

A SPECTROGRAPHIC STUDY OF COPPER-TIN  
ALLOYS

A THESIS

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Master of Science in Chemistry

by

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Date Approved by Chairman 9/4/50

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I PURPOSE OF THE INVESTIGATION

Hasler and Kemp<sup>1</sup>, also Spicer<sup>2</sup> have reported that copper alloys can be analyzed for copper by the variation of the ratio of intensities of a pair of copper lines--one produced by a transition to a ground level (arc) and another, to an excited level (spark). In both cases the method of analysis broke down when the alloys reached a copper content of less than 75% by weight of copper<sup>3</sup>.

Hasler and Kemp, working with copper-aluminum alloys, used the ratio of the 2882.9 A° copper line to the 2441.6 A° copper line and obtained a linear correlation of the log. of this ratio with the copper content in the range 95% to 75% copper. No attempt was made to explain their results.

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<sup>1</sup>M. F. Hasler and J. W. Kemp, J. Optical Soc. Am. 34, 21 (1944)

<sup>2</sup>W. M. Spicer, Anal. Chem., 20, 557 (1948)

<sup>3</sup>In the case of the nickel this percentage was considerably higher, 90%.

Spicer, working with the copper-zinc and copper-nickel alloys, found that better results could be obtained in using the ratio of the 2884.4 A<sup>0</sup> copper line to the 2882.9 A<sup>0</sup> copper line. Spicer noted that a linear correlation between the log. of the ratio of spark line to arc line and the percentage composition existed only on certain portions of the working curve and a decided broad minimum ratio existed for alloys of certain composition. The ratio increased when the copper content was above or below a specific value. The ratio minimum occurred in a neighborhood in which the thermal conductivity also was observed to be a minimum, when the latter was plotted as a function of the percentage composition. Spicer suggested that a possible explanation for the phenomenon might be found on the basis of thermal conductivity. If the thermal conductivity of the alloy is low, the temperature created at the surface of the alloy by the spark would be high, due to the inability of the alloy to dissipate the heat. This high temperature would give rise to a strong arc line, and if it can be assumed that this heat is insufficient to produce any change in the intensity of the spark line, the ratio of the spark to arc line would be lower than the ratio which would be calculated from the spectrum of an alloy of high thermal conductivity.

Langstroth and Brown<sup>4</sup>, reporting in the Canadian

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<sup>4</sup>G. O. Langstroth and W. W. Brown, Can. J. Research, 20 A, 173 (1942)



Journal of Research, ran a series of spectral studies of salts of various metals. Their procedure was to deposit on pure copper electrodes various amounts of the metal ions by evaporation, in a high vacuum, of a known volume of chloride salt solution. A spectrogram was then made under spark conditions, and the relative intensity of two lines of the metal, one arc and one spark, was plotted as a function of the amount of the salt present on the electrode per unit area. The working curves thus obtained bore a noticeable resemblance to the working curves found by Hasler and Spicer; the exception being that in the case of the salts no minimum could be obtained, rather a ratio was approached asymptotically. Langstroth and Brown attributed the effect as being due to partial absorption, mainly within the limits of the discharge, of the arc line. The greater the concentration of the metal, the greater the absorption of the arc line, and consequently the greater the ratio of the intensity of spark to arc line. They further stated that below a certain critical concentration, the absorption effect became relatively unimportant.

It was desired to perform an experiment which would extend the results already found for copper-aluminum, copper-nickel, and copper-zinc alloys to the copper-tin system and in addition to test the suggestions of Spicer and Langstroth. The copper-tin system was chosen because of the relative

complexity of the phase equilibrium<sup>5</sup> which would, in turn, indicate rather irregular thermal conductivity.

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<sup>5</sup>International Critical Tables, Vol. II, (New York: McGraw-Hill Book Company, 1929), p.433.

## II APPARATUS

The experimental equipment used in this work was a grating spectrograph<sup>6</sup>, multisource unit<sup>7</sup>, and a comperator-densitometer<sup>8</sup> all manufactured by A.R.L. Dietert. The range of 2300 A° to 4300 A° is covered by the spectrograph with an uniform dispersion of 7 A° per m.m. in the first order.

