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## Microradiography with Metal Powders

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**Microradiography with Metal Powders**

by  
**Wallace Kluck**

A Thesis submitted to the Faculty of  
Liberal Arts College, Marquette Uni-  
versity, in Partial Fulfillment of the  
Requirements for the Degree of Bachelor  
of Science

Milwaukee, Wisconsin  
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The author wishes to express his grateful appreciation for the valuable assistance rendered by all persons aiding in this investigation. He is particularly grateful to Mr. Arthur G. Barker for obtaining the metal powders used as samples, for the design of the camera, and for his generous help in guiding the work to its completion. To Dr. Jack E. Greene the author is deeply indebted for his invaluable assistance and advice relative to the engraving and developing of the pictures contained in this thesis.

W. A. Fluck

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### Preface

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It is necessary to carefully grind and polish a specimen to a thickness of from one to five thousandths of an inch. This process requires a considerable amount of time and special metalurgical tools. For this reason microradiography, although it is an important industrial tool, has not been widely adopted.

The purpose of this investigation was to determine whether or not a metal powder could be substituted for the thin metal foil specimens which have previously been used exclusively. In a powder much of the detail



## I Introduction

Microradiography is a comparatively recent development in the X-ray field. The term applies to the analysis of a thin specimen by photographing it with X-rays and subsequently enlarging the photograph to bring out minute structural elements. A microradiograph has several advantages over a photomicrograph. First of all it goes beyond the surface and reveals the internal structure of the specimen, a more important advantage at least as concerns this paper is the ability of the microradiograph to enable one to accurately determine the chemical elements making up the specimen.

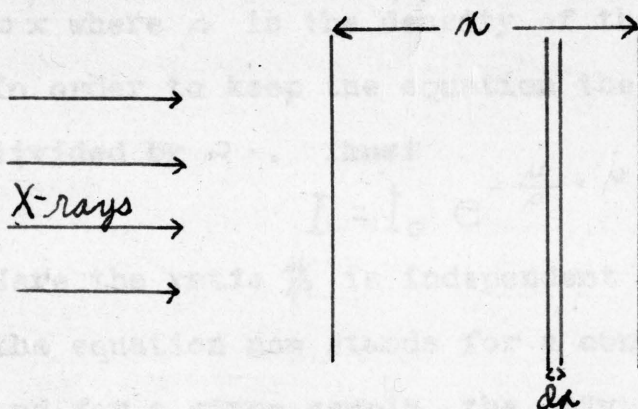
Research into the field has always been carried out by using thin metal foil as a specimen. Since X-rays do not penetrate metals to a very great extent, it is necessary to carefully grind and polish a specimen to a thickness of from one to five-thousandths of an inch. This process requires a considerable amount of time and special metalurgical tools. For this reason microradiography, although it is an important industrial tool, has not been widely adopted.

The purpose of this investigation was to determine whether or not a metal powder could be substituted for the thin metal foil specimens which have previously been used exclusively. In a powder much of the detail



found in a foil would naturally be lost. There was a question as to whether or not it would be possible to even detect various elements since the large range in size of the individual particles might be deceiving. Thus the chief emphasis in analyzing the microradiographs was to be placed on chemical analysis.

To understand the problem better some knowledge of the theory of absorption of X-rays is required. Consider a beam of monochromatic X-rays impinging on a material of thickness  $x$ .



The energy transmitted by a thin element  $dx$  varies directly with the thickness of the absorbing material and on the intensity  $I$  of the incident radiation.

Thus:

$$dI = -\mu I dx$$

on integration this yields

$$\int_{I_0}^I \frac{dI}{I} = -\mu \int_0^x dx$$

$$\ln \frac{I}{I_0} = -\mu x$$

$$I = I_0 e^{-\mu x}$$

(1)

This formula gives the intensity  $I$  after a beam of X-rays with an initial intensity of  $I_0$  penetrates a distance  $x$  in an absorbing medium. The constant  $\mu$  is called the linear absorption coefficient. It depends upon both the wavelength of X-rays used and the nature of the absorber. Thus  $\mu$  varies with a change in temperature, or a change of physical state. This variation can be eliminated if equation (1) is put in terms of mass traversed rather than distance. To make this substitution the term  $x$  is replaced by  $\rho x$  where  $\rho$  is the density of the absorbing material. In order to keep the equation the same,  $\mu$  must be divided by  $\rho$ . Thus:

$$I = I_0 e^{-\frac{\mu}{\rho} \cdot \rho x}$$

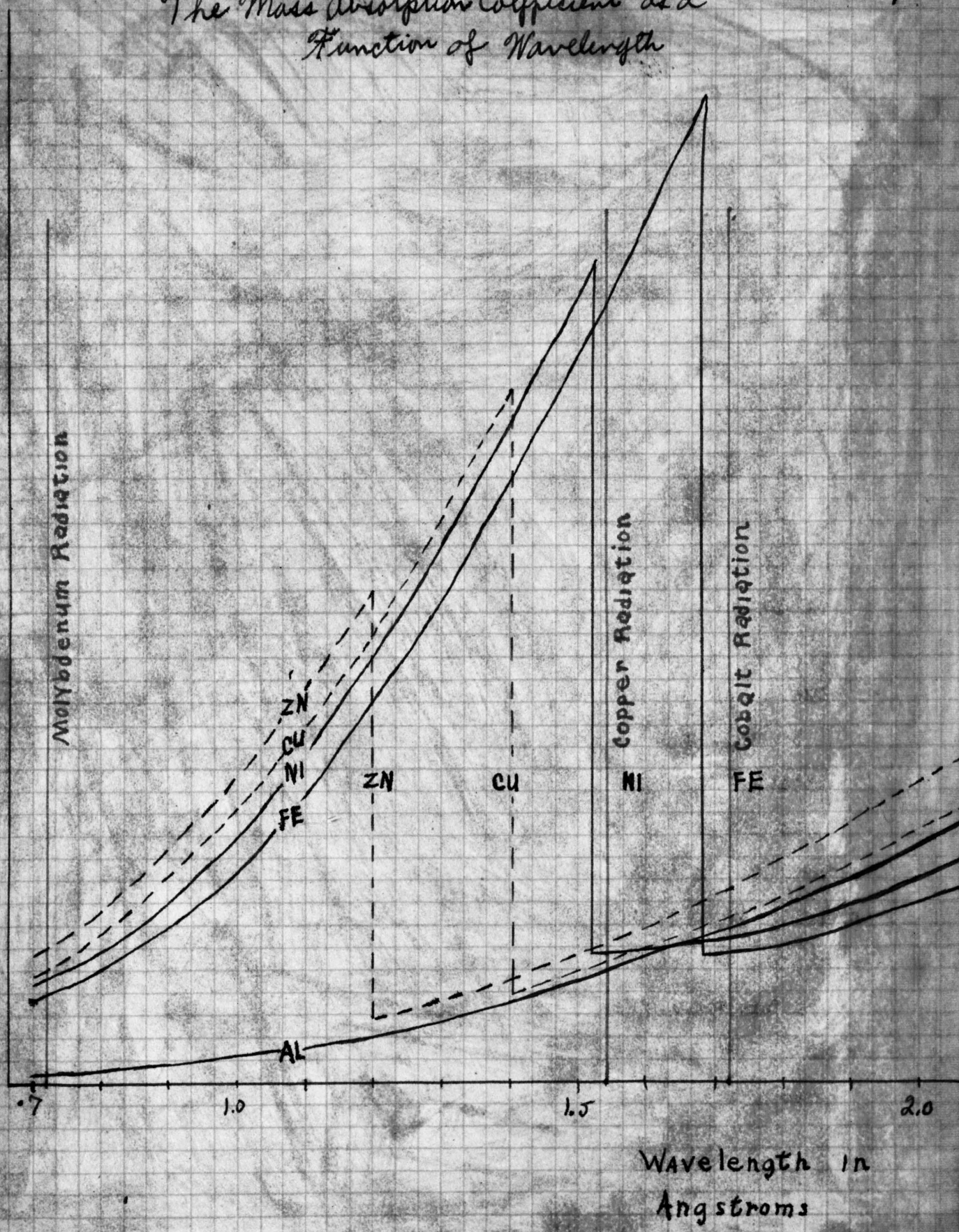
Here the ratio  $\frac{\mu}{\rho}$  is independent of physical state. As the equation now stands for a constant incident intensity and for a given sample, the only thing that will change  $I$  is a change in  $\frac{\mu}{\rho}$ . This ratio is no longer affected by a change of physical state, but depends only on the wavelength of the X-rays used and the element involved. It is the nature of this relation of  $\frac{\mu}{\rho}$  with different wavelengths which makes possible the determining of chemical elements by measuring the amount of absorption of X-rays.

