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RESEARCH PROJECT NF-16, PHASE II: PRELIMINARY PROGRAM

THE INFLUENCE OF INDIVIDUAL ADDITIONS OF TIN, CADMIUM, ANTIMONY AND COPPER ON THE STRUCTURE AND PROPERTIES OF GALVANIZED COATINGS

by

J. J. SEBISTY AND R. PALMER

PHYSICAL METALLURGY DIVISION

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ABSTRACT

This report describes an investigation of the structure and properties of experimental galvanized coatings prepared by the dry galvanizing technique. Iron-saturated zinc baths were used, to which two levels, representing impurity and alloying concentrations, of tin (0.10%, 2.50%), cadmium (0.05%, 1.25%), antimony (0.01%, 0.25%) and copper (0.05%, 1.25%) were added, with and without additions of 0.15% aluminum and 0.50% lead. A constant bath temperature, a range of immersion times and two grades of steel sheet were used.

In the absence of aluminum and lead, the influence of the addition elements studied was primarily restricted to variations in coating surface appearance. These effects were apparent when the additions were present in alloying concentrations. Of importance also was the embrittling effect of the high antimony addition. High concentrations of tin and cadmium were somewhat less harmful in this respect.

With aluminum and lead present in the bath, tin and antimony at either concentration had no significant effect on coating formation or properties. This also applied to cadmium and copper at an immersion time of 0.25 min, but at 2 min, cadmium at either level partially neutralized the inhibiting effect of aluminum. This behaviour was pronounced with the high-copper addition so that coating weight, ductility and adherence were markedly affected.

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Rapport industriel confidentiel

Direction des mines
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RECHERCHE NF-16, ÉTAPE II:
PROGRAMME PROVISOIRE

INFLUENCE DES ADDITIONS SÉPARÉES D'ÉTAIN, DE CADMIUM, D'ANTIMOINE ET DE CUIVRE SUR LA STRUCTURE ET LES PROPRIÉTÉS D'ENDUITS GALVANISÉS

par

MM. J.J. Sebisty* et R. Palmer**

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RÉSUMÉ

Le présent rapport décrit une analyse de la structure et des propriétés d'enduits galvanisés expérimentalement suivant un procédé de galvanisation par voie sèche. On a utilisé des bains de zinc saturés de fer, auxquels ont été ajoutés deux niveaux, qui représentaient l'un les impuretés et l'autre les éléments d'addition: étain (0.10%, 2.50%), cadmium (0.05%, 1.25%), antimoine (0.01%, 0.25%) et cuivre (0.05%, 1.25%), avec et sans addition de 0.15% d'aluminium et de 0.50% de plomb. On a maintenu le bain à une température constante, une gamme donnée de durées d'immersion et deux catégories de tôle d'acier ont été employées.

En l'absence d'aluminium et de plomb, l'effet des éléments d'addition à l'étude s'est limité principalement aux variations dans l'apparence de la surface de l'enduit. Cet effet s'est produit lorsque les additions étaient faites aux concentrations d'alliage. Il convient aussi de mentionner l'effet de fragilité dû à l'addition d'une forte quantité d'antimoine. De fortes concentrations d'étain et de cadmium étaient un peu moins nuisibles sous ce rapport.

En présence d'aluminium et de plomb dans le bain, l'étain et l'antimoine à l'un ou l'autre niveau n'avaient pas d'effet appréciable sur la formation ni sur les propriétés de l'enduit. Il en était ainsi pour le cadmium et le cuivre, dans le cas d'une immersion d'une durée de 0.25 min., mais, après deux minutes, le cadmium à l'un ou à l'autre niveau neutralisait partiellement l'effet inhibiteur de l'aluminium. Ce comportement était bien marqué avec l'addition d'une forte quantité de cuivre, alors qu'il y avait variation marquée du poids, de la ductilité et de l'adhérence de l'enduit.

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INTRODUCTION

In February, 1957, research on the hot-dip galvanizing process was initiated at the Physical Metallurgy Division, Mines Branch, Ottawa, in co-operation with the Canadian Zinc Research and Development Committee. Various processing factors involved in galvanizing of steel sheet were selected for study, with emphasis being placed on examination of the influence of bath composition on coating structure and properties.

In Phase I of the project, which was completed in February, 1958, a statistical study was made on the effects of aluminum and lead added to iron-saturated baths. Bath temperature, immersion time and steel surface roughness were additional variables examined. Various tests, including metallographic examination and accelerated corrosion behaviour, were carried out to evaluate the properties of the experimental coatings prepared. The results of this initial investigation have been described in reports issued to date[1] [2] [3]

In the second Phase of the project the above field of study was extended to cover other elements frequently found in commercial galvanizing baths, namely, tin, cadmium, antimony and copper. As preliminary work, a limited scale investigation of the influence of individual additions of these elements on the structure and properties of laboratory-prepared coatings was begun in September, 1958. Two grades of steel sheet were galvanized in iron-saturated zinc baths, to which small (impurity) and relatively large (alloying) amounts of the above elements were added, with and without fixed additions of aluminum and lead. The coatings were evaluated by the methods used in the previous investigation[1].

This report describes the results of this preliminary study, which was intended to provide a basis for comparison in a more comprehensive program to follow which will form the main part of the project in phase II. In this, it is intended to examine the influence of combined additions of these elements on coating structure and properties.

MATERIALS

Steel

Open-hearth, low-carbon, 24 s.w.g. rimmed steel sheets, bright annealed and temper rolled to two different surface finishes, designated No. 5 and No. 3, were used. The materials were from different heats with the following quoted analyses:

	No. 5 Finish	No. 3 Finish
C %	0.07	0.04
P %	0.013	0.010
S %	0.028	0.026
Mn%	0.32	0.12
Si %	0.002	0.002

Bath Additions

The grades of material used in the experimental baths are listed below. Zinc, lead, tin and cadmium were added directly as required. In the case of iron, aluminum, antimony and copper, the additions were made as master alloys which had been shotted by casting into water.