The spectra was obtained under the following conditions:

## Multisource

Capacitance	2 microfarads
Inductance	50 microhenries
Resistance	0.4 ohms
Output voltage	940 volts
Prespark	various
Exposure	14 seconds
Initiator	continuous

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<sup>6</sup>M. F. Hasler and H. W. Dietert, J. Optical Soc. Am., 33, 218 (1944)

<sup>7</sup>M. F. Hasler and F. W. Kemp, J. Optical Soc. Am., 31, (1941)

<sup>8</sup>H. W. Dietert and J. A. Schuch, J. Optical Soc. Am., 31, 54 (1941)

## Spectrograph

Spark-gap	3 m.m.
Slit width	60 microns
Positive electrode	upper and alloy
Negative electrode	0.25 in. diameter tapered to hemispherical tip.

## III PHOTOGRAPHY

The photographic film used in this study was Eastman Spectrum analysis No. 1 and is recommended by the manufacturer for work in the ultraviolet region. After proper exposure the film was processed in an A.R.L.-Dietert automatic agitating developing machine<sup>9</sup>. The film, being developed for three minutes with D 19 developer, short-stopped in a 3% acetic acid solution for thirty seconds, and fixed in Eastman X-ray fixing solution for one minute, was washed for five minutes in fresh running water and then dried with an infrared film drying machine.

The calibration of the film was carried out by means of a two-step filter technique<sup>10</sup>. A gamma of 1.8 was obtained in the region 2850 Å<sup>o</sup> to 2950 Å<sup>o</sup>. No background correction was necessary.

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<sup>9</sup> J. Schuch, J. Optical Soc. Am., 32, 116 (1942)

<sup>10</sup> J. R. Churchill, Ind. Eng. Chem. Anal. Ed., 16, 653 (1944)

## IV EXPERIMENTAL

For the purpose of this experiment a group of copper-tin alloys were prepared from chemically pure copper and tin. The melts were fused in an induction furnace and each sample cast into a bar and also into a 1/4 in. rod. The bars were machined into flat slabs about 1/8 in. by 1 in. by 5 in. The bars and rods were allowed to stand over a period of time of approximately three years.

To obtain a spectrogram of the bars, they were mounted on a petrey stand<sup>11</sup> and arced, using a 1/4 in. graphite counter electrode with a hemispherical tip. The alloy was made positive. To clean the plates a fine grade of emery cloth was used, and the sample was carefully wiped before using with a clean piece of cotton flannel.

The rods were cleaned on a device described by Varner<sup>12</sup> which consisted of a rotating piece of fine sandpaper mounted on the end of an electric motor. The rods were, after sanding, also cleaned with a piece of clean cotton flannel to remove any silica adhering to the surface of the rod.

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<sup>11</sup> H. V. Churchill and J. R. Churchill, J. Optical Soc. Am., 31, 611 (1941)

<sup>12</sup> J. E. Varner, Master's Thesis, Georgia Institute of Technology, 1949, p. 5.

Though the plates seemed to be solid throughout, the rods contained many air bubbles trapped in the rod during its casting. The same type of counter electrode was used in the case of the rods as was used with the plates.

The analysis of the alloys was furnished by a commercial laboratory.

The lines used in the investigation are described by the M.I.T. Wave Length Tables as follows<sup>13</sup>:

line	arc	spark
Cu 2882.934	3	-
Cu 2884.383	-	30

The exposure time for all samples, plates or rods, was 14 seconds, and the density of the lines on the photographic plate was controlled by a group of filters on the spectrograph.

The room in which this experiment was carried out was maintained at a constant temperature of 79° F by a Carrier air conditioning unit.

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G. R. Harrison, Massachusetts Institute of Technology Wave Length Tables (New York: John Wiley and Sons, 1939)

## V RESULTS AND DISCUSSION

The results of this experiment indicate that the analysis of copper-tin alloys can be effected, plotting the ratio of the intensity of a copper spark line to a copper arc line against the percentage composition of the alloy. The method can be applied to alloys in the composition range of 100% copper to approximately 75% copper. The accuracy of the method is greatly affected by the period of the presparking as is indicated in table I. A noticeable curve shift is to be found, although the room in which the experiments were carried out was maintained at a constant temperature. This curve shift manifests itself not only in a displacement but also on a small distortion of the working curve as shown in figure 1.

Changes in the length of presparking will change greatly the shape of the working curve. Figure 2 illustrates to what extent this is true. If the method is to be used as an analytical device, it is recommended that a long prespark be used and that standards and unknowns be run, as near as possible, at the same time without turning off the multi-source unit between runs. In addition, the samples should be exposed the same length of time, using filters to determine the density of the lines on the photographic film. If the forementioned recommendations are carried out, the method is capable of excellent reproducibility and compares favorably



with other methods of spectrographic analysis<sup>14</sup>.

