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HOMOGENIZATION OF INGOTS

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ABSTRACT

A simple analytical technique is presented for determining adequate homogenization treatments to reduce the microsegregation present in alloy ingots. This technique employs a mathematical description of the homogenization kinetics. In addition, a simple metallographic method of checking for microsegregation is described.

## INTRODUCTION

Alloy preparation is an important, and sometimes neglected, part of any metallurgical research. A major part of a thorough alloy preparation program is an adequate homogenization procedure, designed to minimize the microsegregation in ingots and to, therefore, reduce any effect this microsegregation may have on the subsequently measured properties of the alloy. It is relatively safe to say that all alloyed ingots will have microsegregation. This microsegregation occurs because of solute redistribution during solidification.<sup>1,2</sup> It can be removed only by subsequent thermal processing, i.e., homogenization. Since homogenization is a diffusion process, the kinetics of homogenization will depend on the geometry of the initial solute distribution, on the diffusion coefficients of the various solutes, and on the time of homogenization.<sup>3</sup>

Obviously, a complete study to determine an effective homogenization procedure for a given alloy would be very complex, involving at the very least extensive microprobe studies to determine the initial solute distribution and to determine the progress of homogenization. Such detailed work is not routinely feasible.

What is needed is a simplified method for designing a homogenization procedure, i.e., determining the required time at temperature to achieve homogenization. One would like to minimize the amount of initial analysis--i.e., microprobe and metallographic studies--yet maintain sufficient accuracy in the procedure that its usefulness will not be seriously compromised. The purpose of this report is to describe a simplified procedure which meets these criteria and to illustrate its use.

## TWO-DIMENSIONAL MODEL FOR HOMOGENIZATION

In general the mathematical analysis of homogenization is very complex because it is necessary to solve the diffusion equation in two- or three-dimensions for boundary conditions which are complicated and variable. It is essential, therefore, to simplify the analysis by making approximations. The simplest approximation is to reduce the problem to one dimension, as was done for example by Lavender and Jones.<sup>4</sup> This one-dimensional analysis has proven to be very useful<sup>4-6</sup> and we shall return to it later on. It does, however, lack generality, and in the following analysis a more generally applicable two-dimensional model will be presented.

Kattamis and Flemings, in their excellent paper on the homogenization of low alloy steel,<sup>1</sup> developed a two-dimensional mathematical model for the kinetics of homogenization. This model is based on a rectangular idealization of experimentally observed dendrite morphologies; this idealization is illustrated in Fig. 1. Their analysis of this model will be reviewed in some detail because it is relevant to the subsequent discussion.

For the complete mathematical description of the homogenization of this idealized structure it is sufficient to solve the two-dimensional diffusion equation for just one quadrant of the dendrite rectangle. The general solution for this case (see Kattamis and Flemings<sup>1</sup> for details) is:

$$C(x,y,t) = \sum_{n=0}^{\infty} \sum_{m=0}^{\infty} K_{nm} \cos\left(\frac{n\pi x}{l}\right) \cos\left(\frac{m\pi y}{l'}\right) \times e^{-(n^2/(l)^2 + m^2/(l')^2) \pi^2 Dt} + \bar{C} \quad (1)$$

where  $C$  = concentration

$x, y$  = dimensional coordinates

$d$  = diffusion coefficient, and

$t$  = time.

The constants  $K_{nm}$  are evaluated by the usual Fourier series techniques.

In principal  $C(x, y, t)$  may then be determined exactly if one knows the exact initial solute distribution (i.e.,  $C(x, y, t = 0)$ ). However, since the exact initial solute distribution was not known Kattamis and Flemings adopted an approximate method for determining  $C(x, y, t)$ . This method was to divide the dendrite quadrant into nine equal areas and experimentally measure, with a microprobe, the average concentration of each area. The constants  $K_{nm}$  were then evaluated by approximating the Fourier integrals as summations, and summing over the nine equal areas. Again, the reader is referred to the original work for further details.

The analysis of homogenization kinetics as done by Kattamis and Flemings is rather complex because it is necessary to measure the initial solute distribution experimentally. Ideally, one should need to measure only the dendrite arm spacings,  $2\ell$  and  $2\ell'$  as defined in Fig. 1, in order to determine  $C(x, y, t)$ . This is possible provided one assumes an initial solute distribution  $C(x, y, 0)$ . Geometric similarity of all dendrites is also tacitly assumed.

A very simple initial solute distribution, which agrees well with the observed distribution as reported by Kattamis and Flemings, is a two-dimensional sinusoidal distribution:

$$C(x, y, 0) = \frac{C_M^0 - C_m^0}{4} \left( 1 - \cos \frac{\pi x}{\ell} \right) \left( 1 - \cos \frac{\pi y}{\ell'} \right) + C_m^0, \quad (2)$$

where  $C_M^o$  = maximum solute concentration  
 $C_m^o$  = minimum solute concentration.

A diagram of this distribution is shown in Fig. 2. Figure 3 shows a plot of isoconcentration lines as determined from Eq. (2), and gives the experimentally measured concentration at various points as measured by Kattamis and Flemings. On the basis of the agreement demonstrated in Fig. 3, Eq. (2) is a good approximation to the initial solute distribution  $C(x,y,0)$ .

With the initial solute distribution given by Eq. (2) it is now possible to solve for  $C(x,y,t)$ . The details of the solution are given in the Appendix. The solution is:

$$C(x,y,t) = \frac{C_M^o - C_m^o}{4} \left[ \cos \frac{\pi x}{\ell} \cos \frac{\pi y}{\ell'} e^{-\left(\frac{1}{\ell^2} + \frac{1}{\ell'^2}\right) \pi^2 Dt} \right. \\ \left. - \cos \frac{\pi x}{\ell} e^{-\frac{1}{\ell^2} \pi^2 Dt} - \cos \frac{\pi y}{\ell'} e^{-\frac{1}{\ell'^2} \pi^2 Dt} + 1 \right] + C_m^o \quad (3)$$

This solution gives very nearly the same homogenization kinetics as derived by Kattamis and Fleming as will be shown in the subsequent discussion.

A convenient measure of the microsegregation is the residual segregation index  $\delta$ , where

$$\delta = \frac{C_M(t) - C_m(t)}{C_M^o - C_m^o} \quad (4)$$

Substituting Eq. (3) into Eq. (4), and solving for  $C_M(\ell, \ell', t)$  and



$C_M(o,o,t)$ , it is found that

$$\delta = \frac{1}{2} \left( e^{-\frac{1}{\ell^2} \pi^2 Dt} + e^{-\frac{1}{\ell'^2} \pi^2 Dt} \right) \quad (5)$$

Equation 5 gives essentially the same results for the homogenization of Ni in 4340 as does the analysis of Kattamis and Flemings. The homogenization kinetics predicted by Eq. (5) are compared with the kinetics predicted by Kattamis and Flemings in Fig. 4, which shows the residual segregation index,  $\delta$ , as a function of homogenization time at 1200°C. The experimental points determined by Kattamis and Flemings are also plotted in Fig. 4. These points indicate that both analyses are good approximations of the homogenization kinetics.

As mentioned previously, several investigators have examined homogenization kinetics using a one-dimensional model.<sup>4-7</sup> For this model the initial solute distribution is assumed to be sinusoidal (i.e., parallel plates). The residual segregation index for this one-dimensional case is given by<sup>6</sup>

$$\delta = \exp \frac{-\pi^2 Dt}{\ell^2} \quad (6)$$

Interestingly enough, Eq. (5) reduces to Eq. (6) if  $\ell = \ell'$ .

Equation 6 has been used by Ward,<sup>4</sup> to successfully predict the homogenization kinetics of Mn in a low alloy steel. His results are shown in Fig. 5. Some investigators, however, have obtained experimental results which were not adequately predicted by Eq. (6). For example, for phosphorous Ward<sup>4</sup> found that Eq. (6) predicted homogenization times approximately 2 times longer than those actually observed.