Metal	Grade	Master Alloy
Zinc	special high grade - 99.99%	
Lead	99.99%	
Tin	99.99%	
Cadmium	99.97%	
Iron	electrolytic sheet - 99.98%	Zn - 0.3% Fe approx.
Aluminum	99.99%	Zn - 4% Al "
Antimony	99.75%	Zn - 2.5% Sb "
Copper	70-30 cartridge brass	Zn - 4% Cu "

GALVANIZING CONDITIONS

The galvanizing conditions selected are listed below. The various combinations of bath composition and immersion time, and the order in which the melts in the program were run, are shown in Table 1. For comparison purposes, two melts were included, one of which contained an iron addition only and the second contained iron, aluminum and lead.

Three 4-in. by 6-in. panels and three 3-in. by 4-in. panels of each steel finish were galvanized at each immersion time in the various baths. The 324 large panels prepared provided the main series of test specimens and the same number of small panels were used for steel weight loss determinations.

Conditions	Levels
Bath temperature	455°C (833°F)
Immersion time	0.25, 1.0, 2.0 min
Aluminum content	nil, 0.15%
Lead content	nil, 0.50%
Iron content	saturated (about 0.03% at 450°C (842°F) for pure zinc).

GALVANIZING PROCEDURE

With minor exceptions, the galvanizing procedure followed in this investigation corresponded to that used in the work in phase I. For information on the equipment used, specimen preparation, dipping procedure, bath sampling, etc., reference should be made to the report issued(1) which describes the experimental procedure in detail.

One of the changes referred to involved storage of the specimens in a dessicator in the interval between pickling and fluxing. This eliminated the superficial rusting which was previously encountered. Because of the severe staining of the coating caused by dusting ammonium chloride on the surface of the aluminum-free baths, prior to and during withdrawal of specimens, this practice was discarded.

Typical melt preparation and galvanizing logs for this series are given in Tables 2 and 3. It can be noted from the logs that single baths were used to galvanize the required specimens with the low and high additions of tin, cadmium and antimony. This could not be conveniently done in the case of copper, because of the large amount of master alloy required to raise the copper content from the low to the high level in the same bath.

Table 4 gives the chemical compositions of the baths run and shows how the compositions varied from start to end of dipping in the various tests made.

In the aluminum-free baths, the compositions were at or near the nominal levels, except for somewhat excessive tin and cadmium in tests 2 and 4, respectively.

In the aluminum-containing series no major composition changes, due to interaction of aluminum with the individual addition elements, were apparent. The aluminum behaviour was variable, and in some cases no loss during dipping was found. Where a drop did occur, this was of the order of 7% for any single series of specimens. This value represents the approximate limit of accuracy for the analytical method used, which could account for the variable behaviour noted. It should be mentioned that no extra additions of aluminum were made in the galvanizing runs in this series. No explanation can be offered for the low lead content in the case of test 16.

COATING TESTS

The tests used to evaluate the properties of the experimental coatings were similar to those set up for the previous work in phase I. These included: coating weight, iron content determination and steel weight loss (stripping tests), ductility rating (cupping test), adherence rating (simple bend and lock seam tests), alloy thickness measurement (metallography) and surface appearance ratings (spangle size and contrast, brightness, roughness). The report issued covering the previous investigation[1] describes the procedures followed in each case.

As far as possible, the tests on the large panels were carried out in triplicate. Exceptions included the ductility and surface appearance ratings in which duplicate determinations were made. Single samples only were run in the lock seam test.

Metallographic examination was made of single samples from one of each series of three large panels galvanized. The iron-zinc alloy thickness was measured at a minimum of six, randomly-chosen points on each sample. Calculation of the proportion of alloy in the coatings was based on the average of these measurements and the average coating weight as determined in the stripping tests.

The steel weight loss tests on the small 3-in. by 4-in. panels were carried out in triplicate.

Complete coating test results for only two typical series of specimens are included in this report. These are given in Table 5. In Table 6, average results for all the specimens prepared are listed. The number of determinations which were averaged are indicated in the table. It may also be noted that individual test results, which deviated considerably from expected values, were discarded. These discrepancies were related to experimental error or to inconsistencies inherent in the galvanizing behaviour of the base steel.

The data in Table 6 relating to coating weight, iron content, thickness and properties of iron-zinc alloy in the coatings are graphically presented in Figures 1 to 8. These show the relationships between the above mentioned dependent variables and the independent variables of immersion time, bath composition and steel surface finish. Graphical presentation of the results relating to ductility, adherence and surface appearance was not attempted due to the nature of most of the data, which were more amenable to descriptive treatment.

The photomicrographs in Figures 9 to 13 illustrate typical coating microstructures observed. Reference should be made to the previous report[1] for information on the polishing preparation of the samples.

For reasons which could not be established, the steel weight loss tests showed very erratic behaviour even, in some cases, between specimens which were similarly treated. Since no useful interpretation of the data was possible, all results of this test were omitted from this report.

RESULTS AND DISCUSSION

In the case of the coating tests graphically presented in Figures 1 to 8, separate sets of curves show the results obtained with each of the individual alloying elements, tin, cadmium, antimony and copper in the aluminum-free and aluminum-containing baths.

1. Aluminum-free Coatings

(a) Coating Weight

The very thick coatings produced in this series, even at the minimum immersion time of 0.25 min, are indicated by the upper sets of curves in Figures 1 and 2. At this immersion time, the spread in values with each of the alloying additions was generally related to steel surface finish with the No. 3 steel usually giving slightly thicker coatings. The presence or concentration of the individual elements within the range studied thus had little or no effect on coating weight. An exception can be noted with the 1.25% Cu addition which produced a significant reduction in coating weight with both steels at the minimum immersion time. This appeared to be a real effect unrelated to the normal inconsistencies in behaviour frequently apparent in the tests.

