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ZONE REFINING APPLIED TO A
BISMUTH-TIN SYSTEM

by

John S. Land, Morton Smutz and
George Burnet

August 1967

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TABLE OF CONTENTS

	Page
ABSTRACT	v
INTRODUCTION	1
GENERAL THEORY	2
EXPERIMENTAL PROCEDURE	6
DISCUSSION OF RESULTS	11
Zone Refining Results	11
Electron Microprobe Analysis	18
CONCLUSIONS	21
RECOMMENDED FUTURE WORK	23
LITERATURE CITED	25
APPENDIX A	26
APPENDIX B	28
Nomenclature	28

LIST OF ILLUSTRATIONS

	Page
Figure 1. Approximate concentration of solute after a single zone refining pass through a charge of uniform concentration.	4
Figure 2. Zone melter apparatus.	7
Figure 3. Schematic showing the slug packing arrangement.	8
Figure 4. Sn concentration versus distance along slug 1 before and after zone refining. Average C_0 is 110 ppm.	12
Figure 5. Relative Sn concentration versus distance in zone lengths after three zone refining passes of slug 1.	13
Figure 6. Sn concentration versus distance along slug 2 before and after zone refining. Average C_0 is 460 ppm.	14
Figure 7. Relative Sn concentration versus distance in zone lengths after one and three zone refining passes of slug 2.	15
Figure 8. Sn concentration versus distance along slug 3 before and after zone refining. Average C_0 is 760 ppm.	16
Figure 9. Relative Sn concentration versus distance in zone lengths after three zone refining passes of slug 3.	17
Figure 10. Photograph of the Sn x-ray fluorescence from the cross section of a cast slug containing approximately 560 ppm Sn.	19
Figure 11. Photograph of the Bi x-ray fluorescence from the cross section of a cast slug containing approximately 560 ppm Sn.	19
Figure 12. Photograph of the Sn fluorescence from the cross section of a zone refined slug containing approximately 1600 ppm Sn.	20
Figure 13. Photograph of the Sn fluorescence from the area blocked-in at the bottom of Figure 12.	20
Figure 14. Average furnace temperature versus average zone length, with zoning speed as a parameter.	27

ZONE REFINING APPLIED TO A BISMUTH-TIN SYSTEM*

John S. Land, Morton Smutz and
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ABSTRACT

A study of the purification of a Bi-Sn system by zone refining was the objective of this investigation. The method involved the passage of a molten zone through a charge of material. The determination of a distribution coefficient by comparing experimental data to computed values was of primary interest.

*This report is based on an M. E. thesis submitted by John Stephan Land, Jr., August 1967, to Iowa State University, Ames, Iowa.

INTRODUCTION

The concept of zone melting was initially introduced by Pfann (6) in 1952. The range of possible uses for zone melting seems almost limitless. The general term zone melting denotes a family of methods for controlling the distribution of soluble impurities or solutes in crystalline materials.

Zone refining can be used to purify materials ranging from low melting organic compounds to high melting metals. One particularly successful commercial application has been the purification of germanium for diodes and transistors.

A second zone melting operation, zone leveling, can effect the controlled addition of a desired solute to a crystal. By zone leveling, it may be possible to distribute a solute uniformly throughout a slug.

Zone melting is finding increased use as a method of growing single crystals, particularly by the floating zone technique. It can also do such diverse jobs as joining two solids together, preparing multicomponent eutectic alloys, helping to analyze for trace impurities, and determining portions of phase diagrams.

The purpose of this work was the application of the zone refining process to a Bi slug containing Sn solute and the determination of a distribution coefficient for particular operating conditions.

GENERAL THEORY

The zone refining process involves the passage of a molten zone through a charge containing a solute and, if the concentration of the solute in the solid differs from that of the liquid, effects a redistribution of the solute. If the metal contains a single solute, it may be possible by using many zone refining passes to deposit all of the solute at one end of the charge leaving the opposite end free of solute.

One parameter controlling the movement of the solute is the distribution coefficient. The distribution coefficient of a solute, k , is defined as the ratio of the solute concentration in the solid to the solute concentration in the liquid. Solutes with distribution coefficients less than one tend to concentrate in the final zone length, while solutes with distribution coefficients greater than one will tend to concentrate in the initial zone length. If k is one, purification by zone refining will not occur. In this paper, it will be assumed that k is less than one.

Other factors that influence the purification are: 1 - the diffusion rates of the solute in the solid and liquid phases, 2 - the degree of stirring in the liquid zone, 3 - the irregularity of the solidifying interface, and 4 - the concentration gradient in the liquid near the interface.

The single pass zone refining of a charge with a uniform solute concentration C_0 may result in an initial transition region of low solute concentration, a region of uniform concentration, and an end region of one zone length which has a high solute concentration. As the molten zone advances along the charge, a layer of solid freezes out at the rear of

the zone and a layer of melt is formed at the front of the zone. On the initial pass, the first solid to freeze has a solute concentration of kC_0 which is less than C_0 , and therefore the liquid zone is enriched in solute. As the zone progresses, the liquid continues to increase in solute concentration until it reaches a concentration of C_0/k . (If k is small, this concentration may never be reached, and a region of uniform concentration will not be present.) When this condition is attained, the concentration of the solid layers entering and leaving the molten zone are the same, and hence no further purification is accomplished. When the zone reaches the end of the charge, the remaining liquid solidifies by normal freezing.¹ Figure 1 shows a plot of how the concentration of solute after a single pass might be expected to vary with the distance the zone has traversed along the charge (7).

The equation, derived by Read (6), for the concentration in the solid, C , after one pass at any distance, x , in the direction of zone refining, except in the last zone, is:

$$C/C_0 = 1 - (1 - k) e^{-kx/l} \quad (1)$$

where C_0 is the initial concentration, k is the distribution coefficient and l is the zone length. The assumptions made in deriving this equation are: 1 - the distribution coefficient and the zone length are constant, 2 - the initial solute concentration is uniform, 3 - diffusion in the solid is negligible, 4 - diffusion in the liquid zone is complete (i.e., the solute

¹Normal freezing is the term applied to the freezing of a length of molten metal from one end.

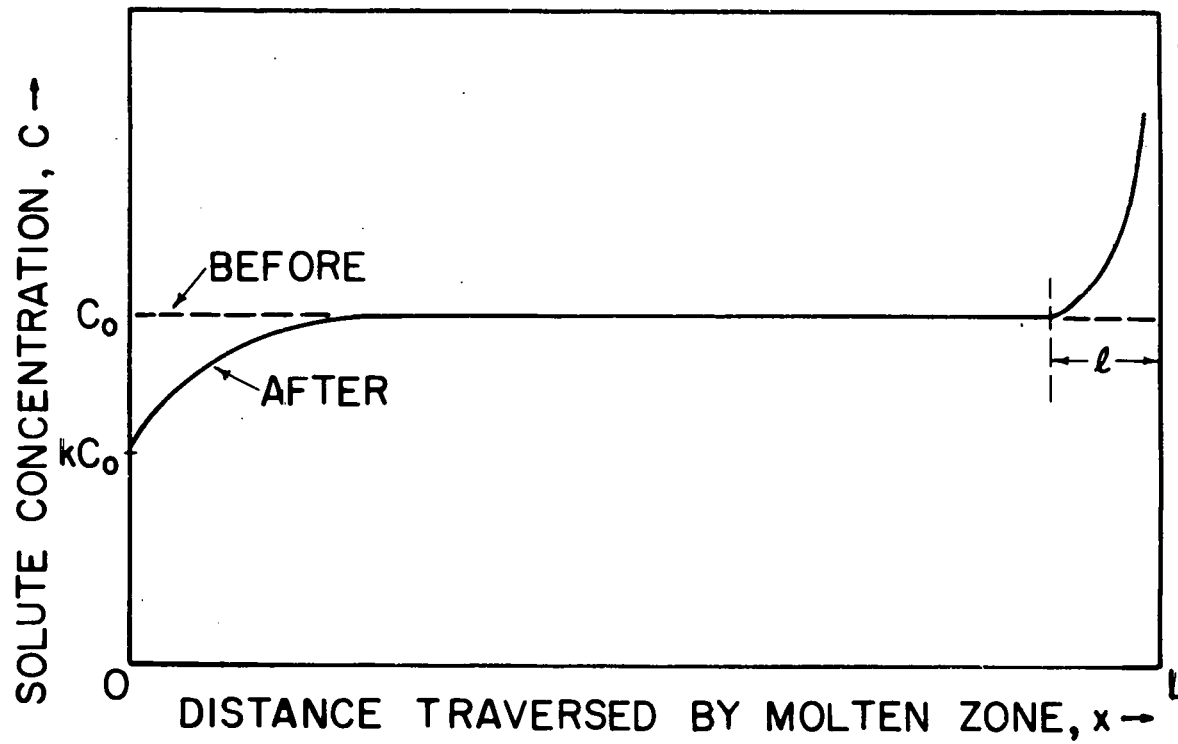


Figure 1. Approximate concentration of solute after a single zone refining pass through a charge of uniform concentration. (Schematic)

concentration in the liquid at any time is uniform), and S - solubility of the solute in the liquid zone is not exceeded.