It is, of course, not surprising that neither Eq. (6) nor Eq. (5) will always adequately describe the homogenization kinetics. The analysis for either equation is necessarily very approximate. For example, in many instances the segregation profile will not be approximated by a simple sinusoidal distribution. An example of a decidedly different profile is shown in Fig. 5. Solute spikes such as those shown in Fig. 5 were in fact suggested by Ward<sup>4</sup> to be responsible for the short homogenization times observed for phosphorous. Purdy and Kirkaldy<sup>3</sup> discuss other problems which limit the accuracy of any quantitative analysis of homogenization kinetics.

Despite the approximate nature of the analysis using either the one- or two-dimensional model, the results of Kattamis and Flemings,<sup>1</sup> Lavender and Jones<sup>4</sup> and Ward<sup>6</sup> do show that the analysis can be sufficiently accurate to be of great utility. In other words, the use of either Eq. (5) or Eq. (6) permits one to predict the homogenization kinetics in a relatively simple fashion. Equation (6) is particularly attractive in this regard since the only physical measurement necessary (provided a reliable value for the diffusion coefficient is available from the literature) is the dendrite arm spacing  $2\lambda$ .

#### METHOD FOR DESIGNING A HOMOGENIZATION PROGRAM

The foregoing provides the formal basis for a simple method of designing a homogenization procedure. In the following, this method will be described in detail, using, as an example, the homogenization of an Fe-12Ni-0.5Ti alloy (wt%).

First, a metallographic examination of the as cast ingot must be performed to determine the appropriate dendrite arm spacing. Normally, this will be easily accomplished by using a "heavy" chemical etch followed by optical examination at low magnification using either interference contrast or phase contrast techniques to reveal the surface topology. Figure 7 shows the as cast segregation revealed, using this approach, for the Fe-12Ni-0.5Ti alloy. In measuring the spacing it is important to be sure the dendrite axes are either perpendicular to, or in the plane of polish; otherwise the measured spacings may be distorted. Normally, the dendrite axes will be perpendicular to the surfaces of the ingot, i.e., the dendrites grow in the direction of the maximum thermal gradient.

In some instances the segregation may not be revealed by a simple etching technique. In such cases it will be necessary to determine the appropriate spacings by microprobe--again being careful to select the plane of examination so that the true dendrite spacing is being measured. An example of the use of this technique to determine the spacing is illustrated in Fig. 8 for the as cast Fe-12Ni-0.5Ti alloy.

The section of ingot chosen for examination is not arbitrary. The dendrite spacing is a strong function of the freezing rate (see, for example, Metals Handbook, Vol. 8, p. 159) and, therefore, varies

considerably within an ingot. The largest spacing will be found in the region which freezes at the slowest rate, which will usually be the last region to freeze--i.e., in the center of the ingot near the top. Thus, if the ingot is to be completely homogenized, one must design the homogenization procedure for the largest dendrite arm spacing found in the ingot.

To complete the analysis one must also find the appropriate value of the diffusion coefficient,  $D$ , from the literature. It is important to obtain an accurate value for  $D$ , as well as  $l$  and  $l'$  (or  $l$  alone), since  $\delta(D, l)$  is an exponential function. It should be noted that there are two different diffusion coefficients that will be found in the literature.<sup>8</sup> These are the chemical diffusion coefficient, usually indicated by  $\tilde{D}$ , and the intrinsic diffusion coefficient,  $D_i$ . The chemical  $\tilde{D}$  is the proper one to use.

Once  $D$  and the appropriate dendrite spacings are known it is a simple matter to calculate  $\delta$ , for any combination of temperatures and times. It is, of course, desirable to use the highest temperature that is practical. One must, however, be careful not to use a temperature so high that any part of the ingot may melt. In this regard it is important to use caution in selecting the material upon which one rests the ingot in the homogenizing furnace. Diffusion is sufficiently rapid at the temperatures used for homogenizing that low melting alloys may be formed quite rapidly and can badly damage the furnace in addition to destroying the ingot.

In general for the elements normally encountered in steel, with the exception of carbon (carbon homogenizes very rapidly), temperatures

below 1175°C require impractically long homogenization times. For example, Table I shows the homogenization time (according to Eq. (6)) for nickel required to reduce the microsegregation by 75% ( $\delta = 0.25$ ) for different homogenization temperatures. The dendrite spacing ( $2\ell$ ) is taken to be 200  $\mu\text{m}$  (a "normal" spacing) and  $D_{\text{Ni}}^{\text{Fe}} = 0.77 e^{-\frac{33,500}{T}}$ .

The selection of an appropriate  $\delta$  is somewhat uncertain. A good guideline would be to choose the minimum  $\delta$  it is practical to achieve, since the principle of homogenization is to minimize the effect of microsegregation on any subsequently measured property. It may also be possible to estimate the required  $\delta$  from the known effect of composition on the properties of interest, and the measured  $C_M^0$  and  $C_m^0$ . For example, the known effect of Cr on the TTT diagram might be used to estimate what value of  $C_M(t) - C_m(t) = (C_M^0 - C_m^0)$  could be tolerated.

In many instances the "rectangular" dendrite morphology encountered by Kattamis and Flemings will not be realized. In these cases Eq. (6) should be used with the appropriate spacing, determined as discussed previously. Here, the analysis may be represented very simply in terms of the dimensionless parameters  $\left(\frac{Dt}{\ell^2}\right)$ , as shown in Fig. 9, i.e.,  $\delta$  may be quickly determined, for given set of  $D$ ,  $t$  and  $\ell$ , from Fig. 9.

In view of the approximations inherent in using either Eq. (5) or Eq. (6) to determine the homogenizing kinetics it is desirable to have some simple, independent technique of checking the homogenization of an ingot. Either metallographic or microprobe examination of a homogenized ingot will generally be suitable for this purpose. As an example, consider the homogenization of the Fe-12Ni-0.25Ti alloy which has been followed by both techniques. The as cast microsegregation at the bottom of this

ingot, where the freezing was very rapid, is easily revealed by the etching technique as previously shown in Fig. 7. The etched "grooves" of Fig. 7 are shown in Fig. 10 to be regions of high titanium content. This can be determined by comparing Fig. 10a with Fig. 10b. Both figures are electron microprobe photomicrographs. Figure 10a is a topological map resulting from back-scattered electrons, while Fig. 10b is the same region viewed with an X-ray analyzer calibrated to  $TiK_{\alpha}$  radiation. The as cast microsegregation at the top of the ingot is shown in Fig. 11a, along with a microprobe X-ray scan for Ti, Fig. 11b. The Ni scan for the same area was previously shown in Fig. 8. Note that the dendrite spacing is clearly much larger at the top of the ingot.

Homogenization of specimens of this alloy was carried out at  $1175^{\circ}C$  (in vacuum  $\sim 10^{-4}$  Torr) for various times. The progress of homogenization as revealed by the etching technique is shown in Fig. 12a for the top of the ingot and in Fig. 12b for the bottom. It is clear from examination of these figures that this technique does show qualitatively the progress of homogenization, i.e., the bottom of the ingot appears to be completely homogenized after 24 hrs whereas the top of the ingot appears to be unchanged after 24 hrs. A microprobe scan of Ni was also taken for the specimen (top of the ingot) homogenized 24 hrs (the scan was taken along side the scratch shown on the micrograph). The results of this scan are shown in Fig. 13, and indicate, in agreement with the micrograph in Fig. 12, that little homogenization has taken place (see Fig. 11 for a comparison with the as cast segregation).

The expected effect of various homogenizing times at  $1175^{\circ}C$ , calculated according to Eq. (6), is shown in Table II for Ni. The

calculations are not shown for Ti since indications<sup>10</sup> are that the diffusion coefficient of Ti is approximately 100× greater than that for Ni in this temperature range. It is apparent that the calculated effect of homogenization time on the microsegregation does agree well with the qualitative results of the etching technique.

### THE EFFECT OF DEFORMATION

It is common practice to forge ingots prior to homogenization. This practice is motivated by the apparently obvious conclusion that reducing the ingot section will correspondingly reduce the segregation distance, i.e., the dendrite arm spacing,  $2\ell$ , and thereby, the homogenization time. It is difficult to believe that very heavy forging, such as severe upset plus cross forging, does not have some benefit in this regard. However, closer examination of the problem indicates that forging will not always be helpful.