The experiments performed for this work indicate that the thermal conductivity plays a small, if not negligible, role in the determination of the working curve. In the system, copper-tin between 100% and 60% copper, there is to be found a great complexity in the phase equilibrium diagram<sup>15</sup>. A large number of solid solutions are found and, in addition, a compound formation. Although no reliable data could be found for the thermal or electrical conductivity for the system in this region of composition, it can be safely assumed that the conductivity would not be a smoothly concave function of the composition as in the case of copper-nickel and copper-zinc. The fact that the working curves did have the same general appearance as the copper-nickel and copper-zinc alloys<sup>16</sup> indicates the lack of influence upon the working curve of the thermal conductivity.

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<sup>14</sup>The line pair used in this work cannot be used if the silica content is high, due to the interference of the 2881.578 Å Si line.

<sup>15</sup>International Critical Tables, loc. cit.

<sup>16</sup>W. M. Spicer, loc. cit.

Spicer found, and this work confirmed, that copper alloy rods gave a lower ratio than the corresponding plates of the same weight percent copper. The explanation, based on a consideration of thermal conductivity, would be that the smaller area of the rods would dissipate the heat slower than the large area of the plates. Thus the rods would give rise to a greater intensity of the arc line, and the ratio of spark to arc would be smaller than in the case of the plates. However, the smaller ratio in the case of the rods can be explained by yet another method which does not depend upon the thermal conductivity.

The experiments here reported were run with the alloy made positive, and in the cases of presparking, considerable quantities of graphite from the counter electrode deposited on the surface of the alloy. These deposits were, for the most part, hard and firmly attached to the surface. The graphite, in the case of the rods, must build up on a very small surface area while, in the case of the plates, a larger surface is available and thus the deposited graphite concentration per area is smaller. Since the spark strikes only the surface of the alloy, the deposited graphite will have the effect of showing less copper in the case of the rods where the graphite concentration per unit area is larger than in the case of the plates where this graphite concentration is small.

To demonstrate the validity of this line of reasoning, several experiments were carried out. A 100% copper rod was run first, using a 14 second exposure with no prespark, next using a 15 second exposure with no prespark made by exposing each frame of the film three times for five seconds, cleaning between each five second exposure, and lastly, an exposure of 15 seconds with no prespark formed by exposing each frame five times for three seconds with cleaning in between sparking. The results were in agreement with the conclusions which would result if the deposition of the graphite upon the surface of the alloy was causing the difference in the ratio of spark to arc lined between plates and rods of the same composition. The ratio is increased from 0.61 in the first case, to 0.71 in the second, 0.76 in the third, and 0.78 in the fourth.

These results taken together with the fact that there is no appreciable change in the ratio between a rod which has been cooled in liquid nitrogen and one which has been heated on a hot plate - i.e., if it is dependent on the thermal conductivity it is a sort of microscopic thermal conductivity<sup>17</sup> - indicate that thermal conductivity does not play a major role. To further confirm this conclusion it was decided to run an experiment in an inert atmosphere.

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<sup>17</sup>W. M. Spicer, Unpublished works.

The reason for this was that it was felt that oxidation affected the results somewhat. This will be discussed in more detail later in this chapter.

To provide an inert atmosphere a small round bottom boiling flask, its neck broken at the spherical portion of the flask to provide a light opening, was fitted with two holes just large enough for the admission of the electrode and counter electrode, and a delivery tube was connected to a supply of helium. A positive pressure was maintained through the system, and the gap was thereby continually swept with the helium. A larger spark gap, 5 m.m., had to be used for this experiment as the atmosphere of helium decreased the resistance across the gap to such an extent that the circuit breaker was thrown in the multisource unit if a 3 m.m. gap was used. A series of spectrograms were made with a 35 second exposure<sup>18</sup> without cleaning the alloy electrode between the various runs.

The ratios decreased from a value of 0.81 to 0.70 as the graphite deposited on the alloy. Apparently a sort of equilibrium amount of graphite eventually deposits on the alloy as the ratios, after falling, steady to a random distribution about a given mean. It was found to be impractical

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<sup>18</sup>Note that the exposure time had to be more than doubled to produce a line which could be read on the densitometer.

to run the series of alloys in the helium atmosphere as the spark did not confine itself, on alloys below 95%, to the neighborhood of the gap, but it played all over that portion of the alloy which was in the helium atmosphere.