Next consider a second pass through a first-pass distribution like that of Figure 1. As the molten zone passes through the initial transition region, it accumulates solute and leaves behind it a lower and somewhat longer initial region. When the front of the zone reaches the beginning of the normal freezing region, the solute concentration of the liquid melt will increase sharply. Thus, the pile-up at the end of the charge is reflected back one zone length during the second pass, and back one additional zone length, with diminishing intensity, for each succeeding pass. Additional passes therefore lower the initial transition region, raise the end region, and decrease the length of the intermediate region. Ultimately all three regions blend into a relatively smooth curve.

The solute concentration profile after multipass zone refining is more difficult to describe mathematically. The basic differential equation, derived independently by Reiss (8) and Lord (4) is:

$$\frac{\ell}{k} dC_n(x) = [C_{n-1}(x + \ell) - C_n(x)] dx \quad (2)$$

where $C_n(x)$ denotes the solute concentration freezing out of the zone at distance x in the n th pass and $C_{n-1}(x + \ell)$ represents the concentration obtained in the previous pass at a distance $x + \ell$ from the starting end. Equation 2 can be applied to all of the slug except the last zone length. The assumptions of Equation 1, excluding the condition of a uniform initial solute concentration, were again used in the derivation of this equation. Equation 2 can be solved readily by a high speed computer.

EXPERIMENTAL PROCEDURE

The zone melter unit was designed by Richard Wornson, Harvey Jensen, and Edwin Olson at Ames Laboratory. A photograph of the apparatus is shown in Figure 2. It is designed to handle samples up to approximately 15 inches in length and 1 inch in diameter. The molten zone is produced by a resistance heater mounted on a movable carriage which is driven by a threaded rod.

Three Bi slugs having approximately 150, 500 and 750 ppm Sn were cast by melting appropriate amounts of Bi and Sn and allowing the solution to mix for about 30 minutes, hopefully obtaining a uniform dispersion of the Sn. The melt was then poured into a graphite crucible and solidified rapidly.

The initial Sn concentration as a function of distance along the cast slug was determined by analyzing the lathe turnings (0.020 in) taken from the surface of the slug. The turnings from each two inch interval of length were collected, dissolved in nitric acid, combined with an appropriate amount of spectrographic graphite, and evaporated to dryness before analysis. The analysis involved an emission spectrographic technique (5).

The slug was then placed in a pyrex crucible as illustrated in Figure 3. Nine chromel-alumel thermocouples located approximately 1/16 inch away from the slug monitored the temperature. The molten zone length was determined by measuring the difference between the time at which a thermocouple would first reach 271 °C, the melting point of Bi, and the time when the same thermocouple would again reach the melting temperature

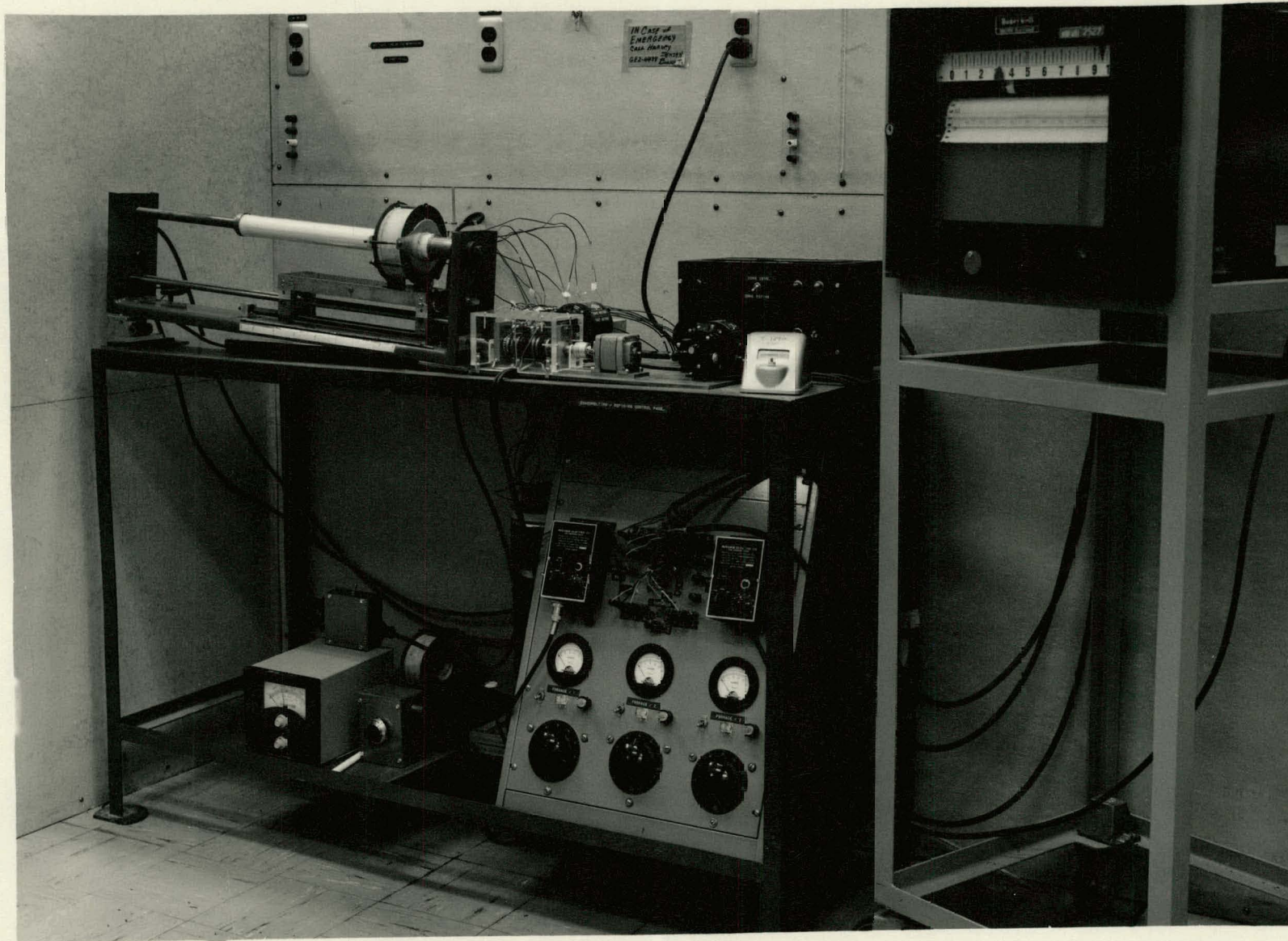


Figure 2. Zone melter apparatus.