Consider, for example, the simple dendrite structure depicted in Fig. 1. If an ingot with this dendritic structure were forged on a rolling mill with, say, the rolling direction along the z axis in the x-z plane, any reduction in the dendrite arm spacing in the y direction,  $\ell'$ , would be accompanied by an increase in the spacing,  $\ell$ , in the x direction. Thus, while homogenization would now occur more rapidly in the y direction it would occur correspondingly slower in the x direction. This situation is in fact reflected in Eq. (5)--i.e., as  $\ell$  becomes very large in comparison to  $\ell'$ ,

$$\delta \rightarrow \frac{1}{2} \left( e^{-\frac{\pi^2 Dt}{\ell'^2}} + 1 \right),$$

and for reasonable homogenization times  $\delta$  will not be reduced below 1/2. Any three-dimensional dendritic structure can be expected to exhibit the same effects, at least for some types of deformation.<sup>3</sup>



Thus, what seemed at first to be an obvious conclusion, namely that ingot reduction will be beneficial to homogenization, is probably not in fact always true. It is unfortunate that very little experimental work has been done on the effect of forging. However, at this point in time the best one can do is to be aware of the possible consequences of forging prior to homogenization.

#### ACKNOWLEDGEMENTS

This work was inspired by some discussions with Professor Parker and his inspiration is gratefully acknowledged. This work was supported by the U. S. Energy Research and Development Administration.

APPENDIX

The initial solute distribution is given by

$$C(x,y,0) = \frac{C_M^0 - C_m^0}{4} \left(1 - \cos \frac{\pi x}{\ell}\right) \left(1 - \cos \frac{\pi y}{\ell'}\right) + C_m^0 \quad (a)$$

The complete problem is

$$\text{D.E.} \quad \frac{\partial^2 C}{\partial x^2} + \frac{\partial^2 C}{\partial y^2} = \frac{1}{D} \frac{\partial C}{\partial t} \quad (b)$$

$$\text{B.C.} \quad x = 0, \ell \quad \frac{\partial C}{\partial x} = 0$$

$$y = 0, \ell' \quad \frac{\partial C}{\partial y} = 0$$

$$t \rightarrow \infty \quad C(x,y,t) \rightarrow \bar{C}$$

$$\text{I.C.} \quad C(x,0,0) = C_m^0$$

$$C(x,\ell',0) = (C_M^0 - C_m^0)(1/2) \left(1 - \cos \frac{\pi x}{\ell}\right) + C_m^0$$

$$C(0,y,0) = C_m^0$$

$$C(\ell,y,0) = (C_M^0 - C_m^0)(1/2) \left(1 - \cos \frac{\pi y}{\ell'}\right) + C_m^0$$

$$C(\ell,\ell',0) = C_M^0$$

The general solution is

$$C(x,y,t) = \sum_{n=0}^{\infty} \sum_{m=0}^{\infty} K_{nm} \cos \frac{\pi x}{\ell} \cos \frac{\pi y}{\ell'} e^{-\left[\left(\frac{n}{\ell}\right)^2 + \left(\frac{m}{\ell'}\right)^2\right] \pi^2 D t} + \bar{C} \quad (c)$$

The average concentration,  $\bar{C}$ , can easily be found by using the fact that  $\bar{C}$  will not change with time. Thus  $\bar{C}$  may be found from the initial solute distribution:

$$\bar{C} = \frac{1}{\ell\ell'} \int_0^{\ell} \int_0^{\ell'} C(x,y,0) dydx = \frac{C_M^0 - C_m^0}{4} + C_m^0 \quad (d)$$

Now, by applying the usual Fourier series techniques the constants  $K_{nm}$  can be evaluated:

for  $n \neq 0, m \neq 0$

$$K_{nm} = \frac{4}{\ell\ell'} \int_0^{\ell} \int_0^{\ell'} [C(x,y,0) - \bar{C}] \cos \frac{n\pi x}{\ell} \cos \frac{m\pi y}{\ell'} dydx \quad (e)$$

for  $n \neq 0, m = 0$  or  $n = 0, m \neq 0$

$$K_{nm} = \frac{2}{\ell\ell'} \int_0^{\ell} \int_0^{\ell'} [C(x,y,0) - \bar{C}] \dots dydx$$

and for  $n = 0, m = 0$

$$K_{nm} = \frac{1}{\ell\ell'} \int_0^{\ell} \int_0^{\ell'} [C(x,y,0) - \bar{C}] \dots dydx$$

Substituting  $C(x,y,0)$  and solving for each case, it is found that

$$K_{11} = \frac{C_M^0 - C_m^0}{4}$$

$$K_{10} = K_{01} = -\frac{C_M^0 - C_m^0}{4}$$

Substitution of these values back into Eq. (c) gives the final solution  $C(x,y,t)$ , i.e., Eq. (3) in the text.

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Table I. Homogenizing times of Ni required for  $\delta = 0.25$ .

Homogenization Temperature (°C)	1050	1150	1200	1300
Homogenization Time (hrs)	645	109	49	11.5

Table II. Homogenization of Ni at 1175°C.

Residual Segregation Index	Homogenization Time (hrs)				
	1	2 1/2	4	12	24
$\delta^*$ Bottom of ingot	0.68	0.37	0.2	~0	~0
	12 hr	24 hr	100 hr		
$\delta^{**}$ Top of ingot	0.80	0.68	0.20	--	--

\* $2\ell = 50\mu$  for bottom of ingot.

\*\* $2\ell = 250\mu$  for top of ingot.

FIGURE CAPTIONS

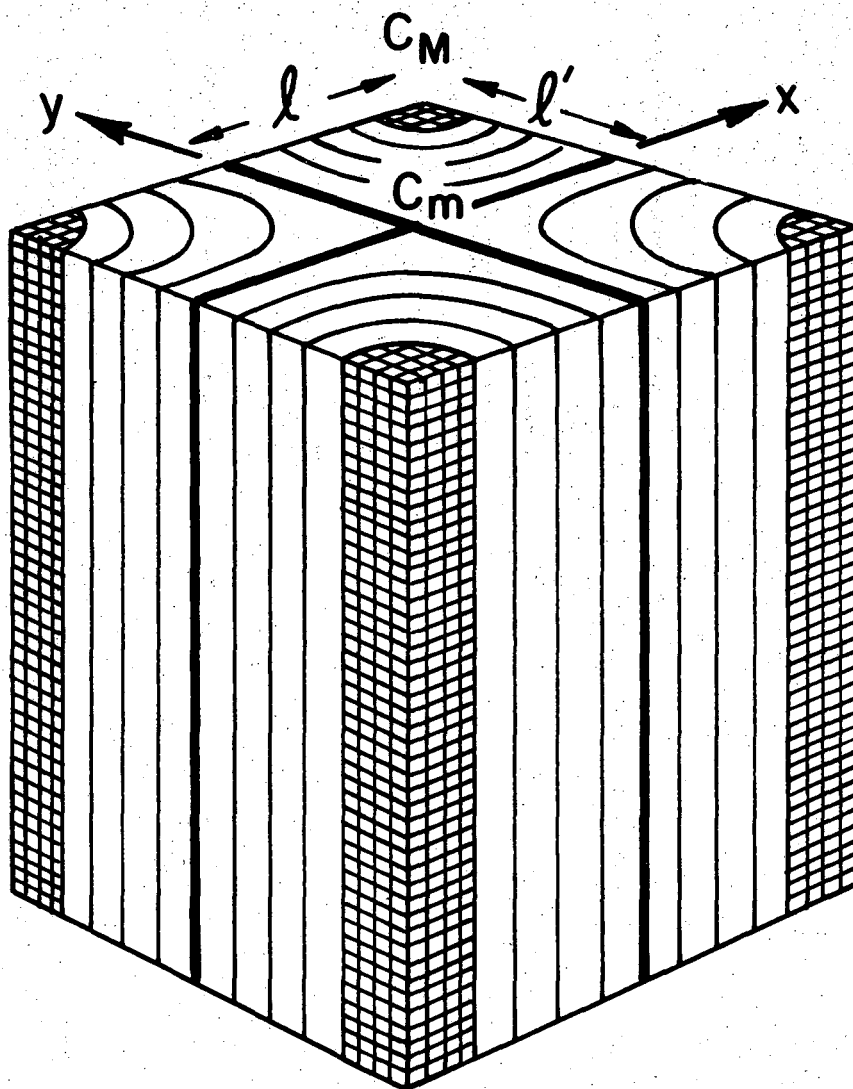
- Fig. 1. Rectangular idealization of dendrite morphology.  $C_M$  = maximum solute concentration and  $C_m$  = minimum solute concentration.  $\ell$  and  $\ell'$  are the dendrite arm spacings (after Kattamis and Flemings<sup>1</sup>).
- Fig. 2. Diagram to illustrate the appearance of the solute field according to the two-dimensional sinusoidal model.
- Fig. 3. Isoconcentration curves plotted according to Eq. (2). Experimental points determined by Kattamis and Flemings<sup>1</sup> are also plotted for comparison.
- Fig. 4. Comparison of the two-dimensional model employed herein with that used by Kattamis and Flemings<sup>1</sup> for the homogenization of Ni ( $\ell = 284 \mu\text{m}$ ,  $\ell' = 188 \mu\text{m}$ ).
- Fig. 5. The results of Ward<sup>4</sup> on the homogenization kinetics of Mn in a low alloy steel. The curves were calculated by Ward according to Eq. (6).
- Fig. 6. A microprobe scan across secondary dendrite arms in a directionally solidified, high Mn alloy. This figure illustrates a decidedly non-sinusoidal solute profile. After Hone and Purdy.<sup>11</sup>
- Fig. 7. As cast segregation as revealed by the etching technique. This sample was from the bottom of the ingot.
- Fig. 8. Microprobe scan for Ni across the dendrite arms near the top of the ingot, as cast.
- Fig. 9. The residual segregation index,  $\delta$ , as a function of the dimensionless parameter  $Dt/\ell^2$ .