At the longer immersion times, the influence of steel surface finish was more pronounced and, except with the high-copper bath, consistently heavier coatings formed on the No. 3 steel. The presence or concentration of tin and antimony showed variable but minor effects on coating weight at these immersion times and the addition of 0.05% Cu was also without effect. The reduction in coating weight provided by 1.25% Cu at dipping times of 0.25 and 1 min disappeared at 2 min.

The behaviour of cadmium was more consistent and, as defined in Figure 1 (b), coating weight was increased sharply and at a more rapid rate with immersion time in the presence of 1.25% as opposed to nil or 0.05% Cd.

(b) Iron Content

The characteristic and marked increase in iron content of the non-aluminum coatings with increasing immersion time is clearly shown in the upper sets of curves in Figures 3 and 4. This trend was the most prominent feature of the tests and the influence of bath composition and steel surface finish was much less consistently defined. Iron content as a function of steel surface finish varied widely in some baths and reversals in behaviour of the two steels was also apparent. However, in view of the very high level of iron present in these coatings, the inconsistencies noted are not unusual.

The variable behaviour of the two steels also masked the influence of bath composition changes but with the alloyed baths some definite trends can be noted. At each of the immersion times used, tin, and to a lesser extent antimony, provided consistent increases in iron content with increase in concentration of these additions. This behaviour can also be noted with cadmium and copper but only at immersion times approaching 2 min. The reduction in iron content associated with 1.25% Cu at the shorter immersion times represents similar behaviour to that found in the coating weight tests.

(c) Alloy Thickness

The behaviour of the individual alloying elements with respect to growth of the iron-zinc alloy layer generally conformed to that noted in the iron determination tests. At the low-concentration levels there was a tendency for minor increases in alloy thickness with each of the additions made as shown in Figures 5 and 6, but this was restricted to the longer immersion times.

Increase in concentration of antimony from 0.01% to 0.25% had little or no effect, but increase in cadmium content to 1.25% promoted increased steel attack and iron-zinc alloy growth as shown in Figure 5(b). High copper also produced heavier alloy, but only at the maximum immersion time of 2 min. Raising the tin level from 0.10% to 2.50%, on the other hand resulted in a reduction in alloy thickness, notably at the shorter immersion times, as illustrated in Figure 5 (a). However, the effective reduction was relatively minor.

With respect to steel surface finish, the relative behaviour of the two steels was again variable. In general, slightly thicker alloy growth was associated with the No. 3 finish sheet, but, as can be seen from the graphs in Figures 5 and 6, this was not consistent.

(d) Proportion of Alloy

The relevant graphs derived for the aluminum-free coatings are shown in Figures 7 and 8. The proportion of alloy in these coatings was in the range of 50-65%, with the higher values being associated with the maximum immersion time of 2 min. In common with the test measurements described above, which were used in calculating the proportion of alloy present, the results showed variable scatter and the influence of bath composition and steel surface-finish factors was ill defined. However, for all practical purposes this is not of great importance in view of the abundance of alloy formed in these coatings.

(e) Ductility and Adherence

As emphasized in the previous report[1], the tests used to establish the relative ductility and adherence of the experimental coatings were essentially rough sorting tests only. It was confirmed that the tests were not sufficiently sensitive to define other than major changes in these coating properties.

According to the ductility and adherence ratings defined in Table 5 (a) the performance of the aluminum-free coatings must be considered inferior. In the cupping test, moderate to severe cracking of even the thinnest coatings was evident. None of the alloying elements at low concentration had any effect, but at high concentrations, ductility was reduced at the longer immersion times. Tin and copper appeared to be more harmful than cadmium, but the evidence supporting this was not as well defined as suggested by Thorley's report [4] on the work of other investigators. The well known embrittling effect of high antimony (0.25%) was confirmed since pronounced cracking was apparent even with the thinnest coatings in this group of specimens.

In the bend tests the only trend observed was a generally consistent reduction in coating adherence with increasing immersion time. This occurred with or without the addition elements and regardless of the concentration level. The single samples subjected to the lock-seam test all cracked and peeled severely without exception. In view of this, the adherence ratings established in the bend test must be classified as being indicative of poor adhesion properties for the non-aluminum coatings.

(f) Metallographic Structure

The various alloying additions at low concentration had no effect on the microstructure of these coatings. Typical microstructures for 0.10% Sn coatings on the No. 5 finish steel shown in Figure 9, were representative of the entire series. The more rapid rate of growth of the zeta and delta iron-zinc phases at short and long immersion times, respectively, as found by Rowland[5], is illustrated in the photomicrographs.

The alloying additions at high concentration also had little or no effect at an immersion time of 0.25 min. In the case of tin and antimony the presence of beads of eutectic inclusions embedded in the zeta phase was a distinguishing feature as shown in Figure 10 (a). At the much longer immersion time of 2 min., tin, antimony and cadmium produced marked columnar growth of the zeta phase as illustrated in Figure 10 (b). This photomicrograph also shows that tin appeared to retard the growth of the delta phase in contrast to the more normal rate of growth with cadmium and antimony.

The combination of high copper and a long immersion time modified the coating structure somewhat. The zeta phase showed more dense packing and columnar growth was not as well defined even after prolonged etching. This is indicative of rapid zinc attack and suggests that copper does increase the iron-zinc reaction rate as claimed by Bablik[6]. However, the discontinuous crystal formations and characteristic hexagonal crystallites, found by Bablik at immersion times of 3 min and longer, were not observed.

(g) Surface Appearance

With the low concentrations of tin, antimony and copper, semi-bright coatings with a spangle-free metallic appearance were obtained. These were similar to the coatings produced in the basic bath containing iron only. The low-cadmium addition on the other hand resulted in considerably brighter coatings with high reflectivity. With respect to steel surface finish, a generally rougher, sand-paper-like texture was apparent with the No. 3 steel.