Further evidence of the lack of effect of the thermal conductivity was illustrated by yet another experiment. If the deposition of the graphite upon the alloy is the major factor in determining the difference in the ratios between plates and rods of similar composition, it seems likely that if the spark were to be confined to the same area in the case of both the rod and plate, then there should be no appreciable difference between the ratios found for the rod and the plate. To test this conclusion the 1/4 in. graphite counter electrode was replaced by a 1/8 in. one which was gradually tapered to a sharp point. This counter electrode was used for sparking the plate and the rod, and it was found that there existed no appreciable difference in the ratios for the plate and the rod.

The complete picture is not, however, as simple as has been outlined above. The description is made more complex due to the gases present in the atmosphere, presumably the oxygen. Calker<sup>19</sup> noted that no suitable working curve could be found for the analysis, in the normal manner of copper-

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<sup>19</sup>J. van Calker, Spectrochim Acta, 2, 340 (1944)

arsenic alloys when the alloys were used as both the electrode and the counter electrode. The irregularities in the plot were attributed to the formation of copper oxide on the surface of the alloy.

To correct for this he found that a counter electrode of graphite hindered the formation of the oxide film and thus made possible a working curve suitable for the analysis of the copper-arsenic alloys. The fact that even when a graphite counter electrode is used there is some reaction of the alloy electrodes with the components of the air has been noted in this work. For when a 100% copper rod is run, using a 14 second exposure time with no presparking and with no cleaning of the alloy between various runs, the ratios start from a high value of 0.70 and then fall to a value which remains relatively constant at 0.55; while with an alloy which has a composition of 85% copper, 15% tin, when treated in a like manner, the ratios begin at a low value of 0.30 and rise to a steady value of 0.41.

It will be noted from a consideration of the free energies of the reduction of the oxides of copper and tin by carbon, that the reduction can be carried out on copper oxide but not on tin oxide. The formation of tin oxide thus tends to increase the ratios produced by the alloy. With this in mind, it is now possible to explain the minimum ratio found in this work, and also in the work of Spicer, when the alloys are exposed with a prespark.

It will be noted in figure I that the minimum ratio is found only in the case where the sample was first presparked before exposing. In the case of the longer prespark, this minimum was approached more rapidly than in the case of a short prespark. The minimum disappeared entirely when no presparking was used. The effect of presparking is thereby indicated to be dual in nature. On the one hand, the graphite is being deposited upon the alloy, and as presparking is increased the ratio decreases due to the increase of the concentration of the graphite per unit area in the surface of the alloy.

The second effect is the tendency of the ratios to increase with presparking due to the reaction of the tin in the alloy with the air, at arc temperatures. This effect becomes greater as the tin content is increased, and even at a content of 15% tin this effect is sufficient to overcome the influence of the graphite coating; moreover, when the tin content is 35%, and a very long prespark is used, this latter effect completely overshadows the effect of the graphite. It can be noted from the plots of the working curves that when the alloy has a composition of about 90% copper, the two opposing reactions very nearly balance one another. It can be clearly seen from an examination of the alloy after sparking that the amount of graphite deposited on the electrode decreases with the decrease of the amount of copper in the alloy, and a white powder - most probably an oxide of tin - begins to

form on the alloy as the tin content is increased.

There is a possibility that the effect of the oxidation of the tin is to merely decrease the amount of graphite deposited in the alloy by the formation of a surface to which the graphite will not adhere. Thus the two effects, noted above, would stem from a single common cause.

In an effort to determine the degree of influence of the thermal conductivity, a 1/4 in., 100% copper rod was drilled, leaving only a very thin shell. This rod was arced and the intensity ratio compared with that of a solid copper rod of the same outside dimension. The ratios calculated for the two rods were not the same. However, a test of significance was made, and this indicated that the difference between the two ratios was not significant.

If the above picture can be accepted as a valid description of the system, a complete explanation can be made by referring to the work of Langstroth and Brown<sup>20</sup> in their work with salts of various elements. They stated that the ratio of certain line pairs, one spark and one arc, was dependent upon the amount of the element introduced into the arc source. They attributed the occurrence as due to partial absorption, mainly with the limits of the discharge, of the line formed by the transition to a low electronic level.

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<sup>20</sup>G. O. Langstroth and W. W. Brown, loc. cit.



The absorption was believed to be directly related to the amount of material, and when the material fell below a certain concentration the absorption effect on the arc line became unimportant. They further stated that partial absorption of lines having a final level considerably above the ground state must be relatively unimportant.

Though the experiments in this work in no way prove the suggestion offered by Langstroth and Brown, they are compatible with the conclusions of their work, if one takes into additional consideration the effect of the graphite film on the alloys formed during sparking by the use of a graphite counter electrode.