Fig. 10. Microprobe photomicrographs showing that the "grooves" in Fig. 6 are regions of higher titanium content. (a) Back-scattered electrons. (b) X-ray analyzer calibrated to  $TiK_{\alpha}$  radiation.

Fig. 11. (a) As cast microsegregation at the top of the ingot. (b) Microprobe scan for Ti from the top of the as cast ingot.

Fig. 12. (a) From the top at the ingot homogenized 24 hr at  $1175^{\circ}C$ . The microsegregation does not appear to have been much reduced by the homogenization treatment. (b) From the bottom of the ingot, homogenized for (1) 2-1/2, (2) 12-1/2 and (3) 24 hr. The microsegregation appears to be completely eliminated at 24 hr and almost eliminated at 12-1/2 hr.

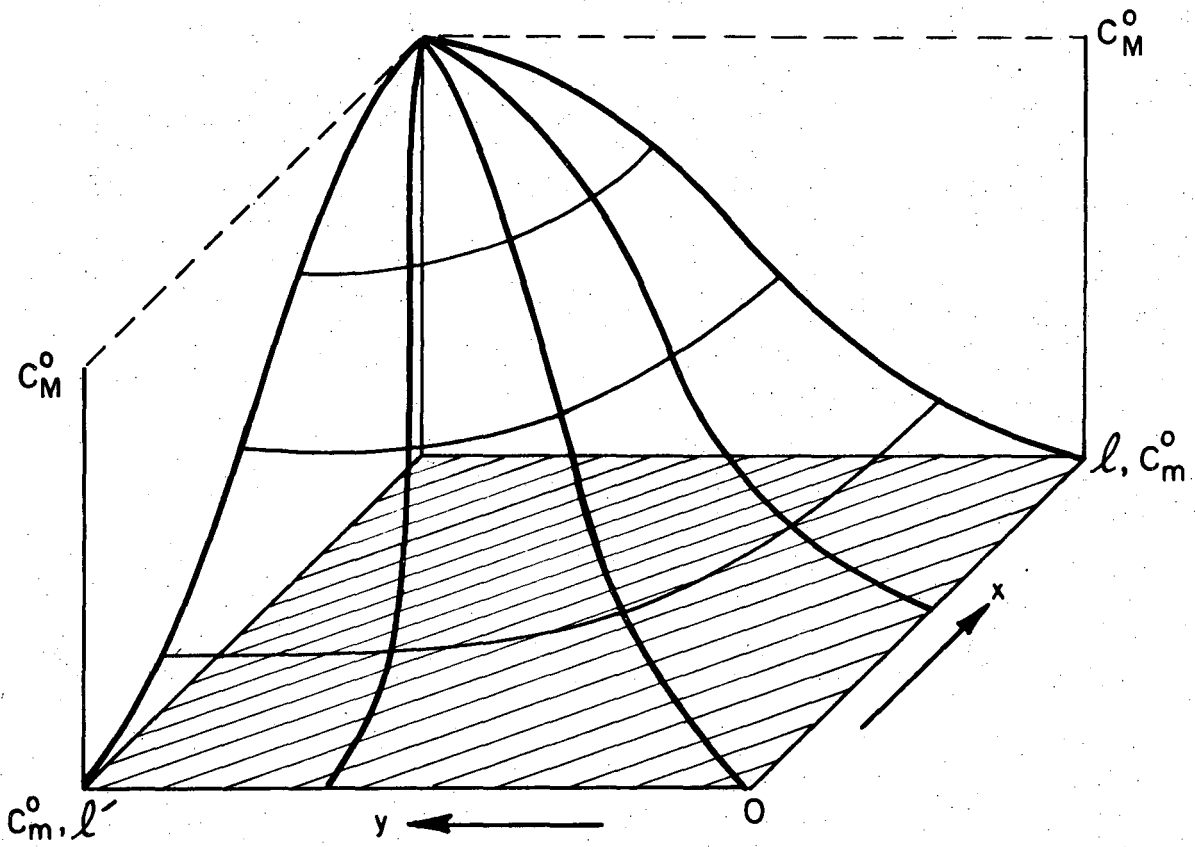
Fig. 13. Microprobe scan of top of ingot after 24 hr at  $1175^{\circ}C$ . This scan was taken along scratch shown in Fig. 12a, and shows that little homogenization has taken place.



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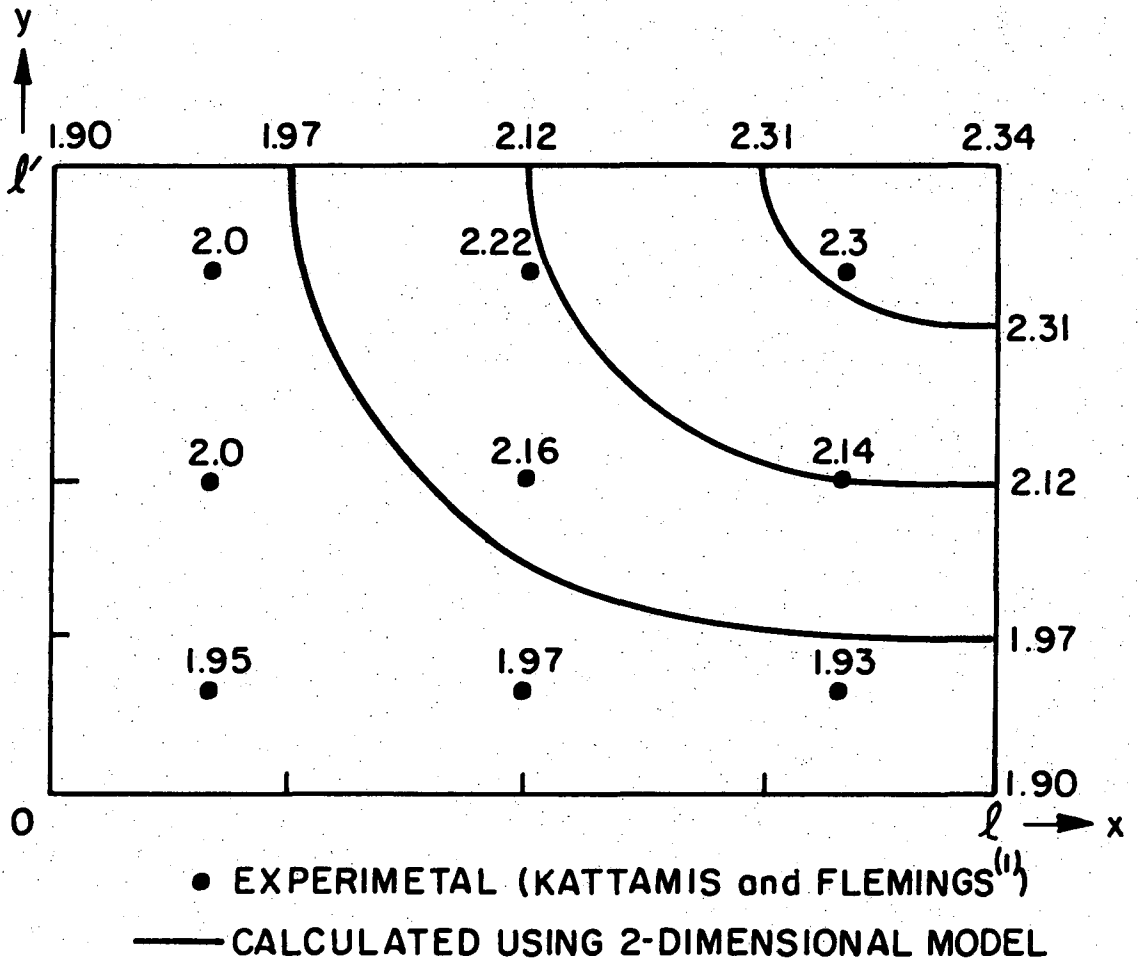
Fig. 1