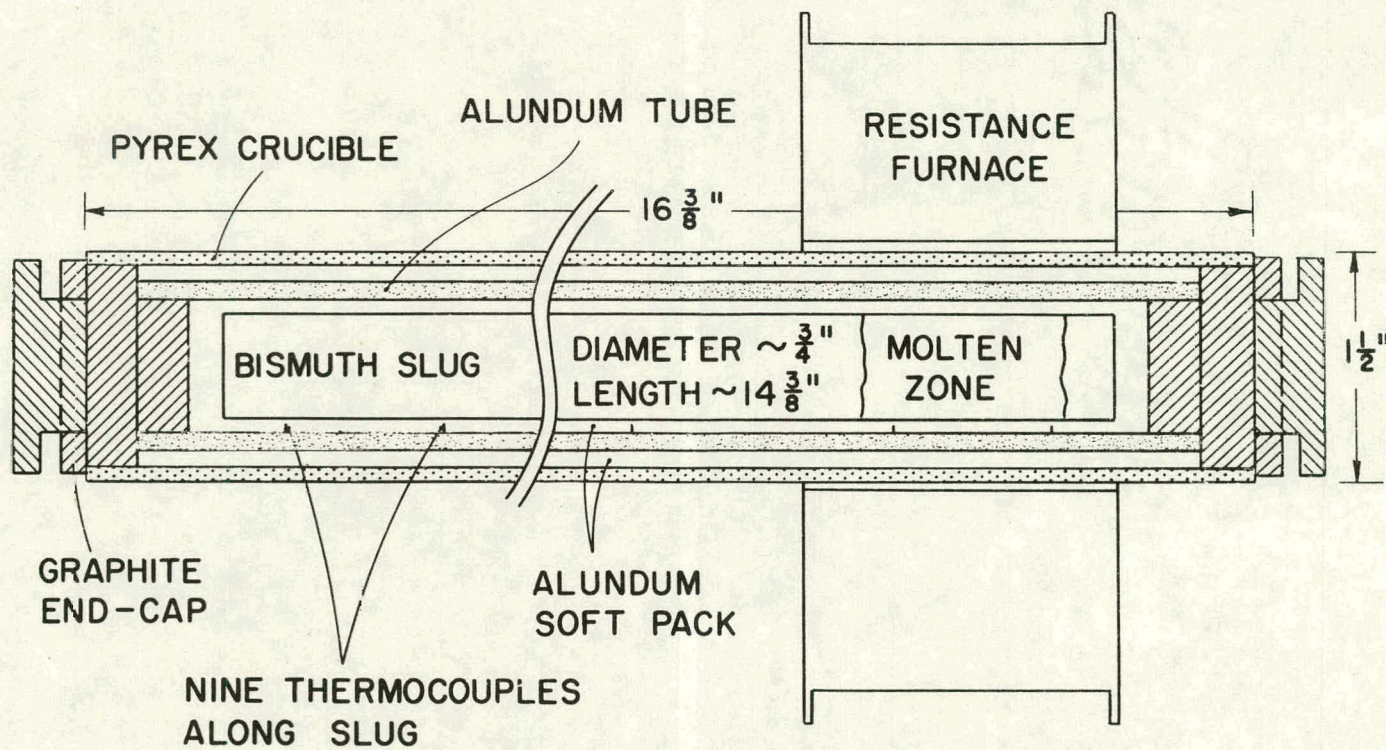


Figure 3. Schematic showing the slug packing arrangement.

of Bi after passing through a maximum temperature when the furnace moved directly over the thermocouple. The difference in the times, multiplied by the furnace speed, gave the distance that the material was molten. This method of determining the zone length assumes that the liquid zone remains constant while passing by the thermocouple.

Zone refining runs were conducted on the three Bi slugs containing Sn solute. The slugs were 14.25 inches in length and 3/4 inch in diameter. The average initial Sn concentration of the slugs were 110, 460 and 760 ppm. The average molten zone length of 1.5 inches and the average zoning speed of 0.7 in/hr were held constant for all of the runs.

To obtain the profile of the Sn concentration along the slug after zone refining, diametrical drillings taken at nine equally spaced positions along the slug were analyzed spectrographically, following the same procedure used for the lathe turnings. The slug containing approximately 460 ppm Sn was sampled after the first and third zone refining passes, while drillings were taken from the slugs containing 110 and 760 ppm Sn only after the third pass.

An electron microprobe analysis (1) was conducted using an electron microprobe Model EMX, manufactured by Applied Research Laboratories. The microprobe measures the x-ray fluorescence given off by specific atoms, such as Sn atoms, as they return from an excited state.

Two cross sectional samples taken from a cast slug and two samples from a slug subjected to three zone refining passes were analyzed on the microprobe. A spark cutter was used to remove the cross sectional slices from the slugs, and the sample surfaces were then polished on abrasive

wheels before analysis. The surface of one sample was electropolished but it proved to be too rough for accurate analysis.

DISCUSSION OF RESULTS

Zone Refining Results

Zone refining of three Bi-Sn slugs was performed to study the purification effect of the process and to determine a distribution coefficient. Plots showing the Sn concentration both before and after zone refining versus the distance along the slug appear in Figures 4, 6 and 8.

Figures 5, 7 and 9 are semi-logarithmic plots of the ratio of the Sn concentration after zone refining to the initial Sn concentration in the cast slug before zone refining versus the distance in zone lengths from the beginning of the charge. The solid curves in these figures were plotted from experimental data while the dashed curves represent values computed by Hamming (7) and Burris, et al. (2). The parameters of the curves are the distribution coefficient, k , the number of zone refining passes, n , and the ratio of the total length of the slug to the zone length, L/l . The total length to zone length ratio was 10 for all the runs.

Figure 7 shows experimental and computed values for one and three zone refining passes, while Figures 5 and 9 compare results only after the third pass.

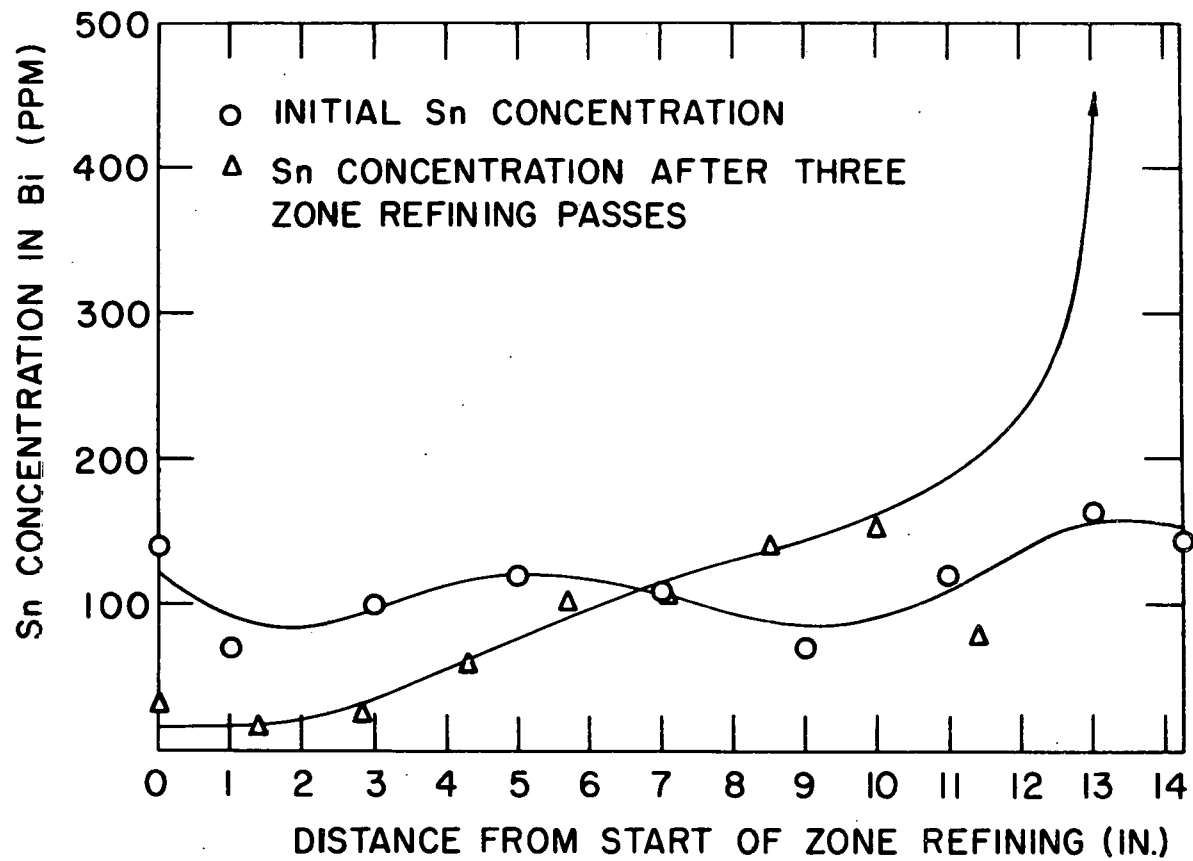


Figure 4. Sn concentration versus distance along slug 1 before and after zone refining. Average C_0 is 110 ppm.