With the alloying addition of 2.50% Sn, the coatings assumed a characteristic frosty appearance with low reflectivity. The spangles were well defined but small in size. For the range of experimental conditions used, this confirms the observations of Phillips[7], that tin without lead will not produce larger crystals or spangles of zinc. The tarnishing effect of tin in excess of 1%, reported by Hall[8], was not observed even with the much higher tin level used.

Spangles of variable size were formed with 0.25% Sb. With a 2 min dip these were of gross size and showed pronounced directional growth. Again, staining effects usually associated with this level of antimony were not observed.

With 1.25% Cd and 1.25% Cu the metallic spangle-free appearance described above was retained. In the former case the coatings had a matte sheen with poor reflectivity. This behaviour contrasted sharply with the brightening effect of the low cadmium addition mentioned above. The only effect of copper was a light brownish staining of the surface.

A distinctly rougher texture was again associated with the No. 3 finish steel with all of the coatings just described.

2. Aluminum-containing Coatings

(a) Coating Weight

The generally marked reduction in coating weight achieved by the addition of 0.15% Al to the bath is indicated by the lower sets of graphs in Figures 1 and 2.

With tin and antimony, at both low- and high-concentration levels, the inhibiting effect of aluminum was essentially unchanged for the range of immersion times used. With these additions, steel surface finish exhibited a moderate effect and the No. 3 steel generally yielded slightly thicker coatings.

The effect of 0.05% and 1.25% Cd was also minor at the minimum immersion time of 0.25 min, but at longer immersion times, coating weight showed a marked increase well above that obtained with no cadmium present. With the No. 5 finish steel increase in cadmium content from 0.05% to 1.25% produced a proportionate increase in coating weight. With the other steel variation in the cadmium level was much less effective.

As with tin and antimony the influence of 0.05% Cu was negligible for the range of immersion times used, and the minor changes in coating weight apparent were related to steel surface finish. However, with 1.25% Cu, gross increases in coating weight resulted at the extended dipping times. In the case of the No. 5 finish steel, values approaching the thickness of the aluminum-free coatings were obtained at 2 min immersion as shown in Figure 2 (b).

(b) Iron Content

From Figures 3 (a), 4 (a) and 4 (b) it can be seen that the iron content of the coatings was not affected by the impurity additions of 0.1% Sn, 0.01% Sb and 0.05% Cu for immersion times between 0.25 and 2 min.

With 1.25% Sn there was a tendency for a minor increase in the iron level which became more pronounced with increasing immersion time. A similar but less clearly-defined trend was indicated for 0.25% Sb. Of much greater significance was the gross increase in iron content of the coating due to the presence of 1.25% Cu in the bath. This was pronounced with the No. 5 finish steel as shown in Figure 4 (b).

Cadmium additions also exhibited significant effects. In this case, however, the harmful effect of 0.05% and 1.25% Cd was similar for immersion times up to 1 min with both steels, and for up to 2 min with the No. 3 steel. At this longer dipping time the behaviour of the No. 5 steel was less consistent as indicated by the significant difference in iron content with increase in cadmium content of the bath (Figure 3 (b).

(c) Alloy Thickness

Tin and antimony at both low and high concentrations provided small increases in alloy thickness, but only at the longer immersion times as shown in Figures 5 (a) and 6 (a).

The behaviour of cadmium showed the same trend but to a more pronounced degree, indicating that the inhibiting effect of aluminum was partially neutralized at the longer immersion times. This can be seen in Figure 5 (b). To what extent this behaviour was influenced by the lower-than-nominal aluminum content of 0.12 - 0.13% in this bath (Table 4) is not known.

The addition of 0.05% Cu produced somewhat heavier alloy growth at all the immersion times used and in the case of 1.25% Cu, the beneficial effect of aluminum was completely overcome (Figure 6 (b)).

Steel surface finish appeared to be a more or less negligible factor with respect to alloy formation in the aluminum-containing coatings.

By way of explanation it should be noted that the alloy thickness measurements on the aluminum-containing coatings are estimates only. At a short immersion time the alloy layer was usually of superficial thickness which was difficult to measure accurately. Increase in immersion time resulted in numerous local iron-zinc alloy growths at sites of heavy steel attack. The frequency and size of these growths in some cases was such that the results had to be weighted in order to take these formations into account. The approximate nature of these measurements must therefore be appreciated.

(d) Proportion of Alloy

According to the relevant graphs in Figures 7 and 8 all of the alloying additions appeared to be effective, to varying degrees, in increasing the proportion of alloy in the coatings. The relative effects appear to be large, but for the reasons mentioned above, and from metallographic examination, it is considered that the results are more nearly representative only in the case of the high-cadmium and high-copper coatings.

(e) Ductility and Adherence

The ductility of these coatings was generally excellent except for the panels dipped at the longer immersion times in the high-cadmium and high-copper baths. The poorer performance in these cases was clearly related to the increased steel attack and iron-zinc alloy formations referred to above. With respect to steel surface finish, the coatings on the No. 3 steel showed somewhat lower ductility, but this was only apparent for the longer dipping times.

The adherence of these coatings was also excellent except for the high-cadmium and high-copper panels dipped for the longer immersion times. These showed a significant reduction in adhesion, particularly with the copper-containing series. The bend test behaviour was confirmed in the lock-seam tests since peeling and flaking was confined to the specimens referred to above.

(f) Metallographic Structure

The pronounced reduction in iron-zinc alloy growth and coating thickness affected by the high aluminum content of 0.15%, were distinguishing features of most of the coatings in this series.