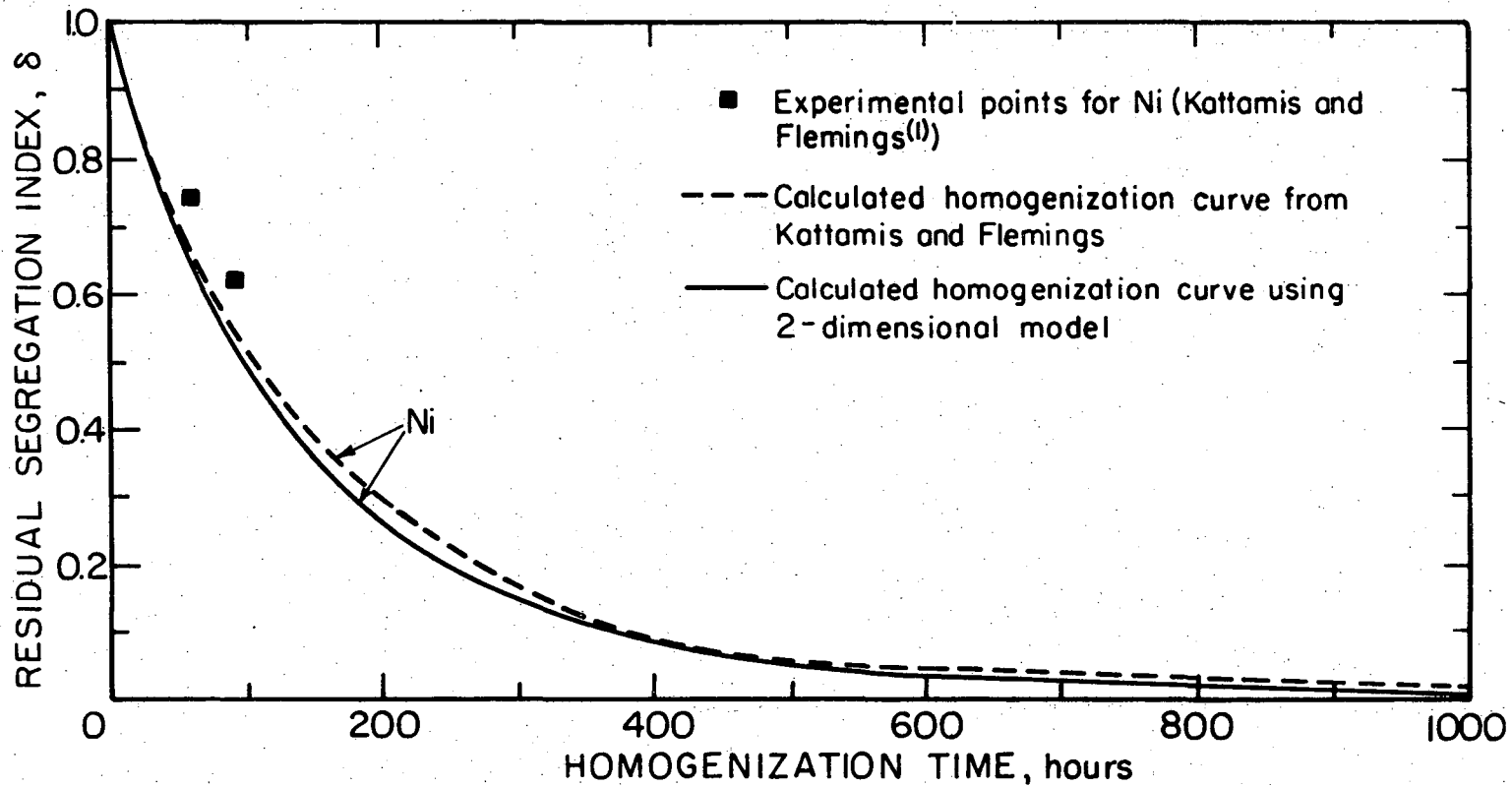
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Fig. 2



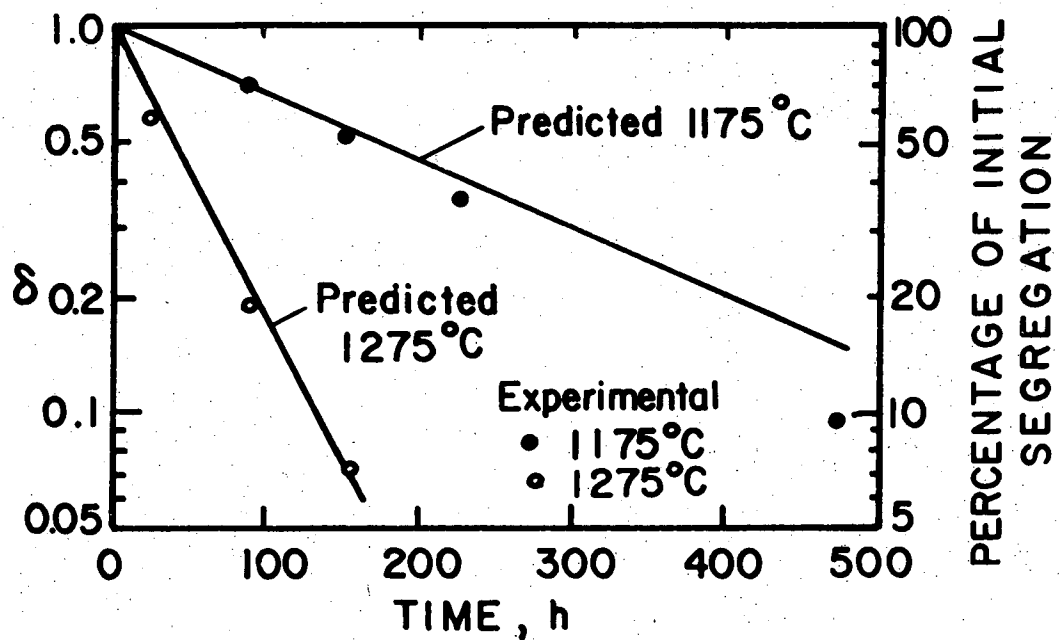
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Fig. 3



