At the outset it is necessary to study the ordering process at room temperature (25°C). Accordingly, a sample is heated for 20 minutes at 200°C and then quenched in icy water. An X-ray photograph of the sample showed that the S reflections are not present. The same sample was left at room temperature for 44 hours. Under such a condition, the 011 S reflection started to appear; however, it is faint in comparison to the F reflections to allow precise experimental measure of S. The determined value of S is .60. This same sample is left for an additional 26 hours (70 hours from the time of quench.) The 011 reflection is becoming stronger in intensity. The calculated value of S is now .67. In addition, the sample is kept for 93 hours from the time of quench. Such an annealing time yielded a value of S = .72. Finally, the sample was left for about 30 days in which case it attained a value of S = .85.

Ordering at 70°C

A similar study carried out at 40°C indicated that the rate of ordering is slow. Furthermore, it is observed that at 70°C the rate of ordering starts to increase rapidly with time. Samples which are similarly prepared are completely disordered at 200°C after which they are quenched in water at 0°C to retain the disordered state. Each sample is annealed according to the time listed in Table 4. The experimental values of S are shown also in the same table. It is important to carry out two calculations of S, one from the upper reflections and another from the lower reflections. The value of S indicative of long range order is taken to be the average of the above two values.

Discussion of Results.

An examination of Table 4 shows the general increase of S with annealing time at constant temperature. Now it is interesting to search for the order of reaction n in Eq. 9. On the basis of the assumption that n = 3, an integration of Eq.9 with the condition that at time t = 0, S = 0, gives

$$\frac{1}{(1-S)^2} - 1 = 2Kt$$

when a graph of $1/(1-S)^2$ versus t is drawn, a straight line is obtained showing that n = 3 holds within the experimental error. From the slope of the straight line the **rate** constant K at $70^{\circ}C$ is determined to be of the order of $1\min^{-1}$.

It is to be pointed out that no research workers investigated the kinetics in the range from S = 0 to S = 1. Most of

Table 4

Sample	Annealing time (minutes)	S Upper Reflections	S Lower Reflections	Average S
1	5	.38	.37	.38
2	10	.56	.57	.57
3	15	.57	.55	.56
4	20	.64	—	.64
5	30	.79	.83	.81
6	60	.87	.86	.86
7	180	.86	—	.86
8	360	.98	.89	.93
9	960	1.0	1.0	1.0

the work was concentrated in the region of S = 1. One of the reasons for such a limitation is that it is believed that the ordering process is not a homogeneous one and that it is not as simple as described above. Other difficulties encountering such a type of study are the existence of dislocations and the dependence of long range order on short range order both of which have been completely neglected in our study. With regard to the experimental determination of S, the main problem that faced us was the broad and faint nature of the super-

structure peaks and background as effected by short range order. In view of this, the determination of the integrated intensity of the S reflections was an uneasy task. Furthermore, another source of error is the assumption that the X-ray beam is monochromatic, a circumstance which is not prevailing in the experiment. All of these added together, it could be estimated that the error is of the order of 5%. An account of these limitations, the above results are to be thought of as of a semi-quantitative nature.

Acknowledgement.

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