Calder in another article entitled, "An Attempt to Establish an Absolute Quantitative Spectral Analysis"<sup>21</sup>, has developed a method for the analysis which seems to be very similar to the method reported in this work. He states that the intensity ratio of a line pair of the same element will change depending on the concentration of the element if the line pair consists of a "resonance line" and a line which is "without, or with very little, self-absorption". He goes further to show that the working curve for the analysis of an element for one matrix element may be used for the analysis of the same element in another matrix if the ratios are multiplied by a uniquely determined constant for the particular

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<sup>21</sup>J. van Calder, loc. cit.

matrix element. He found the method was valid for the analysis of bismuth in a lead and also in a zinc matrix. No attempt was made to confirm these experiments; but in view of the complicating factors found when copper is the major component, it would seem that the method might not be extensible, in practice, to cases where the matrix elements vary considerably in their chemical properties.

It was originally desired to extend the analysis of the copper-tin system from 100% copper to 100% tin, but this proved impractical. When alloys of less copper than 60% were attempted, the Cu 2884.4 line became very broad and diffuse, seeming to almost split into two lines, though no interference could be found in the M.I.T. Wave Length Tables. The background also became very objectionable and no agreement between the ratios produced by the same alloy could be obtained, even when a background correction was made.

In all of the above experiments the alloy was positive. If the alloy was made negative and the graphite counter electrode positive the relative densities of the two lines became interchanged. In the first case the arc line was always of a greater intensity than the spark; while if the alloy was negative, the spark lines became the more intense. For this reason and also due to the heavy formation of copper oxide, no conclusive results could be obtained when the alloy was used for both upper and lower electrode.

It must be mentioned that the electrical conditions under which an analysis of copper alloys can be carried out are most critical. Such factors as analytical gap separation and rotary spark gap distances must be controlled. The operating voltage, in these experiments 940 volts, must be controlled somewhat accurately; although a variation of some ten to twenty volts does not appreciably affect the results. The settings of the inductance, resistance, and capacitance used in these experiments were found most suitable, giving less background and sharper lines than other conditions.

To obtain ratios which are reproducible, the surface of the alloy must be in the same condition for each exposure<sup>22</sup>. This can be accomplished in one of two ways. First, the alloy can be carefully cleaned between each exposure, or secondly, the alloy must be arced a sufficient length of time before starting exposures to ensure that the surface will not change with succeeding arcings. This can be accomplished by continuous arcing for approximately two minutes before exposing the film for the first time. Each succeeding exposure should be made as soon after the preceding exposure as possible. The latter method is found to give results which are more reproducible.

If the method is to be used for analytical work, one must repeat accurately the exposure conditions as nearly as

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<sup>22</sup> For results when this is not true, one is referred to figure 3.

possible, for great differences in the appearance of the working curve can result when the exposure or presparking time is changed.

This method of analysis shows promise as an analytical device, as large percentages of a component may be analyzed with comparative ease. Also, as is pointed out by Calker, this method may lead to a reliable absolute method of spectrographic analysis. In view of these fruitful prospects, which are in general the prime drawbacks to a spectrochemical analysis, one could justify much future research. A most desirable investigation would be to determine the optimum conditions under which an analysis should be carried out. To do this effectively, experiments carried out with a voltage source capable of a highly controlled output voltage and current coupled with well interpreted oscillographic studies of the arcing current are recommended.

After the best working conditions have been found, the next problem which immediately presents itself would be correlating the works of Langstroth, Calker, and Spicer, and the extension of the results of these experimenters to other alloys and salts. A careful study should be made of the line pairs which are suitable for this type of analysis. This might lead to a modification or revision of the explanation which has been suggested by Langstroth.

## SUMMARY

It has been found possible to spectrographically analyze alloys of the copper-tin system satisfactorily in a composition range 100% to 65% copper by a comparison of the ratio of the intensity of two copper lines. The two lines found to give satisfactory results were the Cu 2882.9 A<sup>o</sup> arc line and the Cu 2884.4 A<sup>o</sup> spark line. The working curve has been found to be highly dependent upon the period of presparking. In this method of analysis a shifting of the working curve has been noted, which is common to most methods of spectrographic analysis.

The results of experiments performed indicate that the method is little dependent upon the thermal conductivity, and the cause of the change of intensity ratio with the composition of the alloy might be found in the self absorption of the arc line. The ratios are believed to be somewhat dependent of the graphite which is deposited on the alloy during the sparking process.