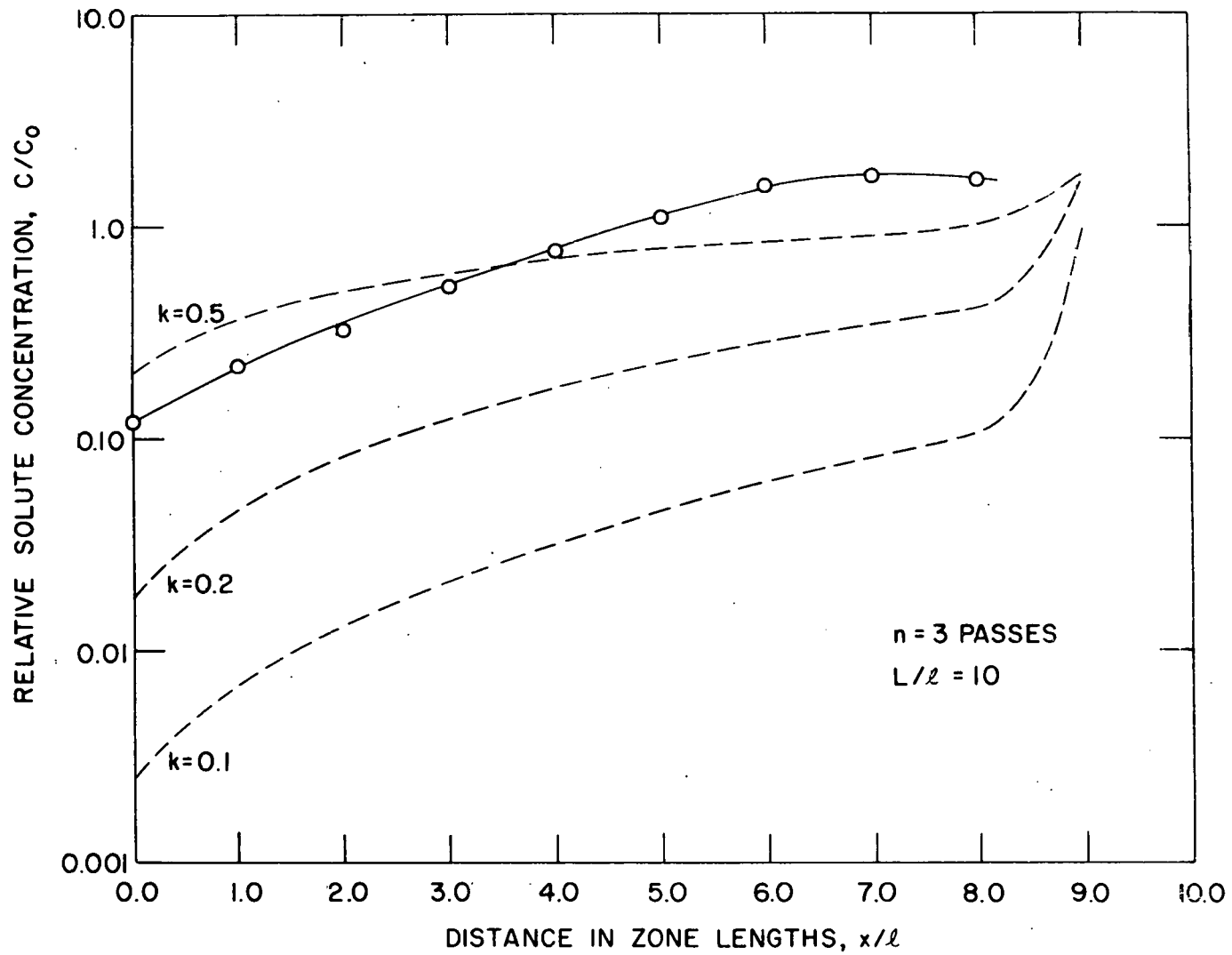


Figure 5. Relative Sn concentration (logarithmic scale) versus distance in zone lengths after three zone refining passes of slug 1. Computed curves are dashed.

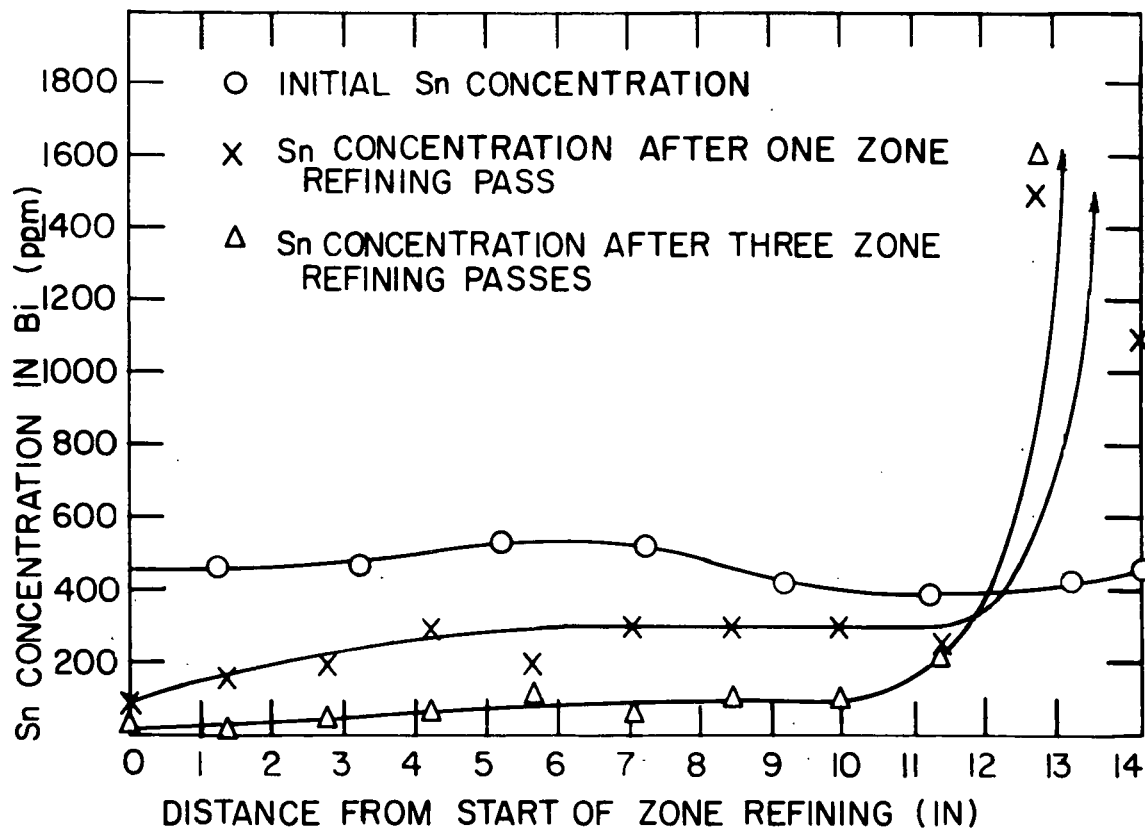


Figure 6. Sn concentration versus distance along slug 2 before and after zone refining. Average C_0 is 460 ppm.