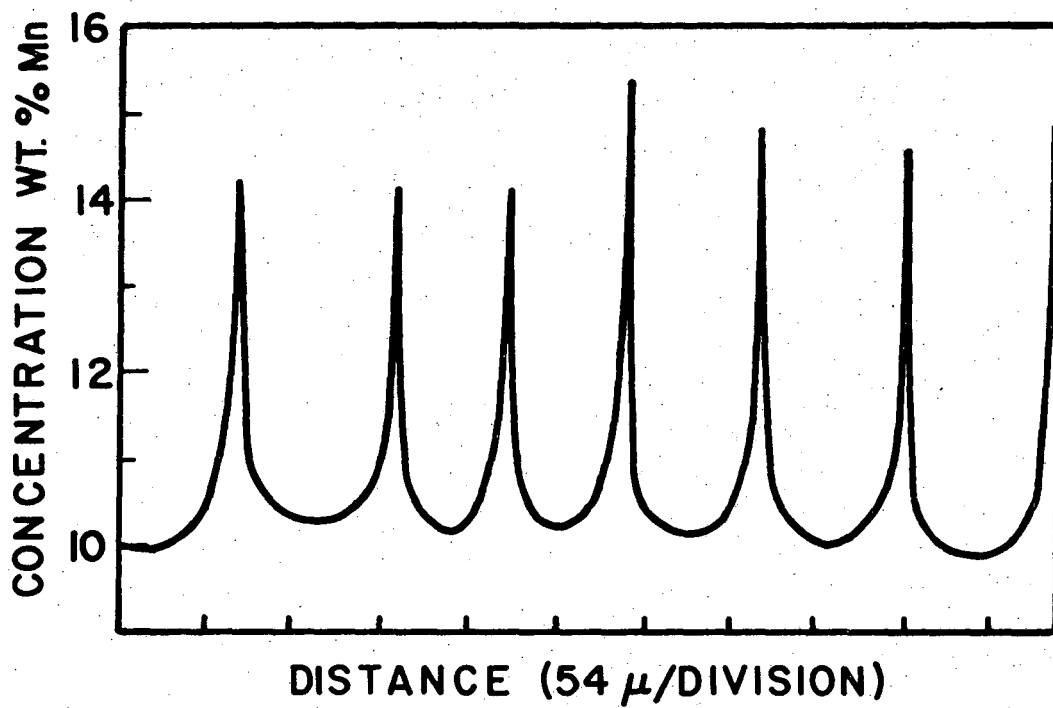
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Fig. 4