The mass absorption coefficient increases rapidly with the wavelength along an exponential curve. However



# The Mass Absorption Coefficient as a Function of Wavelength

4/p



Wavelength in Angstroms



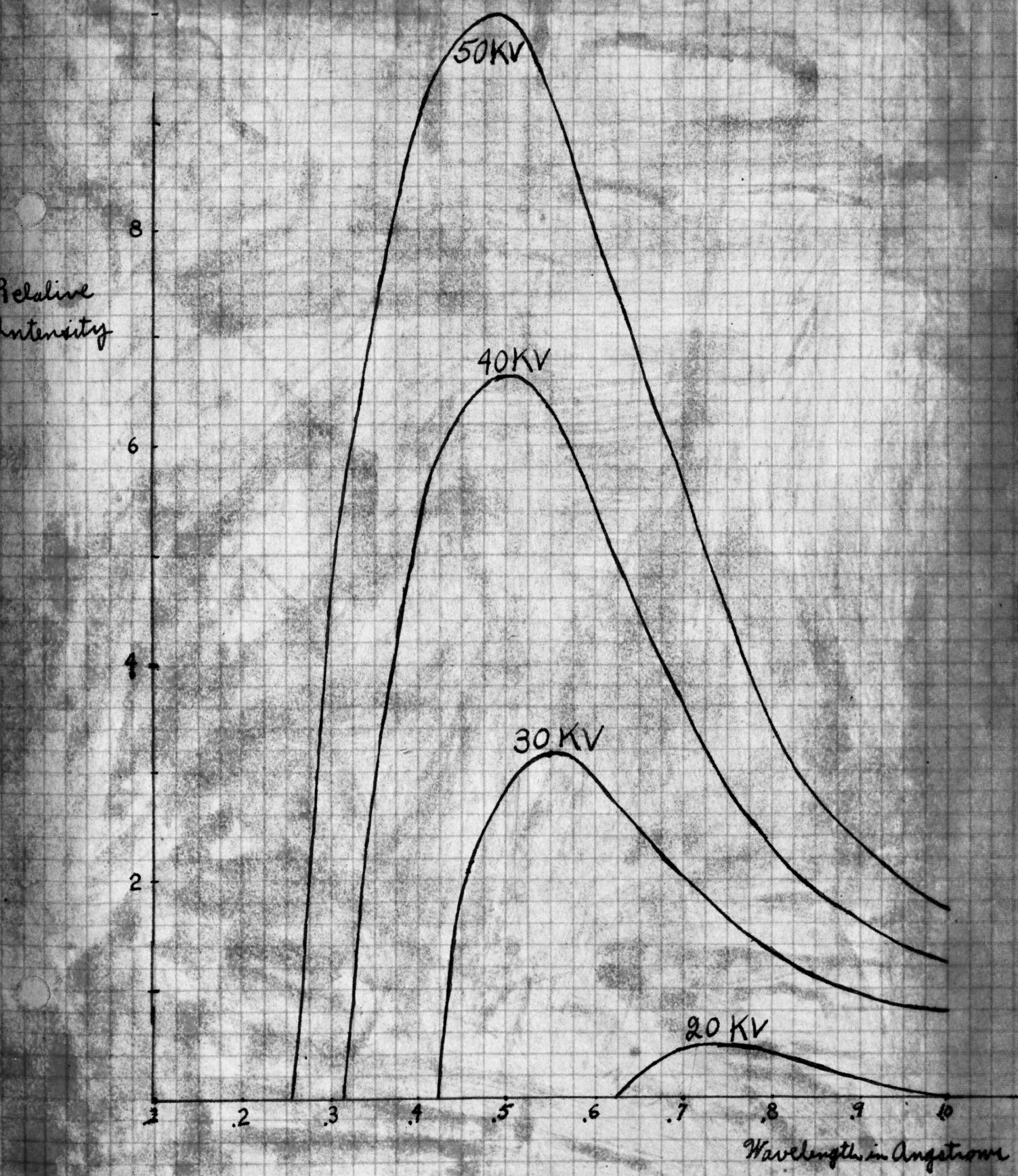
at certain critical regions for a slight increase in wavelength there is a sharp drop in absorption.

Figure 1 shows a graph of the mass absorption coefficient of several common metals plotted against wavelength. It can be noted at once that for a wavelength of one angstrom the five metals shown in the graph have mass absorption coefficients in the order of their increasing atomic numbers. As the wavelength increases past a certain definite value the mass absorption coefficient for zinc drops to a value close to that of aluminum. With further increases in wavelength the mass absorption coefficients of copper, nickel and iron in turn drop to values close to that of aluminum.