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APPENDIX  
TABLES AND GRAPHS

TABLE I: Chemical Analysis of Alloys used in Investigation

Sample	percent Cu
100	100.
98	97.82
95	94.05
90	89.85
85	86.03
80	80.05
75	74.52
70	69.
65	64.



TABLE II. Ratios for Alloy Rods and Average Deviation with various Prespark

Sample	No Prespark		10 sec. Prespark		Long Prespark	
	Mean	Av.Dev.	Mean	Av.Dev.	Mean	Av.Dev.
100	0.84	0.02	0.58	0.03	0.53	0.01
90	0.36	0.02	0.31	0.01	0.37	0.01
80	0.32	0.02	0.25	0.02	0.03	0.01
75	0.31	0.01			0.40	0.01
70	0.30	0.01	0.26	0.01	0.52	0.01
65					0.68	0.02

TABLE III: Ratios for Alloy Plates and Average Deviation

Sample	Average Ratio		Average Deviation	
	1	2	1	2
100	0.81	0.80	0.03	0.01
98	0.58	0.60	0.01	0.01
95	0.47	0.48	0.02	0.01
90	0.41	0.43	0.01	0.01
85	0.38	0.40	0.01	0.02
80	0.34	0.39	0.02	0.01
75	0.33	0.37	0.02	0.01

TABLE IV: Ratios with Continuous Sparking

Ratios for 100% Cu	Ratios for 85% Cu
0.70	0.30
0.73	0.34
0.67	0.36
0.63	0.38
0.58	0.41
0.57	0.40
0.54	0.40
0.55	0.42