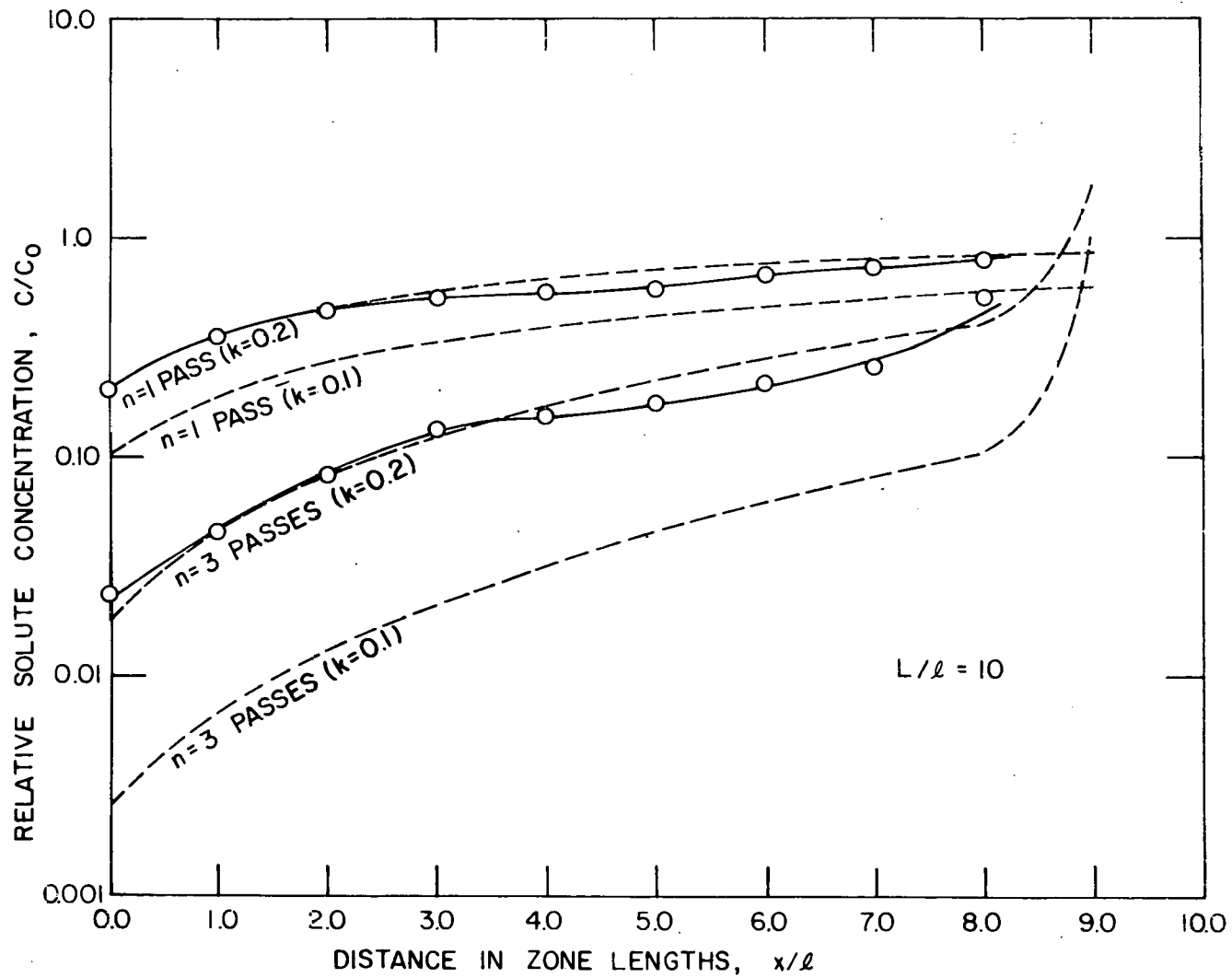


Figure 7. Relative S_n concentration (logarithmic scale) versus distance in zone lengths after one and three zone refining passes of slug 2. Computed curves are dashed.

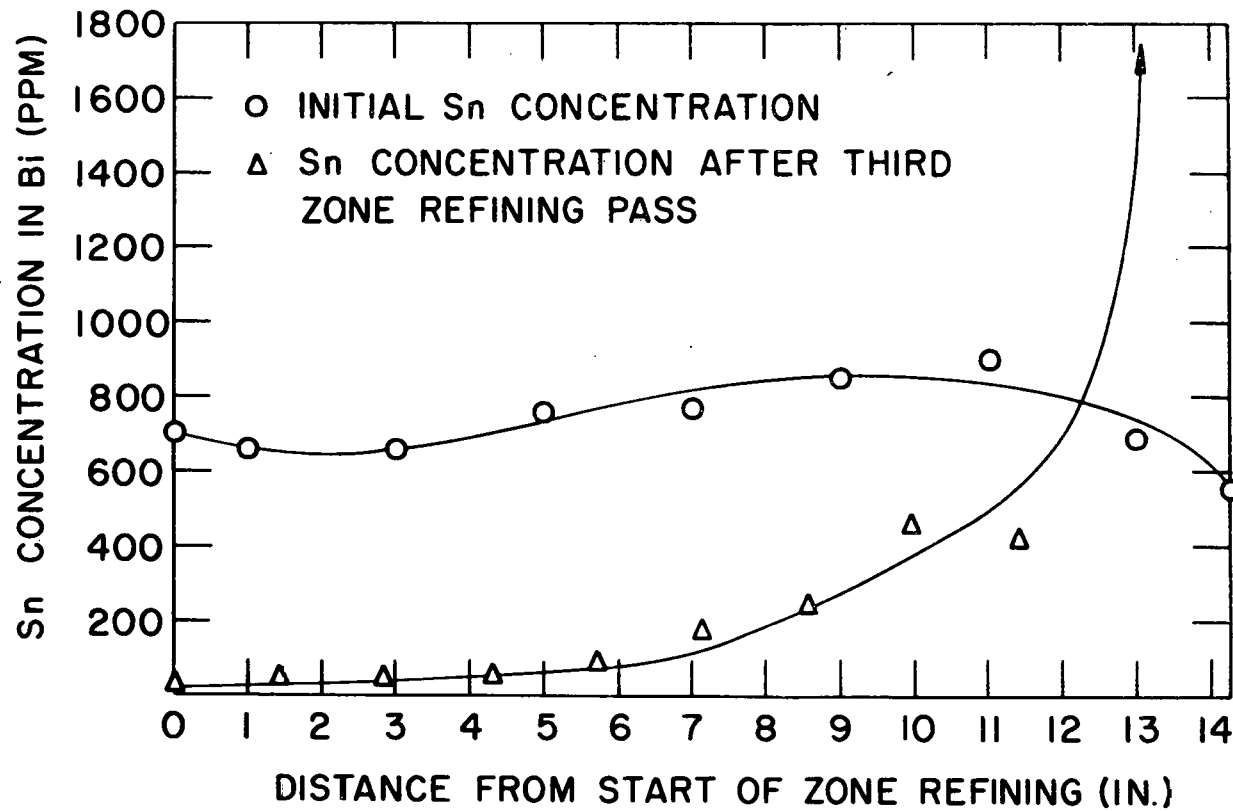


Figure 8. Sn concentration versus distance along slug 3 before and after zone refining. Average C_0 is 760 ppm.