Theoretically the problem of determining the chemical nature of an unknown powder is simply a matter of determining the critical wavelength at which the absorption coefficient suddenly drops off. There simply would be no problem at all if a monochrometer capable of producing an intense beam of X-rays at any desirable wavelength was available. In general, however, a monochrometer does not yield a sufficiently intense beam of X-rays and is not at all suited to this type of an investigation. There are, however, two ways in which suitable radiation can be obtained.

The first method consists of using a tungsten target and by varying the voltage across it obtain some degree of variation in the wavelength of the maximum energy

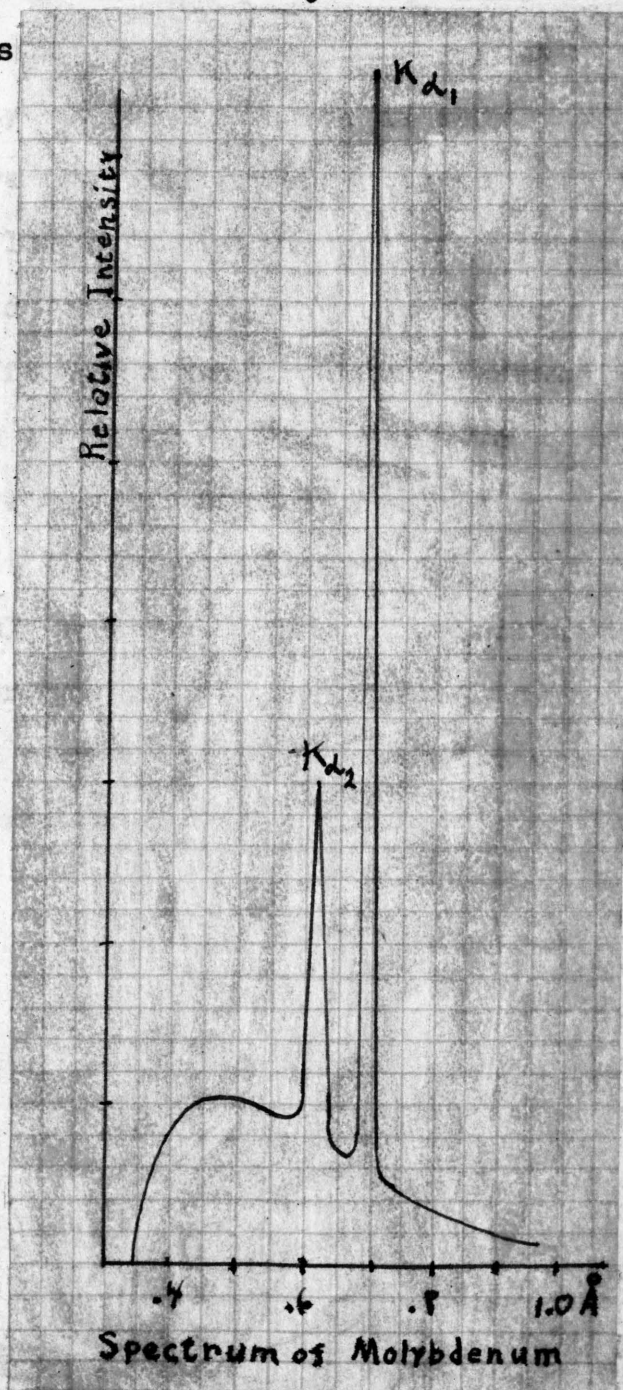
# Spectrum of Tungsten





region of the spectrum. Figure 2 shows a graph of the intensity as a function of wavelength for a tungsten tube operated at several different voltages. It can be noted that the region of maximum intensity shifts toward the shorter wavelengths as the voltage across the tube is increased. From Figure 1 it can be seen that the critical value for the common metals occur in the region between 1 and 2 angstroms wavelength. The intensity in this region is comparatively small. To filter out and use this end of the spectrum would require excessively long exposure times.

The second method of obtaining different wavelengths consists of using X-ray tubes having different targets.





The spectrum of most tubes is very different from that of tungsten previously illustrated.<sup>1</sup> Figure 3 shows the spectrum from a molybdenum target. With a suitable filter practically all radiation except the  $K_{\alpha}$  line can be filtered out leaving an almost pure monochromatic source. The intensity of the  $K_{\alpha}$  line is many times as intense as the rest of the spectrum so the exposure time is not too long. The wavelength for the  $K_{\alpha}$  line cannot be varied with an increase in voltage. Therefore to obtain a different wavelength a different tube must be used. Since tubes are quite expensive it is not possible to have many on hand. Thus the number of wavelengths that can be obtained is limited. Besides this a considerable amount of time is required to change the tubes. Disregarding the objections to this second method, it is more satisfactory and the one actually used in the experiment.

<sup>1</sup>This statement holds only for the region of the spectrum shown in the graph. At about seventy kilo-volts tungsten also gives a characteristic spectrum and looks similar to the molybdenum spectrum. This characteristic spectrum, however, has such a short wavelength that it is too penetrating to be of much value in this work.

## II Equipment

In general the three essential pieces of apparatus necessary to carry on an investigation in the field of microradiography are an X-ray unit, a suitable camera, and an enlarger. The first two of these requirements were easily and satisfactorily obtained. However, the physics department did not possess a suitable enlarger, so a workable model had to be constructed from available material.

The X-ray diffraction unit served admirably as a source of X-rays. The only difficulty involved along that line concerned the changing of tubes. Since all the tubes on hand were not of the same design special fittings had to be installed in order that tubes could be exchanged in a reasonable length of time. In all, three different X-ray tubes were available, containing copper, cobalt, and molybdenum targets respectively. All three tubes were used in the course of the experiment.