The coating microstructure illustrated in Figure 11 (a) was more or less representative of all the specimens dipped for 0.25 min and the presence or concentration of each of the alloying additions, within the ranges studied, resulted in no significant alteration in the coating structure. Thus, under these conditions the aluminum addition remained as the principal factor controlling the iron-zinc reaction.

Increase in immersion time to 2 min in the low-tin and low-antimony baths produced similar structures to that obtained when these elements were absent. A typical photomicrograph is illustrated in Figure 11(b). The corresponding cadmium-containing samples exhibited somewhat more prominent and irregular iron-zinc alloy growth as shown in Figure 12(a). With 0.05% Cu also, the frequency of local steel attack was noticeably increased. Thus, while both tin and antimony were found to have no influence on coating microstructure, the low or impurity concentrations of both cadmium and copper were harmful to some extent, and reduced the inhibiting effect of 0.15% aluminum with an immersion time of 2 min.

At high concentrations of the addition elements, changes observed in the metallographic structure of the aluminum-containing coatings were also confined to longer immersion times.

As can be seen by comparison of Figures 11(b) and 11(c), the effect of 2.50% Sn was relatively minor even with a dipping time of 2 min. Similar behaviour was apparent with 0.25% Sb.

The presence of 1.25% Cd on the other hand, resulted in marked growth of the angular iron-zinc alloy crystals as shown in Figure 12 (b). Prominent local alloy growths of the type illustrated were evident and these were clearly instrumental in promoting heavy zinc drag-out and a thicker coating. The behaviour noted thus, in part, confirms Hall's observation[8] that 0.5% Cd neutralizes an addition of 0.05% Al.

With 1.25% Cu and 2 min immersion, the normal influence of aluminum was more or less completely overcome and rapid steel attack occurred, as indicated by the thick granular-type of alloy structure in Figure 13 (a). This contradicts Bablik's claim[6] that the inhibiting effect of 0.2% Al is not affected by 1% Cu, even with an immersion time of 2 min. However, in Bablik's work a pure zinc bath, presumably free of iron and lead, and a bath temperature of 440°C (825°F) were used. The variation in galvanizing conditions could thus account for the different behaviour noted.

Of interest in the high-copper series was the characteristic hexagonal shape of the crystals initially formed at the steel surface. These showed rapid growth with increasing immersion time until they were eventually undermined by heavy alloy growth consisting of all the normal iron-zinc phases. This can be seen in Figure 13 (b) which represents a second area on the sample used for Figure 13 (a).

(g) Surface Appearance

The aluminum-containing coatings were characterized by uniformly high reflectivity. The alloying additions at low levels had no observable effect on surface appearance, and with or without the individual additions, spangle formation was lacking or vaguely defined. With the No. 5 finish steel, the coatings prepared at the long immersion time were marred by the presence of pimple defects protruding through the coatings at the sites of the local alloy growths described above. These were less evident with the No. 3 steel but the surface texture of the coatings on this material was distinctly rougher.

Increase in concentration of each of the alloying additions produced distinct surface effects. Large flowery spangles with irregular edges were obtained with tin, whereas with antimony and copper the spangles were medium to small in size, respectively. Cadmium gave small spangles and the coatings had the characteristic frosty appearance similar to the high-tin, aluminum-free coatings previously described.

The No. 5 finish steel panels dipped for 2 min in the high-copper bath had a very pleasing appearance due to a combination of good spangle formation and a smooth surface free of defects. The surface of the corresponding specimens for the other additions was generally marred by pimple defects on the No. 5 finish steel, and by the rougher texture on the No. 3 steel. The pimple defects caused by the protruding local alloy growths were most prominent with the tin- and antimony-containing coatings.

SUMMARY

In this investigation, the metallographic structure and properties of laboratory-prepared sheet galvanized coatings, as influenced by impurity and alloying concentrations of tin, antimony and copper in iron-saturated baths, with and without fixed additions of aluminum and lead have been studied. For the experimental conditions used, the results obtained may be summarized as follows:

1. Aluminum-free Coatings

(a) For the coatings prepared in iron-saturated baths with no aluminum or lead present, it was found that the low or impurity concentrations of the addition elements were without effect on coating weight, iron content of the coating, and iron-zinc alloy formation. Coating ductility and adherence were also unaffected. This applied to surface appearance as well except in the case of 0.05% Cd, which appeared to improve the reflectivity of the spangle-free coatings.

(b) At the high or alloying concentrations of each addition, various effects were observed.

Tin tended to reduce iron-zinc alloy formation, but although this was in some cases reflected in reduced coating weight, ductility was adversely affected to some extent. By itself, i.e., with no lead present, tin failed to promote growth of large spangles.

Antimony produced gross spangles and was particularly detrimental to coating ductility and adherence. No other effects were noted.

Cadmium promoted heavier alloy growth with a corresponding increase in iron content and coating weight. Some loss in ductility was apparent and it was also harmful with respect to surface appearance, as indicated by the matte sheen of the spangle-free coatings. This behaviour was in marked contrast to the brightening effect found with the low cadmium addition.

Copper was apparently effective in reducing coating weight and iron content at short immersion times, but the coatings failed to exhibit any improvement in ductility and adherence. Staining of the coatings showed copper to be detrimental to surface appearance.

(c) Although the behaviour of the two steels was not consistent, it was generally found that thicker coatings, with heavier iron-zinc alloy growth and higher iron content, were associated with the No. 3 finish steel. Within the limits of sensitivity of the relevant tests, coating ductility and adherence appeared to be correspondingly reduced. With respect to surface appearance properties, the behaviour of the two steels was essentially similar.

(d) According to the performance standards used in this investigation, all of the coatings prepared without aluminum and lead in the bath were classified as having poor ductility and adherence properties.