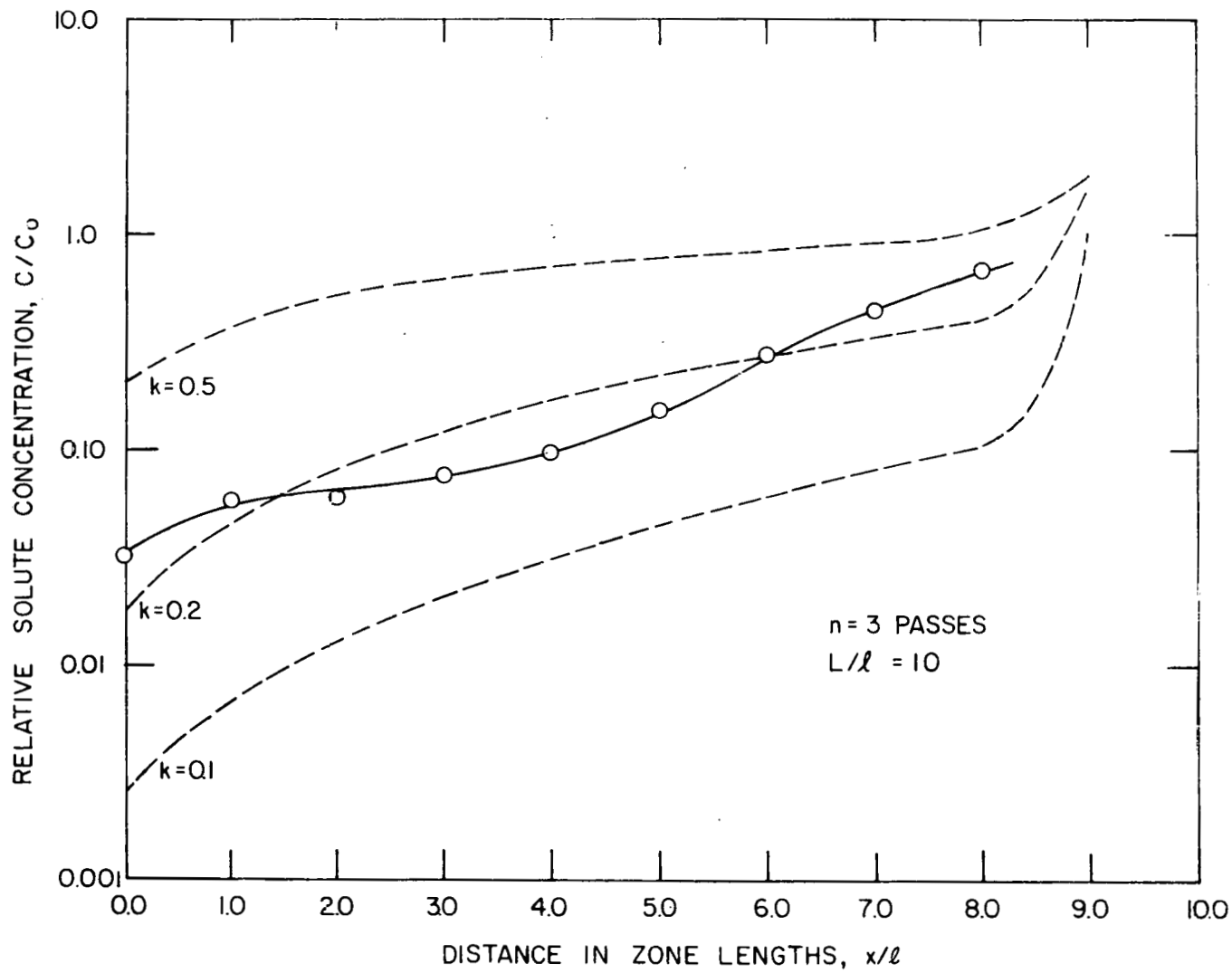


Figure 9. Relative Sn concentration (logarithmic scale) versus distance in zone lengths after three zone refining passes of slug 3. Computed curves are dashed.

Electron Microprobe Analysis

Cross sectional samples of two Bi-Sn slugs were analyzed on an electron microprobe to study qualitatively the uniformity of the Sn concentration in the radial direction and also to detect the presence of eutectic and oxide formations. Two samples of 200 ppm and 1600 ppm Sn, taken from a slug after the third zone refining pass, and two samples of 700 ppm and 560 ppm, taken from the ends of a cast slug before it was zoned refined, were analyzed. None of the samples indicated the presence of appreciable amounts of oxides.

Figures 10 through 13 are photographs taken of the x-ray fluorescence pattern on the oscilloscope screen during the analysis. In Figures 10, 11 and 12, the actual sample areas under observation at the time of the photographs (~ 0.27 sq cm) have been magnified 300 times.

The light spots in Figure 10 are the x-ray fluorescence produced by Sn atoms on the cross sectional surface of the cast slug containing approximately 560 ppm Sn. The fluorescence given off by Bi atoms (light areas) in a different section of the same slug is shown in Figure 11. These two pictures are representative of the x-ray patterns obtained from both the cast and zone refined slugs with Sn concentrations of 700 ppm or less.

Figure 12 shows the Sn x-rays from the cross section of the zone refined slug having a concentration of 1600 ppm Sn. Appreciable segregation of the Sn was present in this sample, as can be observed from the photograph.

Figure 13 is an enlargement of the blocked-in portion at the bottom of Figure 12. The magnification is 2400 times the actual surface area under observation (~ 0.033 sq cm).

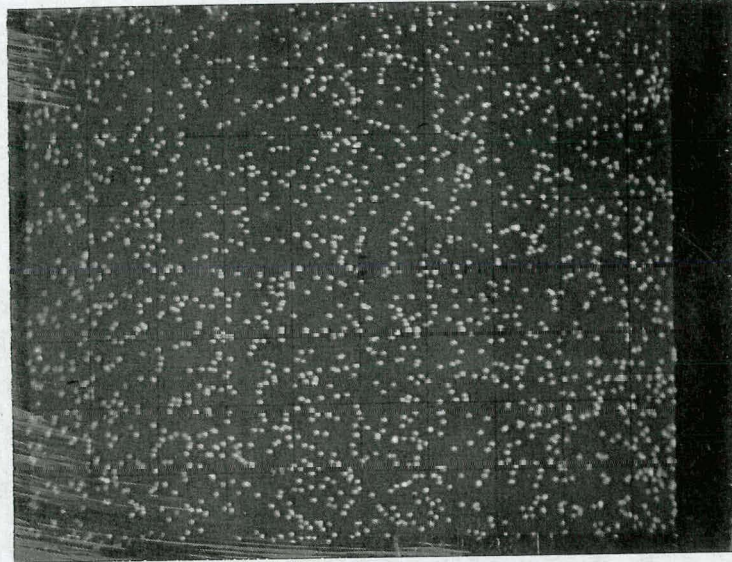


Figure 10. Photograph of the Sn x-ray fluorescence (light areas) from the cross section of a cast slug containing approximately 560 ppm Sn. Magnification is 300X.

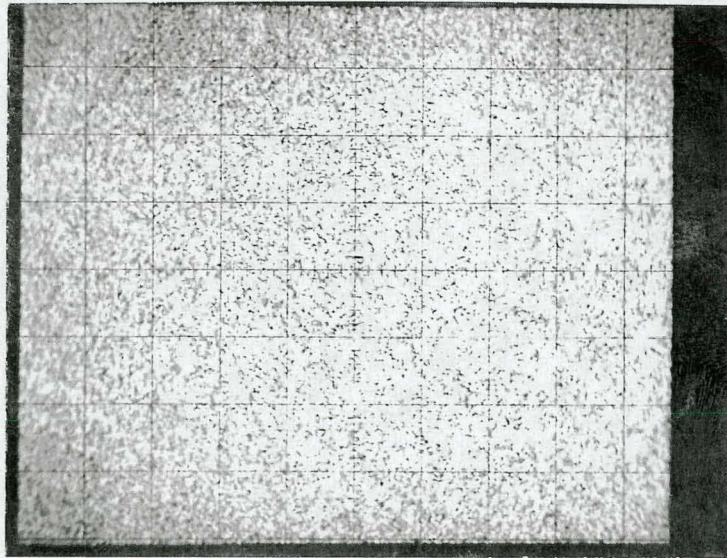


Figure 11. Photograph of the Bi x-ray fluorescence (light areas) from the cross section of a cast slug containing approximately 560 ppm Sn. Magnification is 300X.