The camera shown in Figure 4 consisted of an aluminum plate four inches by five inches. A brass tube one and one-half inches long was mounted over a hole in the center. The specimen holder consisted of a brass tube two inches long. The camera was backed with two one-sixteenth inch sheets of bakelite. The inner one



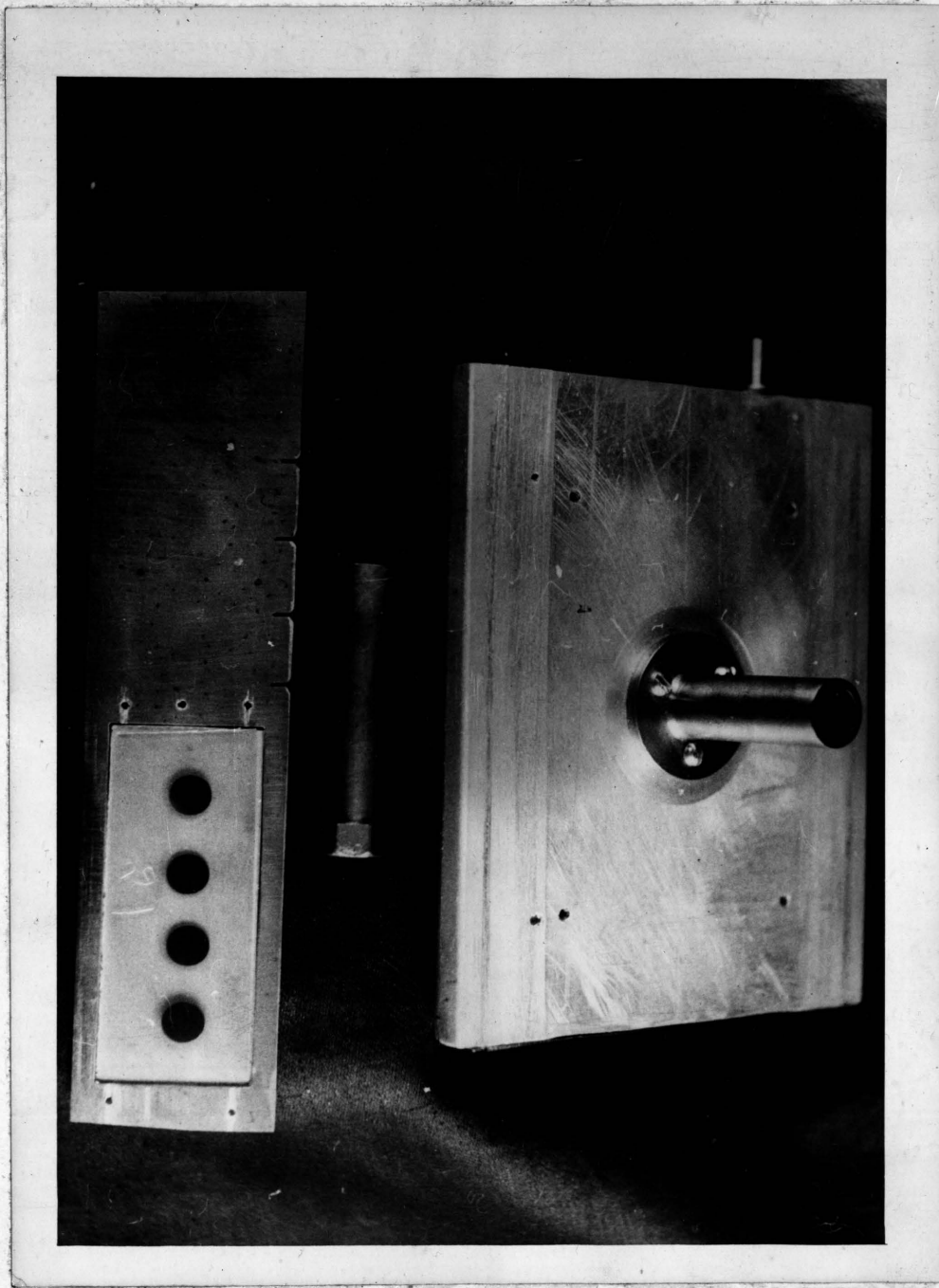


Figure 4. The camera, specimen tube, and the plate holder containing an exposed film.



contained a slot one and one-half inch wide so that the plate holder could be slipped into it. The plate holder, a strip of bakelite six inches long and one and one-half inch wide, contained four small slots so that it could be held in four different positions by the small catch at the top of the camera. In this way four exposures could be taken on one film. This number might easily have been raised to five or six without having the pictures overlap.

The specimen was fastened over one end of the specimen holder (the upper end as it is shown in Figure 4). The other end of the specimen holder was covered with a filter. This filter, besides limiting the X-rays entering the camera to the  $K_{\alpha 1}$  line, also kept ordinary light from entering the camera through the tube. In operation the specimen holder was placed into the tube mounted on the camera and pushed in until the specimen came in contact with the film. The camera was then mounted on the diffraction units as close as possible to the X-ray tube.

The powder specimen was prepared by mixing a small quantity of powdered metal with glue and allowing the mixture to harden. At first the powder and collodion were mixed on a piece of plate glass, then another smaller piece of glass was placed on top of the mixture; by varying the pressure on these two pieces of glass the

thickness of the specimen could be changed. After the collodion had dried, the glass plates were removed and a thin film of cellulose containing the metal particles resulted. This process was used for several of the earlier trial runs. It was found, however, that the collodion as it dried between the two pieces of glass had a tendency to form small bubbles of vapor in the specimen. The number of these bubbles was considerable and often they became quite large. Besides creating holes in the specimen these bubbles had a tendency to push the metal particles off to the side where they would become quite dense. In many cases they actually overlapped to such an extent that they were practically opaque to the X-ray beam. In practically all trials they were opaque enough to prevent an accurate analysis of the picture.

An attempt was made to smear on a piece of glass a mixture of collodion and metal powder. In this manner a bubble free specimen was obtained, but in general the dry collodion was so brittle it could not be peeled off from the glass. It was found that when "Duco Cement" was substituted for collodion the resulting smear could be removed with the aid of a razor blade provided the cement had not hardened completely. The best time to attempt to remove the specimen was found to be about a minute or



two after the smear had been made. In general the density of the metal in the smear was greatest at the center and less towards the edges. Since only a small portion of the smear was required for a specimen the proper density could be obtained by selecting a suitable section of the smear.

Once suitable specimens were obtained, no unusual difficulties were encountered in obtaining the X-ray photographs. Eastman Spectroscopic Safety film type 548-0 was used. Although the grain of this film could easily be detected under high power magnification, the grain size was much smaller than the actual size of the powder. For this reason a suitable magnification could be obtained without showing even as much as a trace of the film grain. At first the film was processed in Eastman Microdol developer. However, since the grain size of the film was much smaller than the powder granules photographed, and since the fine grain developer slowed down the speed of the film to a remarkable degree it was found much more advisable to use regular X-ray developer. The best exposure time for most specimens was around eight to fifteen minutes, with the tube operating at thirty-five kilovolts and fifteen milliamperes. This exposure time varied of course for the lighter elements such as magnesium and aluminum, but in general, about eight minutes was the average exposure time.



The chief difficulty involved the construction of an efficient enlarger to enlarge the X-ray photographs. The ideal device would have been a regular metallographic microscope. However, since the physics department does not possess a great deal of metallurgical equipment, such an instrument was not readily available. Several microscopes were on hand and supplied a good starting point for the construction of an enlarger.