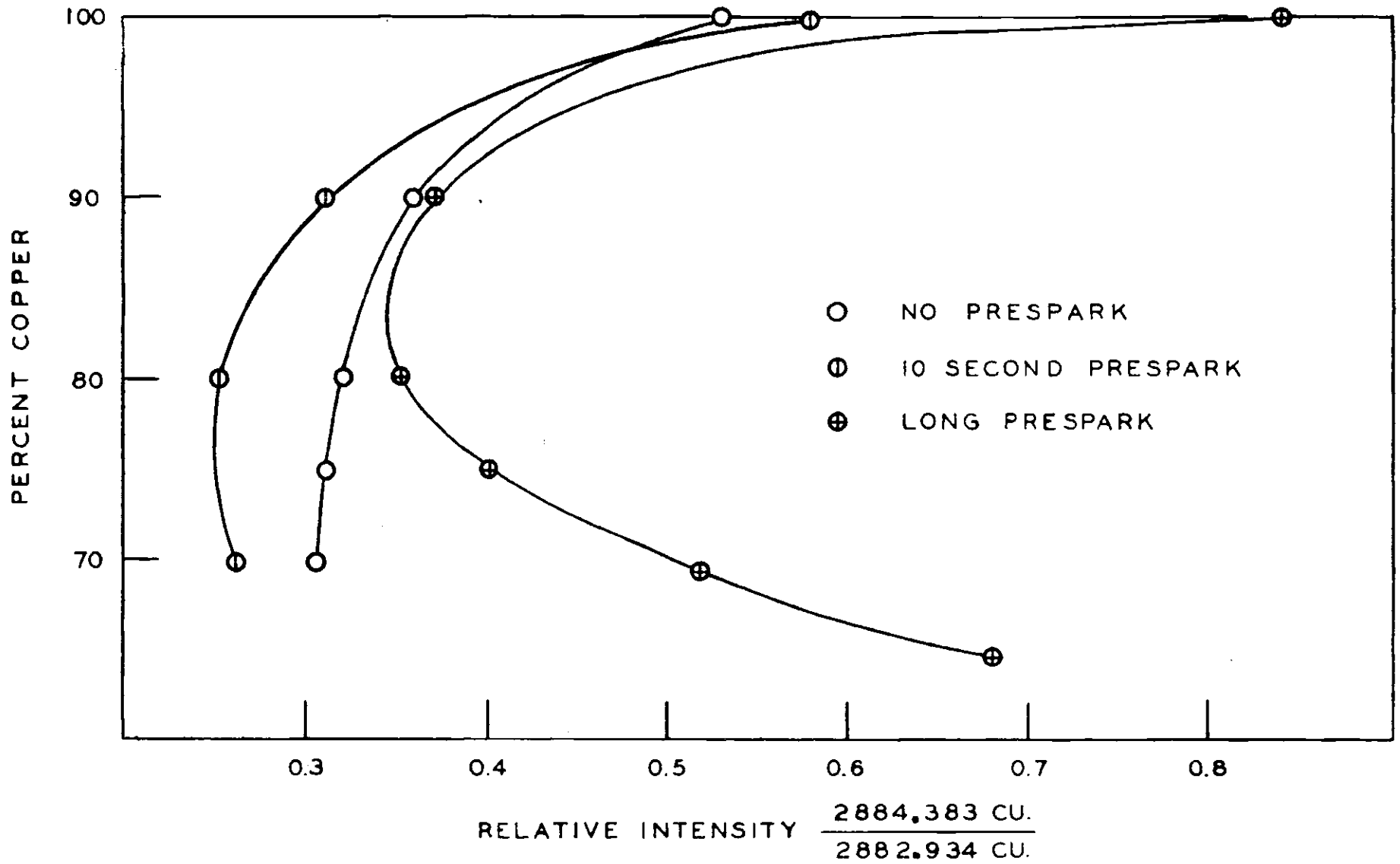


FIGURE 1 - WORKING CURVE FOR ALLOY RODS

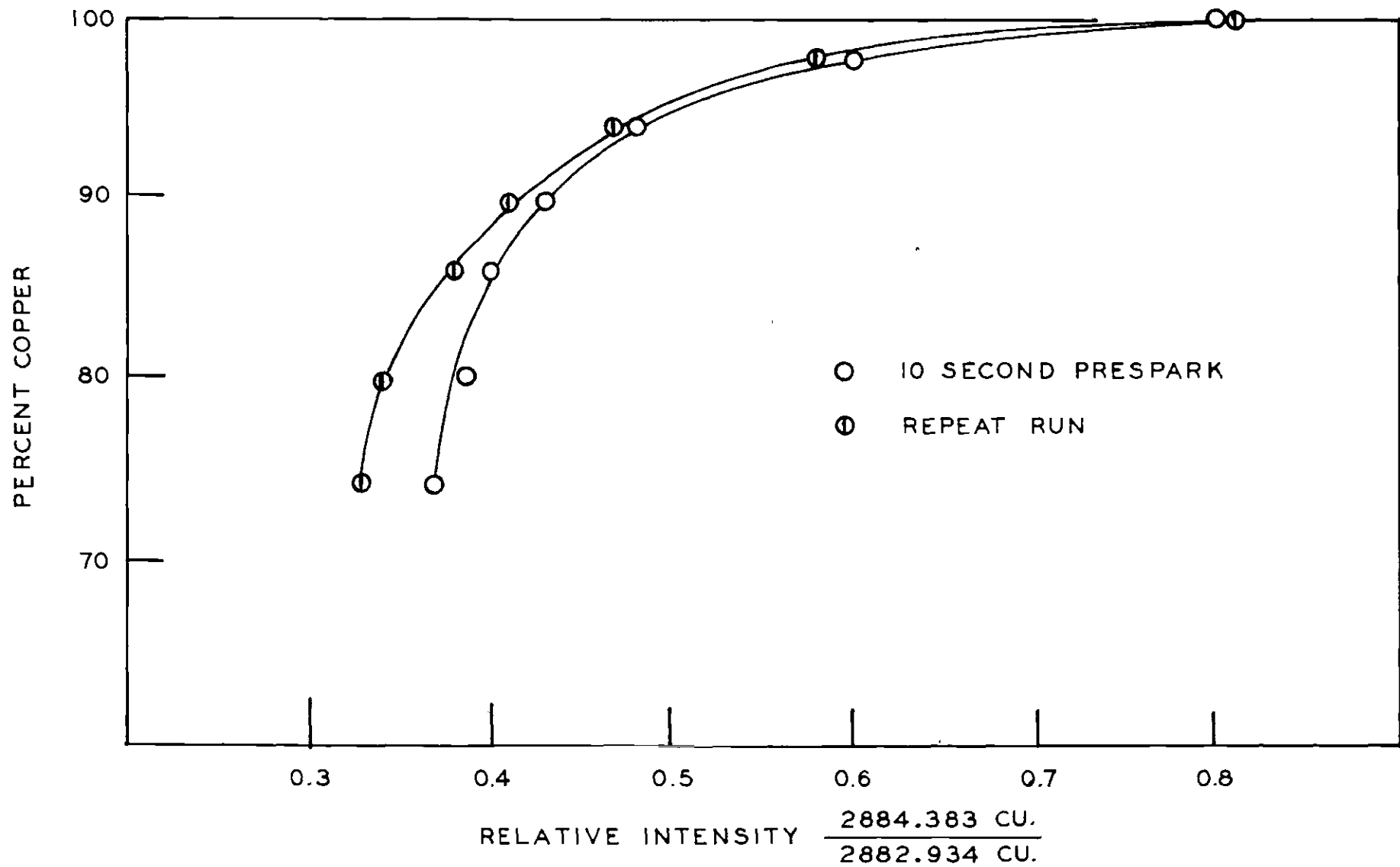