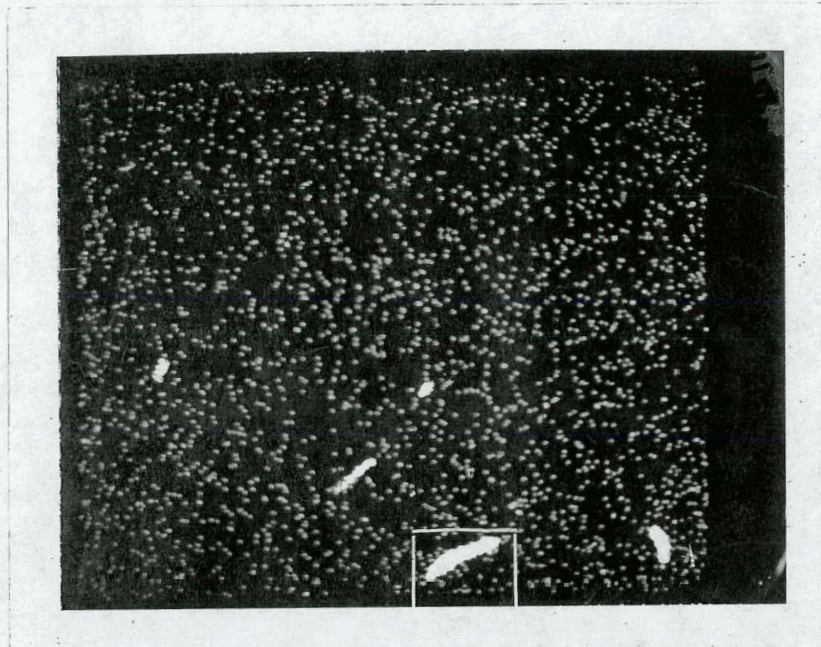


Figure 12. Photograph of the Sn fluorescence (light areas) from the cross section of a zone refined slug containing approximately 1600 ppm Sn. Magnification is 300X.

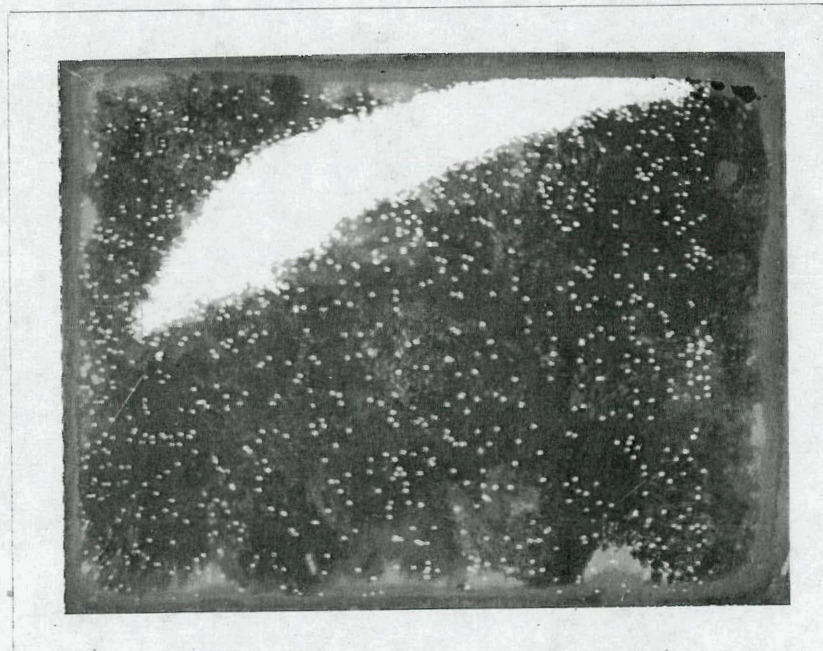


Figure 13. Photograph of the Sn fluorescence (light areas) from the area blocked-in at the bottom of Figure 12. Magnification is 2400X.

CONCLUSIONS

In Figure 7, which correlates the relative Sn concentration for slug 2 (~ 560 ppm) as a function of zone lengths along the slug, the experimental curves show good agreement with the computed curves indicating a distribution coefficient of 0.2 for a distance of about four zone lengths, and then the experimental values fall slightly below the computed curve equal to 0.2. One of the assumptions used in calculating the computed curves is that the initial solute concentration, in this case Sn, is uniform along the length of the slug. In Figure 6, it can be seen that a uniform initial Sn concentration profile does exist along the first half of the slug, which corresponds to the region of close agreement in Figure 7. The initial Sn concentration does decrease somewhat in the latter half of the slug, possibly causing the dips in the experimental curves of Figure 7.

In Figures 5 and 9, the experimental curves do not fit the computed curves as closely. The distribution coefficient for slug 3 (~ 760 ppm) appears to be near to 0.2, while k for slug 1 (~ 110 ppm) seems to be closer to 0.5, although neither of these plots shows good agreement between the experimental and computed curves. By comparing Figures 4, 6 and 8, one can readily see that the initial Sn concentration in slugs 1 and 3 is less uniform than the initial concentration in slug 2. This non-uniformity of the initial Sn concentration is likely a major cause of the deviation in the shapes of the experimental from the computed curves.

Other assumptions made in deriving the theoretical curves that possibly were not met in the experimental work are 1 - complete diffusion in the liquid melt, 2 - a constant distribution coefficient, and 3 - a

constant zone length. No attempt was made to determine the degree of diffusion in the liquid melt or to measure the constancy of the distribution coefficient along the length of the slug. The calculated zone lengths for the middle two-thirds of the three slugs were all within 5% of the average zone length of 1.5 inches, although the zone lengths were up to 20% longer than the average near the ends of the bar.

Inaccuracy in the spectrographic determination of the Sn concentration could also be a possible cause of the difference between the experimental and computed values. The reported accuracy of the spectrographic technique was to the nearest 50 ppm, and this degree of accuracy became questionable at the lower Sn concentrations. Duplicate samples were analyzed, and these values varied from 5% to over 100% from the original values. This lack of precision in the analytical data, especially at lower Sn concentrations, could account for the inconsistency of the distribution coefficient for slug 1 (~ 0.5 as compared to ~ 0.2 for the other two slugs) which had an average C_0 of only 110 ppm Sn.

The electron microprobe analysis showed that segregation did not occur when the Sn concentration was approximately 700 ppm or below, but that an appreciable amount of eutectic phase was present at a Sn concentration of approximately 1600 ppm. Probably the Bi-Sn eutectic does not appear until the Sn concentration is in the 1000 to 1600 ppm range.

RECOMMENDED FUTURE WORK

Experimental work in zone refining requires that the solute concentration profile in the solid be known to a high degree of accuracy, and therefore the development of a more precise analytical method than the emission spectrographic technique used in this investigation is a necessity.

Also, before one proceeds further with the determination of distribution coefficients, some consideration should be given to the significance of the values obtained. The distribution coefficient in this paper is defined generally as the ratio of the solute concentration in the solid to the solute concentration in the liquid. Moreover, an equilibrium distribution coefficient, k_0 , can be defined as the ratio of the concentration of the solute in the solid to that in the liquid, when equilibrium exists between the two phases. If freezing occurs slowly so that the solute concentration in the liquid remains uniform, the concentration in the freezing solid is k_0 times that in the liquid, provided one may assume diffusion in the solid to be negligible. However, if crystallization does not proceed slowly, solute atoms are rejected by the advancing solid more rapidly than they can diffuse into the bulk of the melt. A concentration gradient thus develops in the liquid just ahead of the advancing interface. It is the solute concentration in this enriched layer, rather than that in the main body of the liquid, which directly determines the rate of solute incorporation into the solid. True equilibrium presumably occurs only if the crystallization speed is essentially zero. When crystallization occurs at a measurable speed, the equilibrium at the interface will be disturbed to some degree.

To compensate for any deviation from equilibrium at the solid-liquid interface, Burton, et al. (3) defined an effective distribution coefficient, k_{eff} , which describes the overall reaction of the solute incorporation into the crystal at non-zero growth speeds, as the ratio of the solute concentration in the solid to that in the main body of liquid. The effective distribution coefficient is the quantity measured experimentally in the zone refining process. It may be a function of the zoning speed, the interface orientation, the diffusivity and stirring conditions in the liquid, and the solute concentration. It is important to know k_{eff} because it is needed in predicting the purification achievable by zone refining, but if the deviation from equilibrium at the solid-liquid interface is great, the value may be highly empirical and therefore applicable only to a particular piece of equipment operating under certain conditions.