The first enlarger constructed consisted of a light source, microscope, and plate holder mounted in line in that order. The X-ray photographs were placed on the stage of the microscope and focused on the plate holder. The enlargement of this arrangement was very good, but for some reason the resulting pictures lacked contrast. A series of changes in exposure and developing times did not seem to remedy this situation. The plate holder was then replaced with a Leica camera. The image could be focused on the ground glass with considerable precision. Since the camera used thirty-five millimeter film, if any sizeable portion of the X-ray picture was to be reproduced, the magnification had to be kept down; actually, the magnification obtained during this step was only about ten. These photographs were enlarged once more with a regular photographic enlarger which brought the total magnification up to about sixty. The resulting pictures showed a fair degree of contrast and a good deal of clarity,

considering that two separate focusing operations were involved in the production of each one. However, it was impossible to approach in any way a reproduction of the minute detail that was found on a visual microscopic examination of the X-ray photograph.

cause the specimens were too thick on the exposure time was incorrect. Many others were examined, although photographically satisfactory, did not exhibit any unusual characteristics not found in the majority of the pictures, and for this reason are not reproduced here. The microradiographs which are presented here give a representative picture of the results obtained through a visual examination of all the pictures taken.

The enlarged photographs do not possess the clarity gained by a visual microscopic examination of the original X-ray pictures. For this reason all the conclusions which can be drawn from an examination of the enlargements can be best and most fully supported by direct microscopic examination. In fact the interpretation of the X-ray photographs are all done visually. The enlargements shown here are presented to show as far as possible the basis for the conclusions reached. The only part to be kept in mind while comparing one group of enlargements of various magnifications in the enlargements. Due to a defect in the enlarger, the lower portion of all enlargements is darker than the remainder. For this reason it is well to compare



### III Experimental Results

During the course of the experiment about one hundred and seventy-five individual X-ray pictures were taken. Many of them were unsatisfactory because the specimens were too thick or the exposure time was incorrect. Many others when examined, although photographically satisfactory, did not exhibit any unusual characteristics not found in the majority of the pictures, and for this reason are not reproduced here. The microradiographs which are presented here give a representative picture of the results obtained through a visual examination of all the pictures taken.

The enlarged photographs do not possess the clarity gained by a visual microscopic examination of the original X-ray pictures. For this reason all the conclusions which can be drawn from an examination of the enlargement can be much more soundly supported by direct microscopic examination. In fact the interpretation of the X-ray photographs was all done visually, the enlargements shown here are presented to show as far as possible the basis for the conclusions reached. One thing must be kept in mind while comparing the color densities of various particles in the enlargements. Due to a defect in the enlarger, the lower portion of all enlargements is darker than the remainder. For this reason it is well to compare color

density from the same portion of the print, or from portions with the same shade of background.

The first pictures taken were of fine granules of a single metal. These pictures acted as standards by which the metals contained in mixtures could be ascertained. Figure 5 shows a microradiograph of a sample of powdered zinc. There is a wide range in the shade of the various zinc particles. Referring back to the equation derived in the introduction,

$$I = I_0 e^{-\mu/\rho \cdot \rho x}$$

one sees immediately the cause of this. The amount of incident radiation absorbed and thus also the shade of the photographed particles depend upon both the mass absorption coefficients and the mass traversed. In the case of the zinc granules the mass absorption coefficient is constant, therefore the difference in shade must be due to different sizes of particles.

On a close examination of Figure 5 one finds that this relationship holds for most of the particles. For example, the four particles numbered from one to four show a decrease in size and also a comparable decrease in intensity. This same property can be observed in any number of the particles. However, there are some exceptions in which it does not hold true. Particles labeled five and six are fairly small, yet they show up as very light spots; conversely, particle seven is relatively large and yet



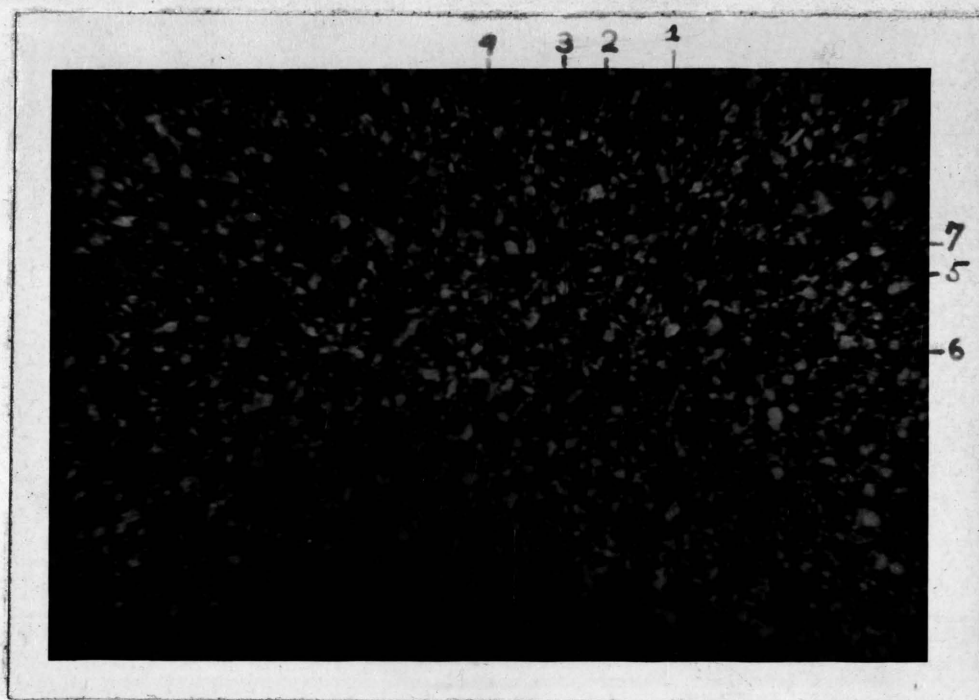


Figure 5. Microradiograph of powdered zinc taken with copper radiation. X60



Figure 6. Microradiograph of iron powder taken with cobalt radiation. X60

appears as a dim shadow. This can be explained on the assumption that particles five and six are long slender granules standing on end, while seven is a thin flat granule. Thus five and six present a small area and a great depth to the X-ray beam while seven presents a large area and small depth.

This variation of color density with size must be kept in mind while examining all succeeding photographs. Figure 6 shows a sample of powdered iron. Here too can be found the relation of size and color, as well as a few exceptions.