2. Aluminum-containing Coatings

(a) For the coatings galvanized in iron-saturated baths containing 0.15% Al and 0.5% Pb, the presence of aluminum was, in most cases, the principal factor controlling the thickness and other properties of the coating. Apart from the exceptions noted below, the galvanizing variables investigated were of secondary importance and the coatings were characteristically very thin with excellent ductility and adherence properties.

(b) The low or impurity concentrations of tin and antimony in these baths had little or no effect on the various coating properties examined.

This also applied to the low-cadmium and low-copper addition at an immersion time of 0.25 min, but at 2 min, alloy formation and coating weight were increased somewhat. This suggests that in these cases, the inhibiting effect of 0.15% Al in the bath was partially neutralized.

The characteristic surface defects normally obtained with a high-aluminum concentration in the bath were not altered by the various additions nor was there any improvement in spangling behaviour.

Variation in steel surface roughness generally had no significant effect on coating properties with these baths.

(c) At the high or alloying concentrations of the addition elements some distinct effects were again observed.

The influence of tin or antimony on coating formation and properties was negligible except with respect to surface appearance. In both cases, spangles of medium to large size were formed.

The same behaviour was observed with cadmium and copper at an immersion time of 0.25 min. Under these conditions, spangles varied from very small to medium in size, respectively. As the dipping time was increased to 2 min, cadmium, and more especially copper, were shown to be increasingly detrimental. General thickening of the alloy layer and increasingly severe local steel attack combined to promote much heavier coatings which had poor ductility and adherence. Iron content of the coating was increased proportionately. Thus, at long immersion times, cadmium and to a greater degree, copper, neutralized the inhibition of steel attack normally obtained with 0.15% Al.

(d) As indicated above, steel surface finish was in most cases a factor of negligible importance, and the only difference in behaviour noted was the generally rougher texture of the coatings on the No. 3 steel. This tended to mask pimple defects occurring at the sites of local steel attack, so that these were much more prominent with the thinner coatings on the smoother No. 5 finish steel.

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(Tables, graphs and figures)
(follow, on pages 31-48.)

JJS:vb

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TABLE 1

Experimental Galvanizing Conditions

Bath Number	Test Number	Bath Temperature °C	°F	Immersion Time min	Fe %	Al %	Pb %	Sn %	Cd %	Sb %	Cu %
1	1	455	851	0.25	0.03			0.10			
		"	"	1.0	"			"			
		"	"	2.0	"			"			
	2	"	"	0.25	0.03			2.50			
		"	"	1.0	"			"			
		"	"	2.0	"			"			
2	3	"	"	0.25	0.03				0.05		
		"	"	1.0	"				"		
		"	"	2.0	"				"		
	4	"	"	0.25	0.03				1.25		
		"	"	1.0	"				"		
		"	"	2.0	"				"		
3	5	"	"	0.25	0.03					0.01	
		"	"	1.0	"					"	
		"	"	2.0	"					"	
	6	"	"	0.25	0.03					0.25	
		"	"	1.0	"					"	
		"	"	2.0	"					"	
4	7	"	"	0.25	0.03						0.05
		"	"	1.0	"						"
		"	"	2.0	"						"
5	8	"	"	0.25	0.03						1.25
		"	"	1.0	"						"
		"	"	2.0	"						"
6	9	"	"	0.25	0.03	0.15	0.50	0.10			
		"	"	1.0	"	"	"	"			
		"	"	2.0	"	"	"	"			
	10	"	"	0.25	0.03	0.15	0.50	2.50			
		"	"	1.0	"	"	"	"			
		"	"	2.0	"	"	"	"			
7	11	"	"	0.25	0.03	0.15	0.50		0.05		
		"	"	1.0	"	"	"		"		
		"	"	2.0	"	"	"		"		
	12	"	"	0.25	0.03	0.15	0.50		1.25		
		"	"	1.0	"	"	"		"		
		"	"	2.0	"	"	"		"		
8	13	"	"	0.25	0.03	0.15	0.50			0.01	
		"	"	1.0	"	"	"			"	
		"	"	2.0	"	"	"			"	
	14	"	"	0.25	0.03	0.15	0.50			0.25	
		"	"	1.0	"	"	"			"	
		"	"	2.0	"	"	"			"	
9	15	"	"	0.25	0.03	0.15	0.50				0.05
		"	"	1.0	"	"	"				"
		"	"	2.0	"	"	"				"
10	16	"	"	0.25	0.03	0.15	0.50				1.25
		"	"	1.0	"	"	"				"
		"	"	2.0	"	"	"				"
11	17	"	"	0.25	0.03						
		"	"	1.0	"						
		"	"	2.0	"						
12	18	"	"	0.25	0.03	0.15	0.50				
		"	"	1.0	"	"	"				
		"	"	2.0	"	"	"				

TABLE 2

Typical Galvanizing Melt Log

MINES BRANCH PHYSICAL METALLURGY DIVISION	NON-FERROUS METALS SECTION GALVANIZING MELT LOG	Project NF-16 Date: Sept. 19/58

Melt No. DU (Bath No. 1)

Charge 37 lb

Metal	Composition	Form	Amount
Zn	99.99%	ingot	31.93 lb
First Sn addition	99.99%	bar	16.8 g
Second Sn addition	99.99%	bar	430 g
Zn-Fe master	0.22% Fe	shot	2290 g

Procedure	Time	Temp	Remarks
Furnace on	8.35 a.m.	-	
Zinc charged	8.40 "	-	
	10.18 "	500°C	
Alloying			
First Sn addition	10.30 "	500°C	
Zn-Fe	10.40 "	480°C	
Second Sn addition	2.45 p.m.	455°C	
Poured to ingot after galvanizing run	4.30 p.m.	460°C	

Bath Composition	Fe %	Sn %	
Test No. 1			
Nominal	0.030	0.10	
Actual			
Start (1.35 p.m.)	0.032	0.11	
End (2.40 p.m.)	0.032	0.11	
Test No. 2			
Nominal	0.30	2.50	
Actual			
Start (2.50 p.m.)	0.31	2.78	
End (4.20 p.m.)	0.31	2.78	