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Fig. 5



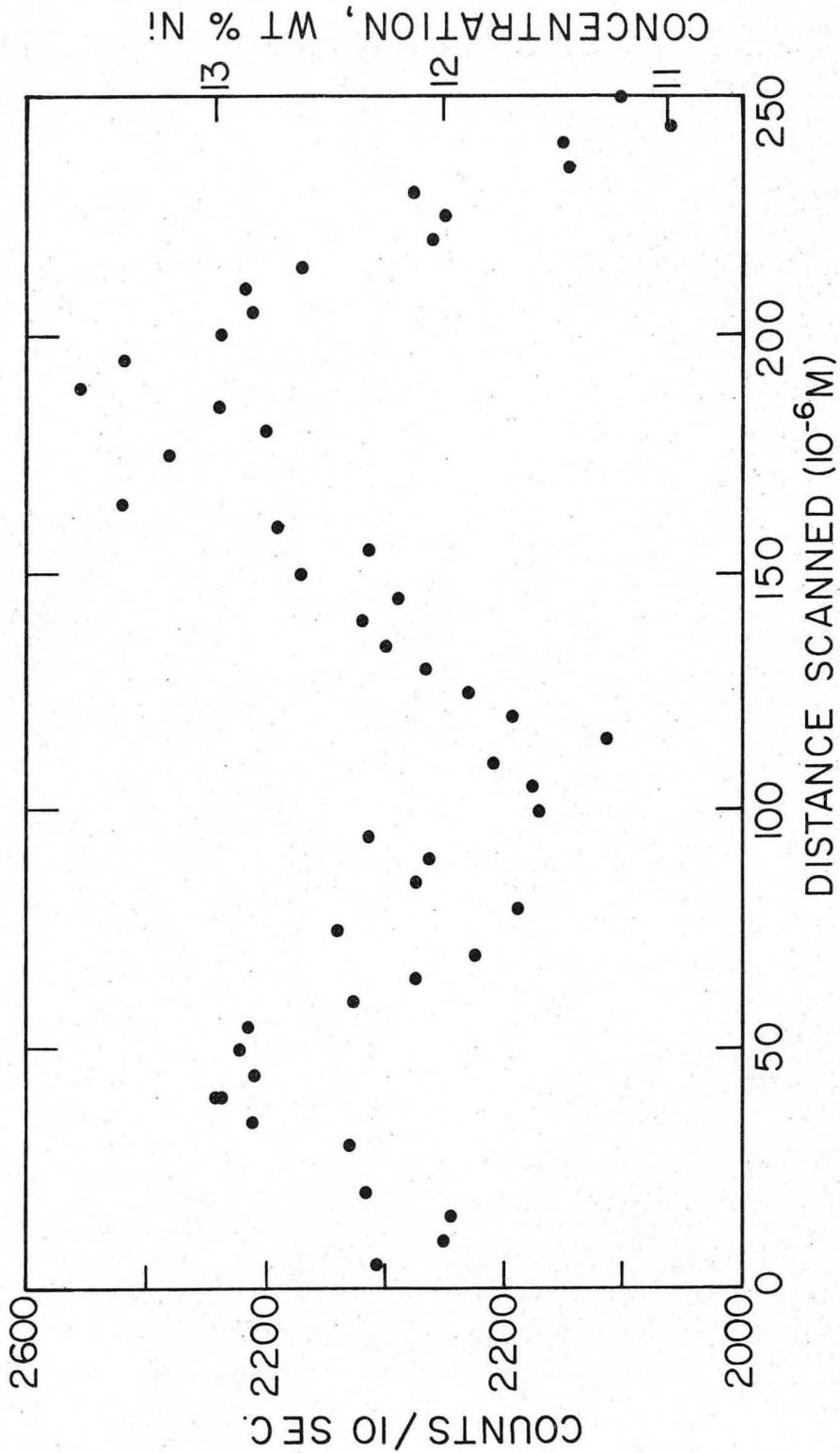
XBL 753 - 5978

Fig. 6



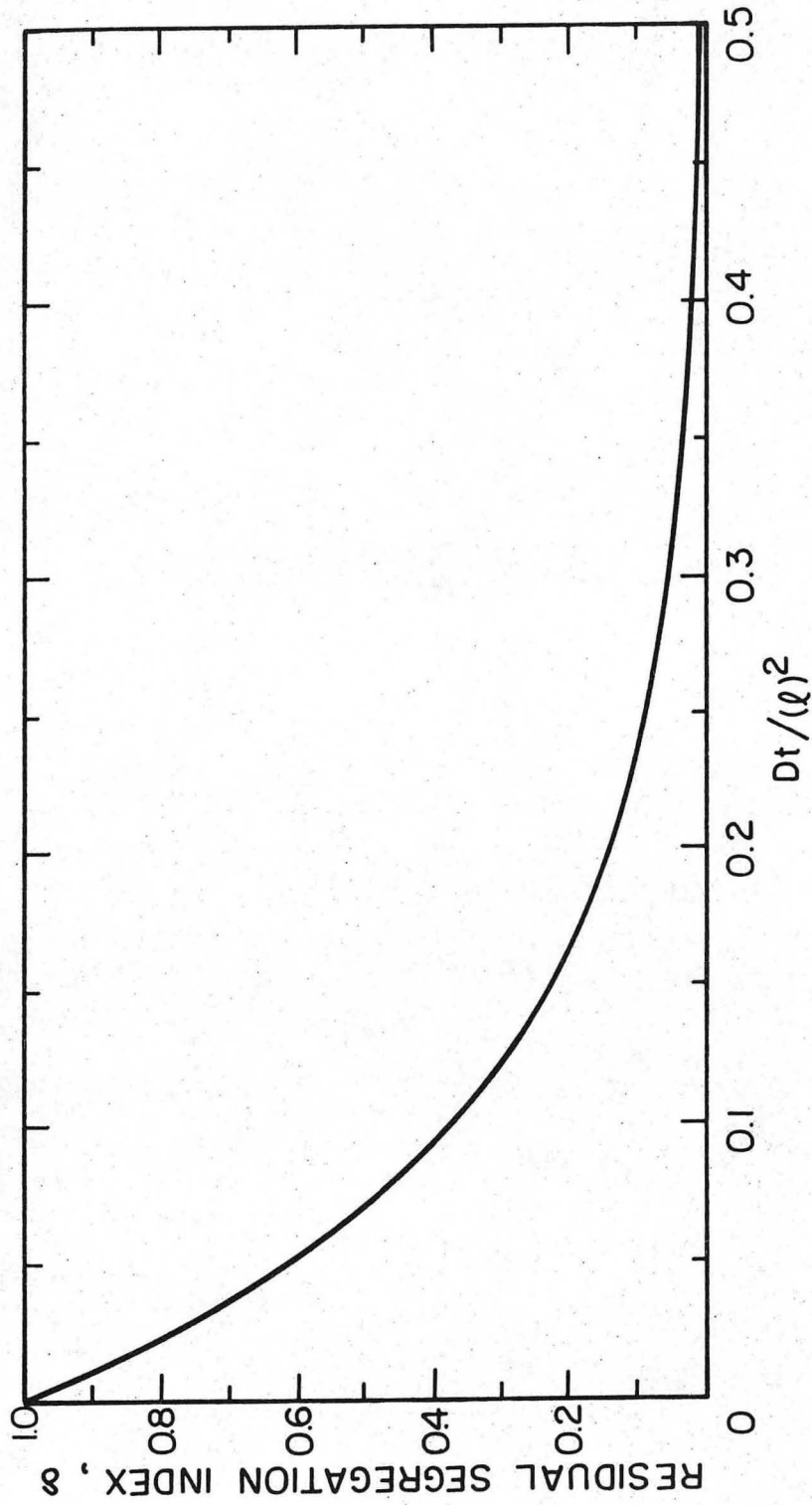
XBB 753-2223

Fig. 7.



XBL 753 - 5979

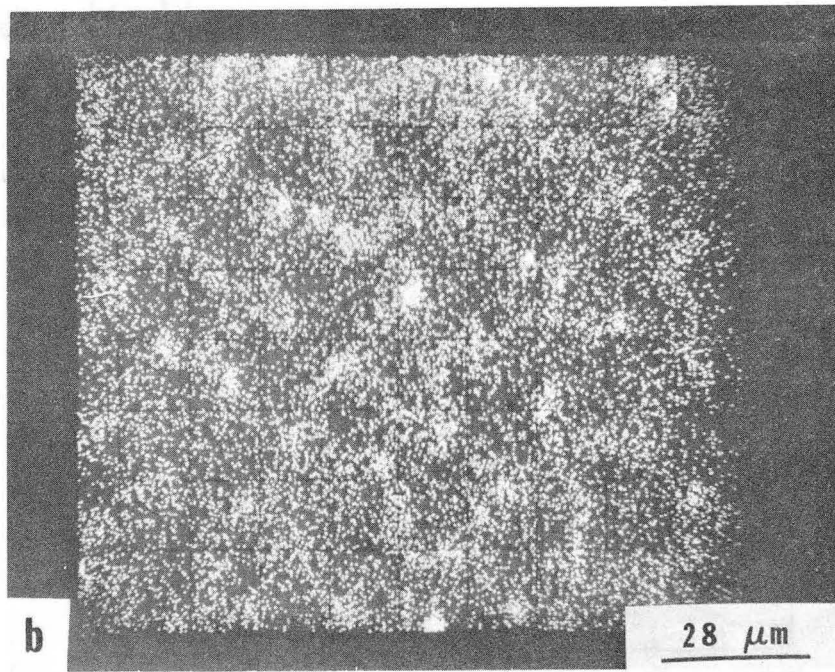
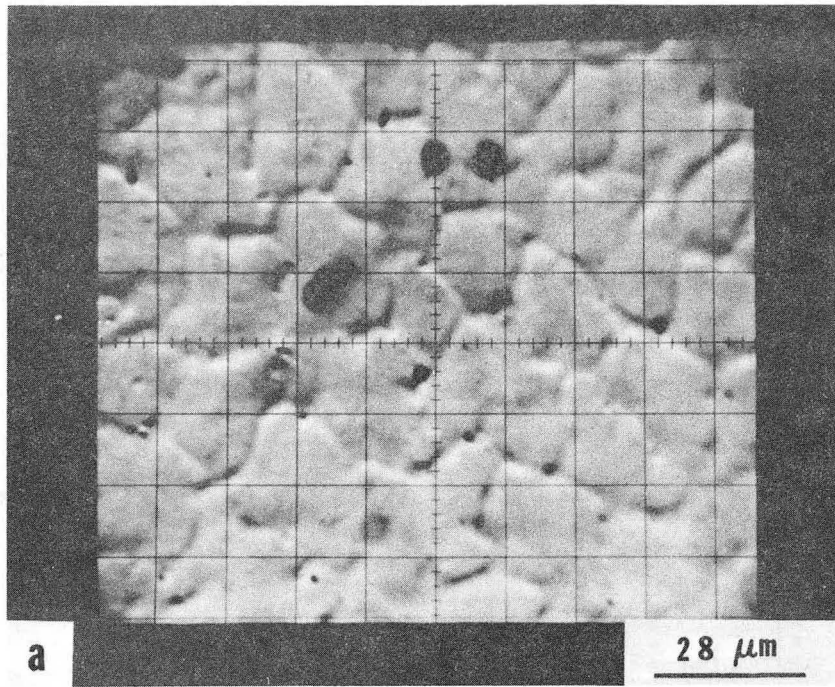
Fig. 8