A second difficulty encountered in the study of powder not arising in the analysis of a thin foil is the change in color density within a single particle of a pure metal powder. This can be seen very well in Figure 7. The magnesium particles shown here were photographed with a rather short exposure, to bring out as much detail as possible. Still the particles do not show up too well. There is not much change in contrast between them and the background. In many of them there is a very considerable change in color density. For example, particle number one has a relatively large degree of whiteness in the central portion but shades off towards the edges to such a degree that it is almost impossible to distinguish its boundaries against the gray of the background. This same effect can be seen in most of the other particles in this picture.





Figure 7. Microradiograph of magnesium taken with cobalt radiation and given an exposure of two minutes at five milli-amperes. X60



Figure 8. Microradiograph of silicon taken with copper radiation. X60

It is due to the fact that the thickness of the individual particles varies considerably. More X-rays are absorbed in the thicker portions than in others.

One other drawback to powder analysis can be found in the magnesium picture. The particles indicated by the number two show a considerable change in color density due to overlapping. There is no way to prevent the occurrence of this overlapping, so it must also be taken into consideration when the pictures are interpreted.

Figure 8 shows the occurrence of this overlapping in a specimen of silicon powder. Several instances of the overlapping of particles are pointed out, and many others can be found on a closer examination.

The four microradiographs of different fine metal specimens described so far point out three of the difficulties involved in the analysis of a powder. The problem of this investigation was to find out if these difficulties could be taken into account or whether they would so confuse the interpretation that an accurate analysis of the chemical elements involved would be impossible. After all if different metals in the specimen are to be detected through a difference in absorption, three other factors which also affect the absorption can be considered a definite menace. Figures 9 and 10 illustrate very well this difficulty.



Figure 9 shows an enlargement of some pure chromium powder. Since most of these granules are larger than the iron particles used in a comparison in Figure 10, this discussion will be limited to only those rather small grains shown clearly in the open spaces. In general most of the small chromium particles are very opaque to X-rays and show up as distinctly white spots. The particles labeled one and two illustrate this point. There are, however, a few particles, three and four for example, which are fairly dim, and in all probability represent thin flakes. A look back at Figure 6 shows that iron particles of a size comparable with the small chromium particles are considerably darker. It seems reasonable on this account to expect to be able to detect iron particles from chromium by their color density.

Figure 10 shows a mixture of iron and chromium. Here again the discussion will be limited to the smaller particles. Thus the particles numbered one and two are fairly light and are probably chromium, while particles three and four are considerably darker and for this reason are probably iron. It is impossible to say with certainty whether a given particle is iron or chromium because in the pure chromium sample there are a few thin particles which are much darker than the ordinary particles. These particles could easily be mistaken for iron. Also in the pure iron powder there are a few particles light

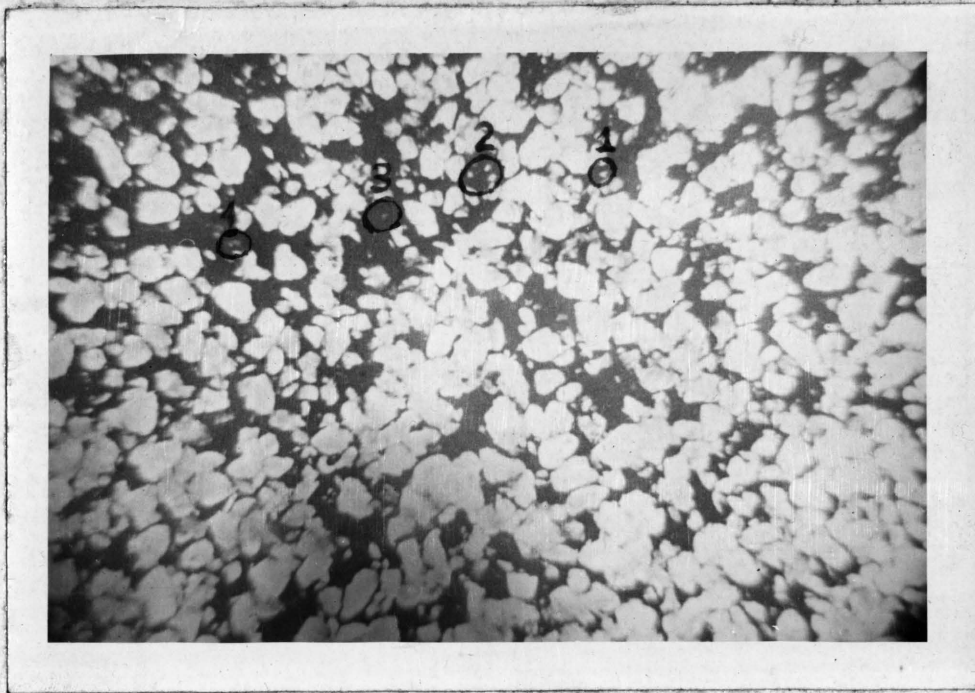


Figure 9. Microradiograph of chromium powder taken with cobalt radiation and given an exposure of five minutes. X60

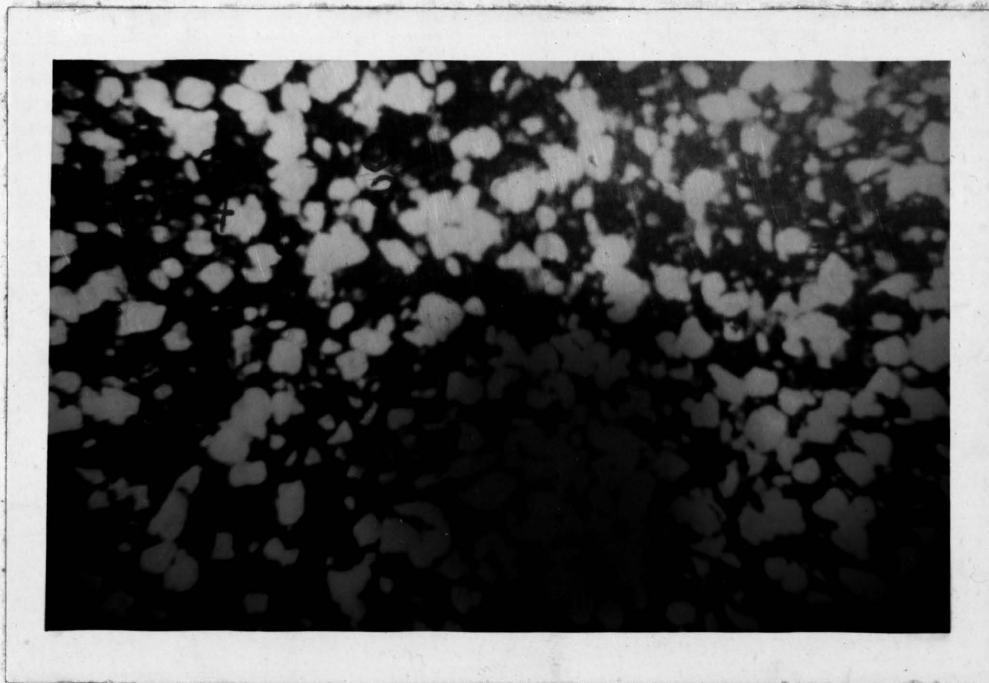


Figure 10. Microradiograph of chromium and iron taken under the same conditions as Figure 9.



enough to be mistaken for chromium in a mixture of the two metals. Thus in the mixture of iron and chromium one can only state with probability whether a given particle is iron or chromium.