TABLE 3

Typical Galvanizing Log

MINES BRANCH PHYSICAL METALLURGY DIVISION	NON-FERROUS METALS SECTION GALVANIZING LOG SHEET	Project NF-16 Date: Sept. 19/58

Melt No. DU (Bath No. 1)

Test No. 1

Material Treated
36 specimens, 4-in. x 6-in.
(18 each of No. 5 & No. 3 steel)
36 specimens, 3-in. x 4-in.
(18 each of No. 5 & No. 3 steel)

Pickling

Sample No.	Acid Conc	Inhibitor	Time & Temp	Rinse
All	5% H_2SO_4 sol'n.	1/2% by volume of acid (Rodine 92)	5 min at 71°C	Scrubbed and then rinsed for 1 min in cold running water. Dried in acetone.

Fluxing

Sample No.	Flux	Density	Time & Temp	Drying Time & Temp
All	Zinc chloride - Ammonium chloride (1.27:1.35 ratio flux)	10.4° Baumé	1 min at 82°C	1.5 to 2 min at 160 to 170°C

Galvanizing

Sample No.*	Bath Temp °C	Immersion Speed	Immersion Time	Withdrawal Speed	Remarks
Large Specimens					
1-1 to 1-3	457, 457, 457	6 fpm	30 sec	3 fpm	No. 5 finish
1-4 to 1-6	457, 456, 457	"	1 min	"	"
1-7 to 1-9	457, 457, 457	"	2 min	"	"
1-10 to 1-12	457, 457, 457	"	30 sec	"	No. 3 finish
1-13 to 1-15	455, 454, 454	"	1 min	"	"
1-16 to 1-18	454, 455, 455	"	2 min	"	"
Small Specimens (for steel weight loss measurements) (each group of three dipped prior to each series of three large specimens)					
1-19 to 1-21	454	Manual-approx. 8 fpm	30 sec	Manual approx. 8 fpm	No. 5 finish
1-22 to 1-24	455	"	1 min	"	"
1-25 to 1-27	457	"	2 min	"	"
1-28 to 1-30	457	"	30 sec	"	No. 3 finish
1-31 to 1-33	457	"	1 min	"	"
1-34 to 1-36	457	"	2 min	"	"

* Samples shown are 0.1% Sn series. Similar sequence repeated at 2.50% Sn in same bath.

TABLE 4

Galvanizing Bath Analyses

Bath Number	Test Number	Sample Number *	Fe %	Al %	Pb %	Sn %	Cd %	Sb %	Cu %
1	1	N	0.030			0.10			
		1	0.032			0.11			
		2	0.032			0.11			
	2	N	0.030			2.50			
		1	0.031			2.78			
		2	0.031			2.78			
2	3	N	0.030				0.050		
		1	0.030				0.049		
		2	0.031				0.050		
	4	N	0.030				1.25		
		1	0.032				1.37		
		2	0.032				1.40		
3	5	N	0.030					0.010	
		1	0.032					0.011	
		2	0.032					0.010	
	6	N	0.030					0.25	
		1	0.030					0.24	
		2	0.031					0.21	
4	7	N	0.030						0.05
		1	0.037						0.05
		2	0.036						0.05
5	8	N	0.030						1.25
		1	0.032						1.26
		2	0.030						1.26
6	9	N	0.030	0.15	0.50	0.10			
		1	0.030	0.15	0.47	0.11			
		2	0.031	0.14	0.47	0.11			
	10	N	0.030	0.15	0.50	2.50			
		1	0.031	0.14	0.48	2.57			
		2	0.032	0.13	0.49	2.56			
7	11	N	0.030	0.15	0.50		0.050		
		1	0.032	0.13	0.48		0.051		
		2	0.032	0.13	0.48		0.051		
	12	N	0.030	0.15	0.50		1.25		
		1	0.032	0.13	0.47		1.26		
		2	0.034	0.12	0.48		1.27		
8	13	N	0.030	0.15	0.50			0.01	
		1	0.031	0.15	0.49			0.01	
		2	0.032	0.15	0.49			0.01	
	14	N	0.030	0.15	0.50			0.25	
		1	0.030	0.14	0.52			0.21	
		2	0.031	0.13	0.52			0.21	
9	15	N	0.030	0.15	0.50				0.05
		1	0.030	0.14	0.47				0.05
		2	0.029	0.14	0.49				0.05
10	16	N	0.030	0.15	0.50				1.25
		1	0.030	0.15	0.11				1.23
		2	0.027	0.15	0.30				1.22
11	17	N	0.030						
		1	0.029						
		2	0.031						
12	18	N	0.030	0.15	0.50				
		1	0.028	0.14	0.45				
		2	0.027	0.14	0.46				

* N - nominal composition
1 - sample at start of run
2 - sample at end of run

TABLE 5

Coating Test Results For Typical Series of Specimens

Test Number	Immersion Time min	Steel Finish	Coating Wt, oz/sq ft-sheet	Iron Content mg/sq ft	Iron Content g/m^2	Alloy Thickness mm x 10^{-3}	Proportion of Alloy %	Ductility	Adherence	Spangle Size	Spangle Contrast	Brightness	Roughness
1	0.25	5	1.37	1942	20.9	-	-	-	-	-	-	-	-
	0.25	5	1.37	1814	19.5	-	-	3	4	4	4	2	2
	0.25	5	1.36	1797	19.3	17.3	59.6	3	4	4	4	2	2
	0.25	3	1.45	1720	18.5	-	-	-	-	-	-	-	-
	0.25	3	1.39	1785	19.2	-	-	3	4.5	4	4	2	3
	0.25	3	1.41	1795	19.3	15.7	52.6	3	4.5	4	4	2	3
9	0.25	5	0.60	382	4.1	1.9	15.7	-	-	-	-	-	-
	0.25	5	0.58	233	2.5	-	-	1	1	4	4	2	2
	0.25	5	0.53	288	3.1	-	-	1	1	4	4	2	2
	0.25	3	0.54	214	2.3	1.9	18.1	-	-	-	-	-	-
	0.25	3	0.54	214	2.3	-	-	2	1	3	3	2	3
	0.25	3	0.53	196	2.1	-	-	2	1	3	3	2	3

Note: Alloy thickness values are averages of at least six measurements on single samples.