In future work, it is recommended that an attempt be made to determine how closely k_{eff} approaches k_0 under various operating conditions. This research might include a study of the solute concentration build-up in the melt at the solid-liquid interface as a function of time, distance, and initial solute concentration. Possibly, this investigation could be carried out by monitoring the actual zone refining process with an electron microprobe.

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APPENDIX A

Zone refining passes were conducted at various temperatures on a pure Bi slug to determine a correlation between the furnace temperature and the zone length. The runs were made at zoning speeds of 0.7 and 2.1 in/hr.

Figure 14 is a plot of the average furnace temperature versus average zone length, with zoning speed as a parameter.

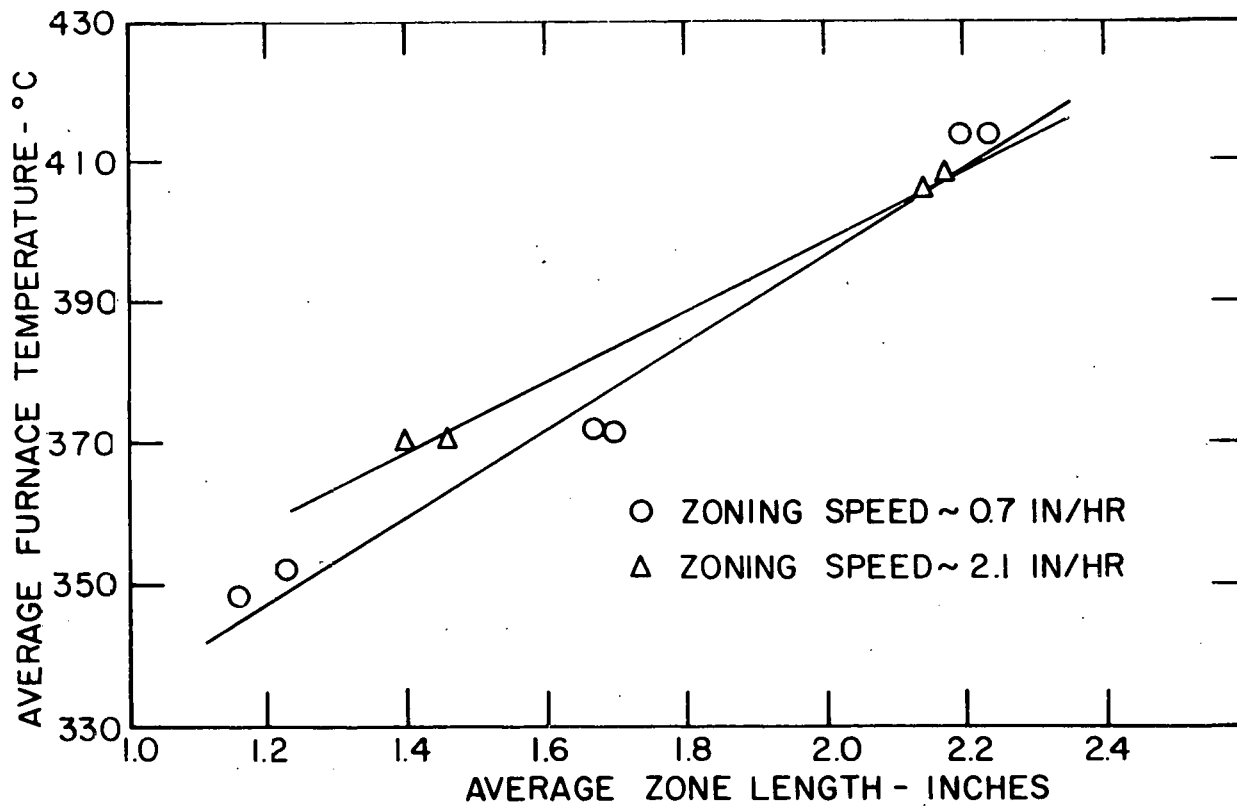


Figure 14. Average furnace temperature versus average zone length, with zoning speed as a parameter.

APPENDIX B

Nomenclature

C_0	initial solute concentration (Sn) in the slug before zone refining, ppm.
C	solute concentration (Sn) in the slug after zone refining, ppm.
k	distribution coefficient - ratio of the solute concentration in the solid to that in the liquid.
k_{eff}	effective distribution coefficient - ratio of the solute concentration in the solid to that in the main body of liquid.
k_0	equilibrium distribution coefficient - ratio of the solute concentration in the solid to that in the liquid, when equilibrium exists between the two phases.
l	molten zone length, in.
L	total length of the slug, in.
n	number of zone refining passes.
x	distance along the slug traversed by the molten zone, in.

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02 BOTANY	20D FLUID MECHANICS	20G PARTICLE ACCELERATORS	18I REACTOR ENGINEERING
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07A CHEMICAL ENGINEERING	18A FUSION DEVICES (THERMONUCLEAR)	04A PHYSICS (ATMOSPHERIC)	18I REACTOR HAZARDS
07 CHEMISTRY (ANALYTICAL)	8G GEOLOGY	20M PHYSICS (LOW-TEMPERATURE)	18J REACTOR MATERIALS
07B CHEMISTRY (INORGANIC)	06R HEALTH AND SAFETY	20H PHYSICS (NUCLEAR)	18I REACTOR SAFETY
07C CHEMISTRY (ORGANIC)	20M HEAT TRANSFER	20H PHYSICS (PARTICLE)	18K REACTOR THEORY
07D CHEMISTRY (PHYSICAL)	08H HYDROLOGY	20I PHYSICS (PLASMA)	18L REACTORS--POWER
07E CHEMISTRY (RADIO- AND RADIATION)	18D INSTRUMENTATION	20L PHYSICS (SOLID-STATE)	18M REACTORS--PRODUCTION
07A CHEMISTRY (SEPARATION)	18B ISOTOPES	18C PLOWSHARE	18M REACTORS--RESEARCH
09B COMPUTERS	14B LABORATORIES AND TEST FACILITIES	PROGRESS REPORTS	11B REFRACTORIES
11F CORROSION	20I MAGNETOFLUIDMECHANICS	21 PROPULSION	08K SEISMIC PHENOMENA
20H COSMIC RADIATION	11 MATERIALS	06Q PROTECTIVE EQUIPMENT	18N SNAP TECHNOLOGY
18H CRITICALITY STUDIES	12 MATHEMATICS	04A RADIATION BELTS	22 SPACE TECHNOLOGY
20B CRYSTALLOGRAPHY	MEETINGS	RADIATION EFFECTS	13M STRUCTURES
07A DECONTAMINATION	11F METALLURGY	18F RADIATION SHIELDING AND PROTECTION	18 TRACER STUDIES
06R DOSIMETRY	04B METEOROLOGY	18G RADIOACTIVE WASTES	TRANSLATIONS
06F ECOLOGY			19 WEAPONRY

IOWA STATE UNIVERSITY

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AMES, IOWA 50010

Institute for Atomic Research

Ames Laboratory

U. S. Atomic Energy Commission

Iowa State University

F. H. Spedding, Director

October 17, 1967

Mr. Robert L. Shannon
Extension Manager, DTIE
U. S. Atomic Energy Commission
P. O. Box 62
Oak Ridge, Tennessee

Dear Mr. Shannon:

Re: IS-1689

Enclosed is a paper entitled "Zone Refining Applied to a Bismuth-Tin System" by John S. Land, Morton Smutz and George Burnet.

No copies have been printed for TID-4500 and Federal Clearing-house distribution. DTI is requested to reproduce in Microfiche form from copy enclosed and make TID-4500 distribution.

Yours very truly,

A handwritten signature in cursive script, appearing to read "W. E. Dreeszen". The signature is written in black ink and is positioned above the printed name.

W. E. Dreeszen
Head, Information & Security

WED: vnm