FIGURE 2—WORKING CURVE FOR ALLOY PLATES

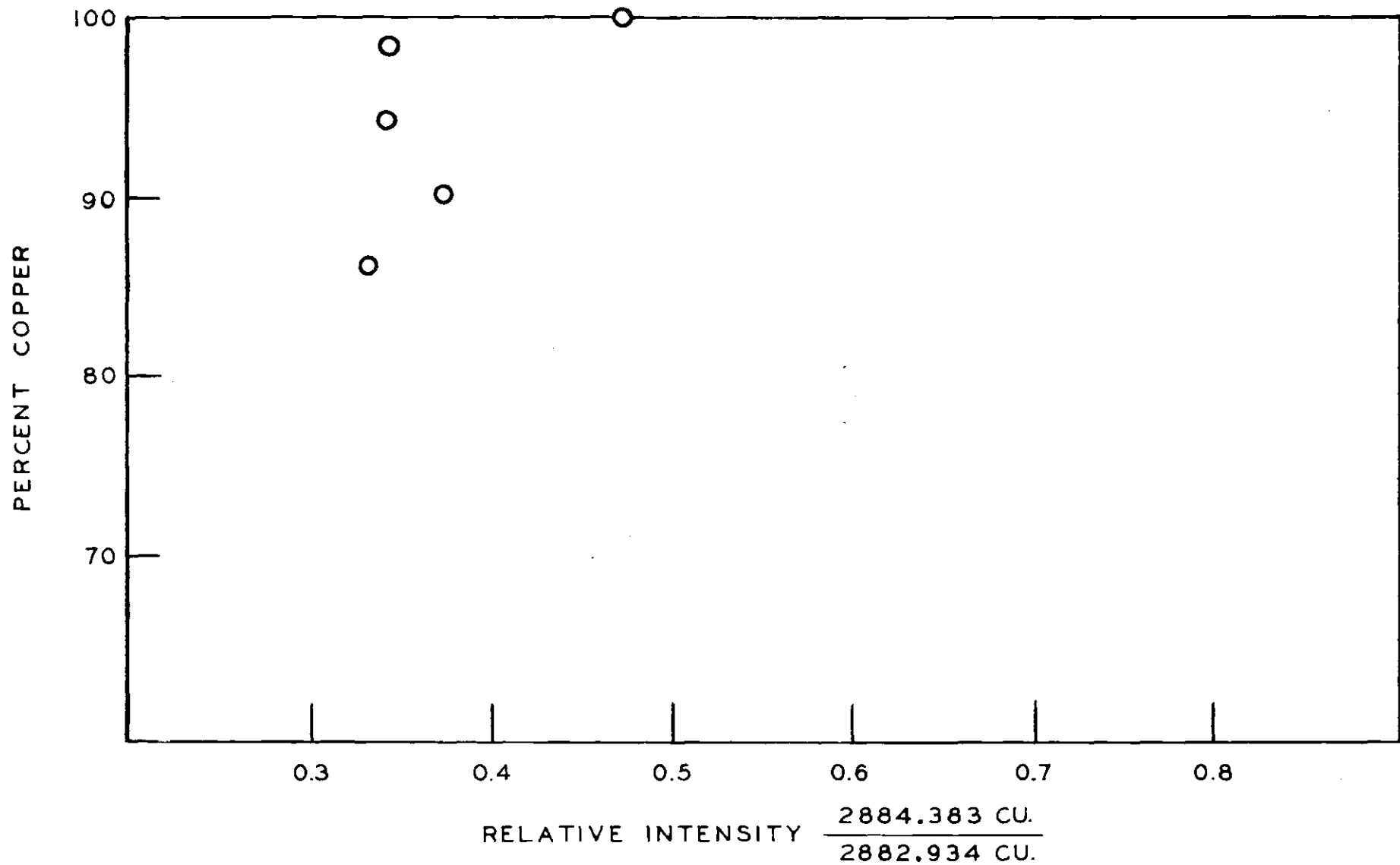


FIGURE 3 - EFFECT OF NON-UNIFORM SURFACE