The only possibility of obtaining conclusive results rests in the analysis and comparison of powders under different wavelengths. If this variation of particle size were great enough to overcome the differential absorption of X-rays under different wavelengths, powder analysis would be inconclusive. However, it was found that when the same mixtures of metal powders were photographed under different wavelengths of X-rays the chemical nature of a given particle could be determined with certainty.

Figures 11 and 12 show the same portion of a mixture of copper and aluminum taken with cobalt and molybdenum radiation respectively. On referring back to the graph showing the mass absorption coefficient as a function of wavelength one will find that for a wavelength corresponding to cobalt radiation the mass absorption coefficient of copper and aluminum are practically the same while at a wavelength corresponding to molybdenum radiation copper has about the same coefficient while that of aluminum has dropped to an insignificant value. In the picture taken with cobalt radiation one would expect to find copper and aluminum particles of the same size have the same color

Figure 12. Microphotograph of the same portion of aluminum and copper powder shown in Figure 11. All conditions were the same except cobalt radiation was used.



Figure 11. Microradiograph of aluminum and copper taken with molybdenum radiation. X60



Figure 12. Microradiograph of the same sample of aluminum and copper powder shown in Figure 11. All conditions were the same except cobalt radiation was used.



density, while in the molybdenum picture the copper particles should remain the same and the aluminum, now practically transparent to the X-ray beam should be nearly the same shade as the background. In this way by studying the two pictures it should be possible to tell definitely which particles are aluminum and which are copper.

This actually shows up very well in the photographs. In Figure 11 particle one represents a fairly light granule of aluminum. In Figure 12 this same particle is much darker. The same effect shows up in all the aluminum particles seen in the picture. In fact the particles labeled two and three in Figure 11 have become so transparent under molybdenum radiation that they do not show up at all.

The same results as obtained with the samples of copper and aluminum can be obtained with many other metals. The only limiting factor is the range of X-ray wavelengths available. Using the three tubes available and the more common elements, one can see from the graph that the same change in the absorption coefficient should occur in zinc as occurred in copper. Likewise a change in the absorption coefficient of iron should be readily found when photographed under copper and cobalt. Tests were made with mixtures of iron and nickel and iron and zinc. Both of these tests indicated very conclusively that the change in the absorptivity of iron was great enough

to enable a positive identification of the chemical nature of any specific metal granule.

An enlarged section of the iron and zinc specimen taken with cobalt radiation is shown in Figure 13; the same section photographed with copper radiation is shown in Figure 14. Under cobalt radiation iron and zinc should have the same coefficient of absorption. Figure 13 shows that all particles have a shade of gray depending upon their relative size. In Figure 14 there is a marked difference as many of the particles have practically disappeared when compared to the remaining ones which are very light. Under copper radiation the iron particles absorb much more radiation than the zinc and therefore appear much lighter than the zinc. Therefore any particle which is lighter under copper radiation than cobalt is iron whereas any particle lighter under cobalt radiation is zinc. The particle labeled number one is considerably lighter in Figure 14 under copper radiation and therefore is definitely iron. Particle two is much lighter under cobalt radiation and is therefore zinc. Particles three and four are very light under cobalt radiation and practically invisible under copper radiation definitely proving that they are zinc. Particles five, six and seven are much lighter under copper radiation and thus are iron. The chemical nature of any other particle can be ascertained by comparing its color