XBL753-5972

Fig. 9





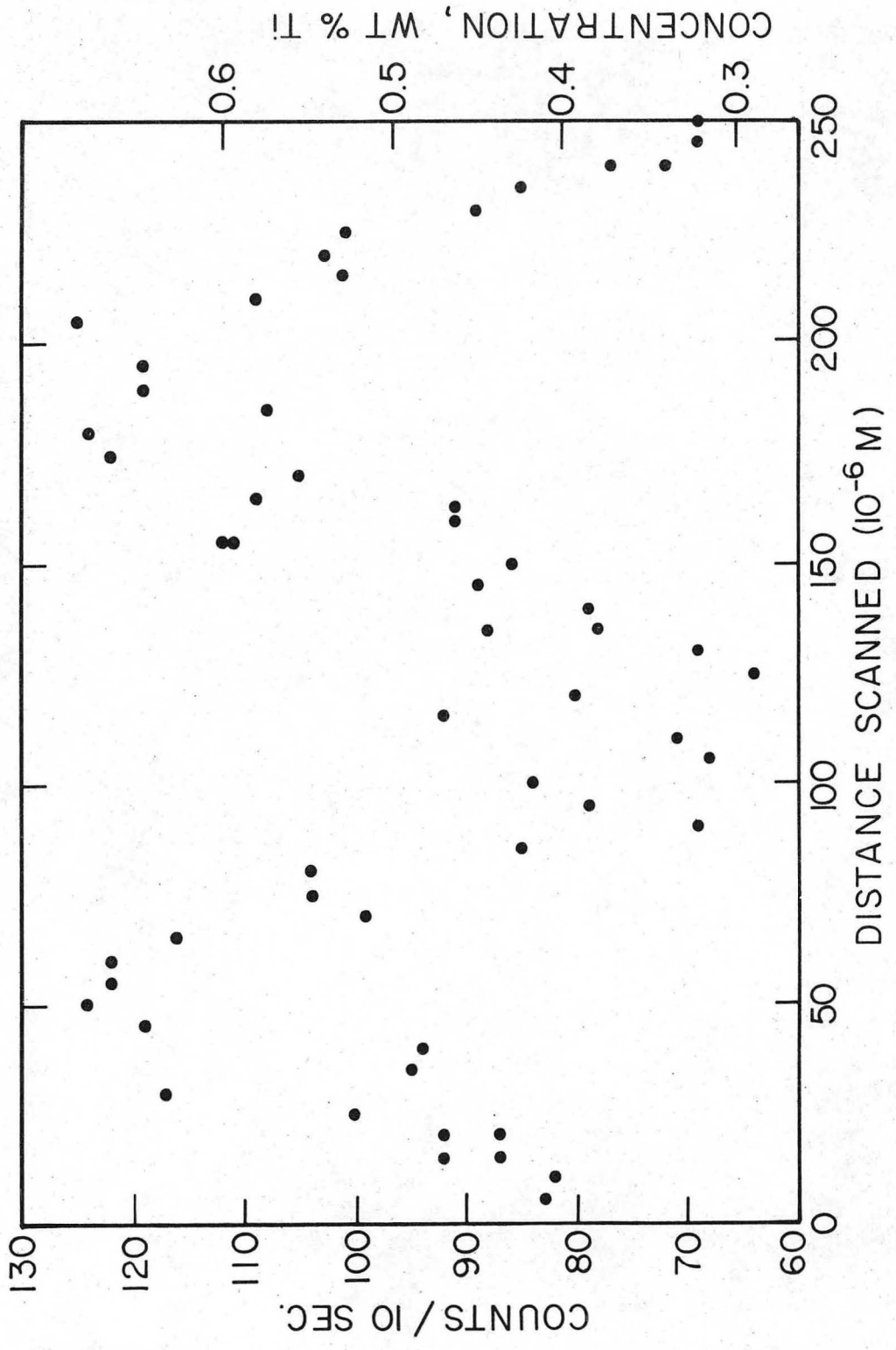
XBB 753-2210

Fig. 10



XBB 753-2222

Fig. 11a



XBL753-5981

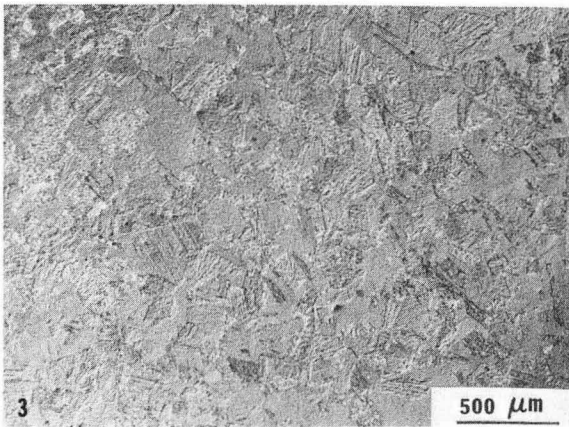
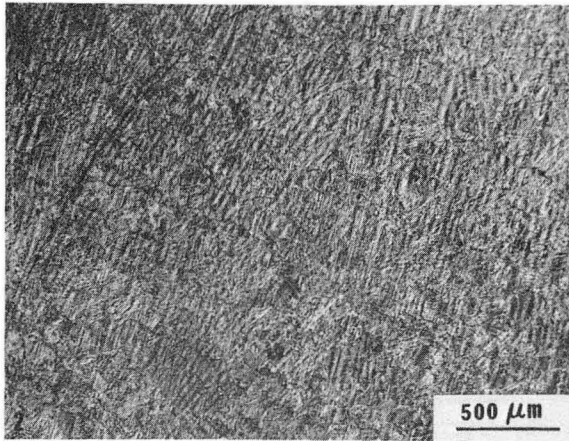
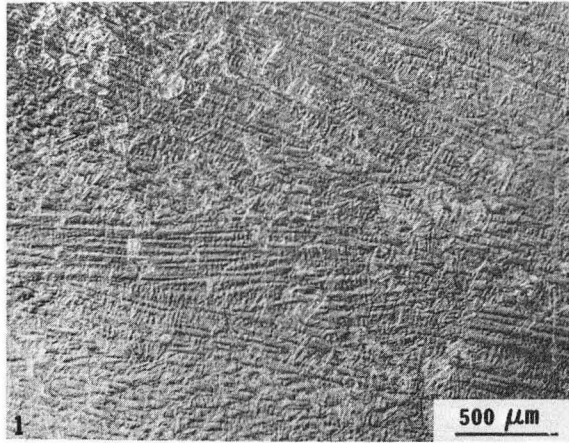
Fig. 11b





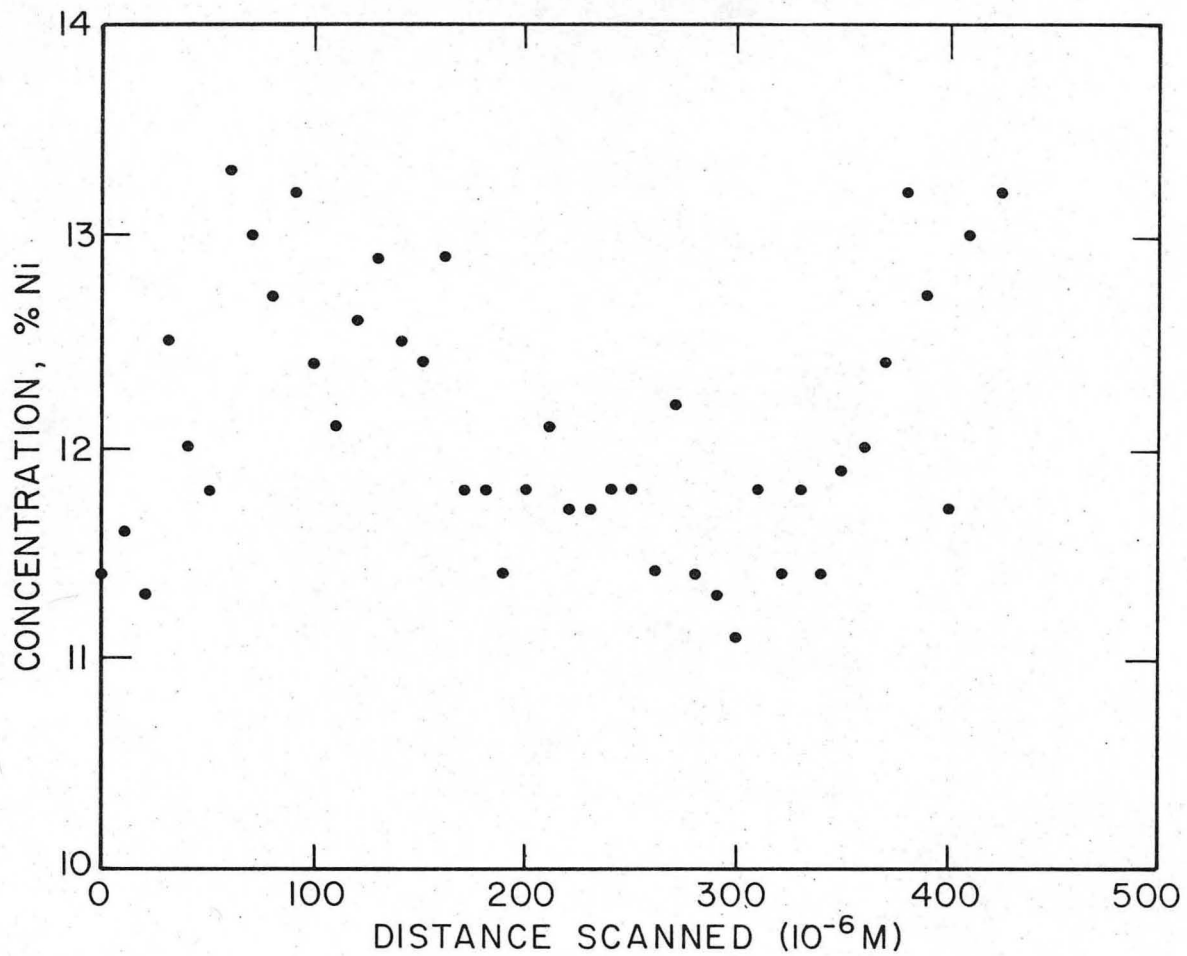
XBB 753-2221

Fig. 12a



XBB 753-2202

Fig. 12b



XBL 753-5980

Fig. 13

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