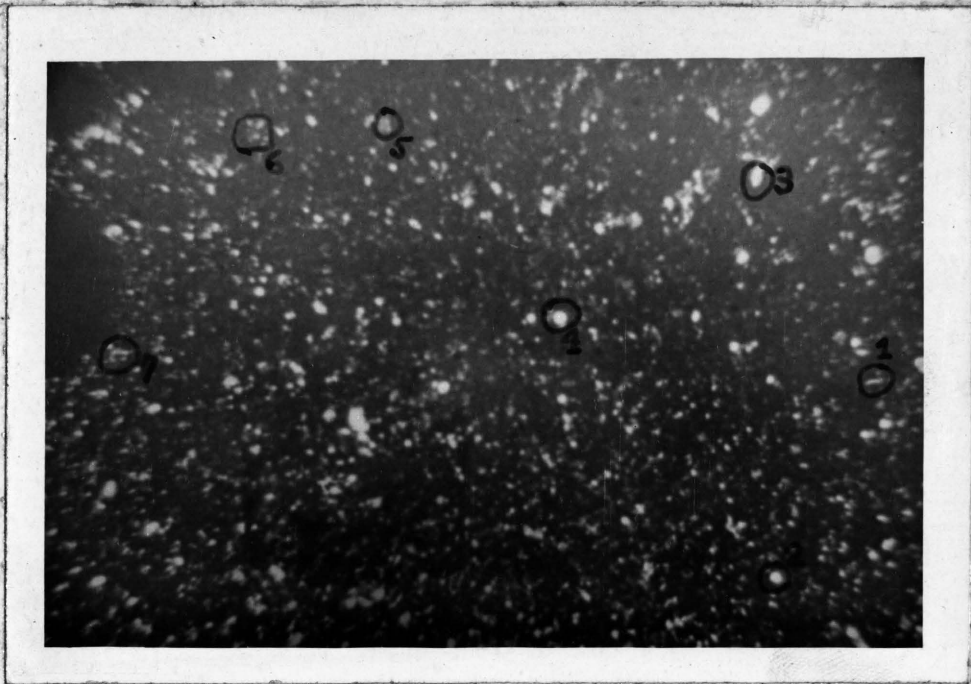


Figure 13. Iron and zinc photographed with cobalt radiation. X60

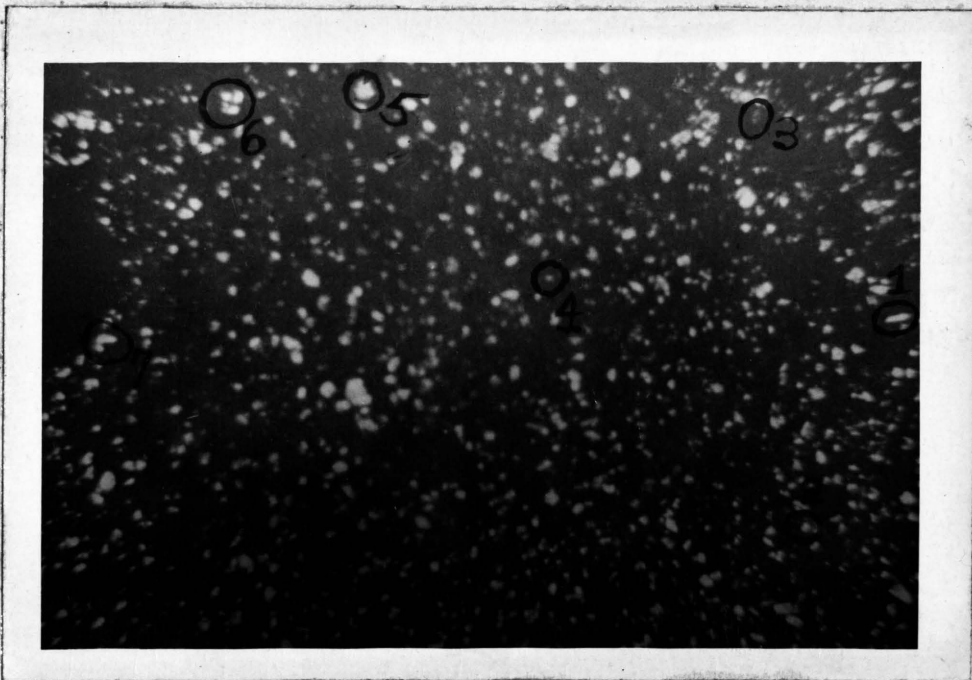


Figure 14. Same specimen shown in Figure 13 and taken under the same conditions, except copper radiation was used instead of cobalt.

in the two photographs. Since the two photographs are so strikingly different it is usually necessary to use a compass to locate the same particle on both pictures, by measuring its distance from two known particles.

In working with powders it was hoped that a method might be found of determining the chemical nature of a powdered alloy. In this respect not much satisfaction was obtained. The variation in thickness of the powder particles was great enough to overshadow any color variation due to the different absorption coefficients of the metals present. Figure 15 shows a good example of this phenomenon. The metal powder is an alloy of about ninety percent lead and small percentages of tin and antimony. Many of the particles show great variations in color but the fact that all lighter portions of each grain are lined up along the central axis of the particles shows that they are not due to a variation of the absorption coefficient but to different thicknesses of metal. For example, the particle labeled one shows how the thickness of the particle varied instead of the arrangement of the three metals present.





**Figure 15. Microradiograph of a powdered alloy containing lead, antimony, and tin.**

#### IV Conclusion

On the whole the results obtained in this experiment were very satisfactory. A powder specimen greatly simplifies the process of microradiography as compared to the regular thin foil method generally used and yet gives just as conclusive results. About the only precaution that must be observed in preparing a powder specimen is that the individual particles be spread out enough so that they do not overlap.

Several minor difficulties were encountered in the powder method which do not arise in the foil method. The overlapping of powder granules or a varying thickness in a single granule might lead one to wrongly infer that the resulting lighter area on the photograph was due to the inclusion of different metal. Also in the analysis of a powder, some long narrow granules standing on end tend to confuse the interpretation of a photograph. These difficulties, if kept in mind, do not present too serious a problem and can be overcome completely by analyzing the same section of a specimen under different wavelengths of radiation.

Photographs of corresponding sections of the same specimen when taken with different tubes show very conclusively the chemical constituents of the individual powder granules. In fact if the proper radiation is chosen by referring to the graph of absorption vs.



wavelength it is possible to obtain pictures varying very widely in appearance. One can obtain a picture where all particles have a color density corresponding to their relative size and another of the same section of the specimen where particles of one element practically disappear while those of other elements remain almost unaffected. In this manner any doubt due to the overlapping or varying thickness of the particles can be overcome.

In an alloy the various metals are so finely divided that they are indistinguishable when photographed in the form of a powder, at least to the extent that this investigation was able to go. In this case the variations in thickness completely mask the effect of the different absorption coefficients of the metals present. Although the results with alloys were not too satisfactory it may be possible that more favorable results could be obtained by using a finer grained film and a better and more powerful enlarger.

The technique developed in this work could be applied to good advantage in solving certain industrial problems of testing and control. Its most obvious application would be in the testing of a material used in the powdered form, such as testing the mixed ingredients for ceramic or plastic manufacture. Microradiographic testing could reveal the approximate percentage of each ingredient, the degree of mixing or the average particle size of any

single ingredient. Powder microradiography can also be applied to solid materials since it is much easier to prepare a powder specimen than a foil.

An attempt was made to measure the percentage composition of various metals present in a mixture from an enlarged microradiograph. This was to be done by taking photograph, cutting out all the particles of a particular element with scissors and weighing them on a chainomatic balance. The percentage of a metal present could then be obtained by comparing the weight of the pictures of that metal with the weight of the pictures of all particles present. There is little doubt that this method would under suitable conditions give an accurate quantitative analysis, but due to the fact that the enlarger used did not give sharp images of the individual particles it was impossible to carry out this test.



## V Summary

- I Powder specimens simplify greatly the work involved in obtaining a suitable microradiograph.
- II Several difficulties arise due to the variation of size, thickness, etc., and must be kept in mind when interpreting the enlarged photographs.
- III The proper selection of wavelengths of X-rays used enables one to obtain photographs which show very clearly the different metals present in the specimen.
- IV The results with powdered alloys were not very conclusive; more satisfactory results could probably be obtained by using a finer grained film and a better enlarger.

## Bibliography

1. Barret: Structure of Metals, McGraw-Hill Book Co., New York, 1943.
2. Clark: Applied X-rays, McGraw-Hill Book Co., New York, 1932.
3. Compton: X-rays & Electrons, D. Van Nostrand Co., New York, 1926.
4. Compton & Allison: X-rays in Theory & Experiment, D. Van Nostrand Co. Inc., New York, 1935.
5. Maddigan, Industrial Radiography, 4, 22, 1946.
6. Richtmyer & Kennard: Introduction to Modern Physics, McGraw-Hill Book Co., Inc., New York, pp. 464-596, 1942.
7. Stranathan: The "Particles of Modern Physics, The Blakiston Co., Philadelphia, pp. 260-314, 1942.



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