For ductility, adherence and surface appearance rating codes see Table 5 (a).

TABLE 5 (a)

Surface Appearance Rating Codes

Ductility

Rating:
- 1 - Excellent, no cracking
- 2 - Good, network of fine cracks
- 3 - Fair, general cracking, with coating broken into small blocks
- 4 - Poor, wide separation of medium size blocks
- 5 - Very poor, general peeling of coating into large blocks

Spangle Size

Rating:
- 1 - Large
- 2 - Medium
- 3 - Small
- 4 - No spangle

Brightness (Photometer readings)

Rating:
- 1 - 0 to 1.25
- 2 - 1.5 to 2.75
- 3 - 3.00 to 4.25
- 4 - 4.5 +

Adherence

Minimum bend radius causing flaking (90° bend plus 180° reverse bend)

Rating:
- 1 - 0.050 in.
- 2 - 0.070 "
- 3 - 0.100 "
- 4 - 0.144 "
- 5 - 0.192 in.
- 6 - 0.252 "
- 7 - 0.320 "
- 8 - 0.400 "

Spangle Contrast

Rating:
- 1 - Good, spangles well defined
- 2 - Moderate, spangles well defined
- 3 - Low or no contrast. Spangles outlined only.
- 4 - No contrast (no spangles)

Roughness

Rating:
- 1 - Very smooth
- 2 - Moderately smooth
- 3 - Fine to moderately rough sandpaper texture
- 4 - Rough texture or uneven surface caused by various defects (ridges, dewetting, black spots, pimples)

TABLE 6

Average Coating Test Results

Test Number	Immersion Time min	Steel Finish	Coating Wt, oz/sq ft-sheet	Iron Content mg/sq ft	Iron Content g/m²	Alloy Thickness mm x 10-3	Proportion of Alloy %	Ductility	Adherence	Spangle Size	Spangle Contrast	Brightness	Roughness
1	0.25	5	1.37	1850	19.9	17.3	59.6	3	4	4	4	2	2
	1.0	5	1.95	2766	29.8	25.2	61.0	2	5.5	4	4	3	2
	2.0	5	2.41	3650**	39.3**	34.2	67.2	3	6	4	4	3	2
	0.25	3	1.42	1765	19.0	15.7	52.6	3	4.5	4	4	2	3
	1.0	3	2.26	2647	28.5	26.0	54.0	3	5	4	4	3	3
	2.0	3	2.68	3382	36.4	33.7	59.6	4	7	4	4	3	3
2	0.25	5	1.36	1943	20.9	13.8	47.8	3	3	3	1	4	2
	1.0	5	1.83	2831	30.4	21.8	56.3	4	5.5	3	1	4	2
	2.0	5	2.34	3850	41.4	32.2	65.0	4	6	3	1	4	2
	0.25	3	1.42	2025	21.8	14.6	48.6	3	3	3	1	4	3
	1.0	3	2.15	3140	33.8	25.2	55.4	4	6	3	1	4	3
	2.0	3	2.70	4175	44.9	33.7	59.2	4	7	3	1	4	3
3	0.25	5	1.45	1980*	21.3*	16.9	55.0	3	3	4	4	1	1
	1.0	5	2.00	2920	31.4	25.6	60.5	3	4.5	4	4	1	1
	2.0	5	2.28	3738*	40.2*	33.1	68.5	3	5.5	4	4	1	1
	0.25	3	1.53	1953	21.0	17.7	54.6	3	4	4	4	2	3
	1.0	3	2.19	2760	29.7	25.2	56.0	3	6	4	4	2	3
	2.0	3	2.62	3678	39.6	34.5	62.5	4	7	4	4	2	3
4	0.25	5	1.53	1897	20.4	17.6	55.4	3	3.5	4	4	2	2
	1.0	5	2.07	2839	30.5	25.5	58.2	3	5	4	4	2	2
	2.0	5	2.51	4100	44.1	35.9	67.8	4	6	4	4	3	2
	0.25	3	1.54	2082	22.4	18.7	57.4	3	5	4	4	3	2
	1.0	3	2.41	2940	31.6	28.8	57.8	4	7	4	4	3	3
	2.0	3	2.99	3863	41.6	42.1	65.8	4	8	4	4	3	3
5	0.25	5	1.45	1775	19.1	17.6	57.3	3	4	3	3	2	1
	1.0	5	2.01	2560	27.5	26.2	61.7	3	5	3	3	2	1
	2.0	5	2.33	3223	34.7	32.6	66.2	3	5.5	3	3	3	1
	0.25	3	1.53	1820	19.6	17.4	53.7	4	5.5	4	4	3	3
	1.0	3	2.25	2640	28.4	24.9	52.3	4	6	4	4	3	3
	2.0	3	2.63	3380	36.4	31.4	56.6	5	7	4	4	3	3
6	0.25	5	1.42	2055	22.1	17.0	58.8	4	4	1	1	2	2
	1.0	5	2.10	2920	31.4	26.8	60.2	4	5.5	1	1	2	2
	2.0	5	2.35	3550	38.2	32.5	65.4	4	6	1	1	3	2
	0.25	3	1.48	1905	20.5	17.3	54.1	4	5.5	1	2	3	3
	1.0	3	2.24	2790	30.0	26.0	55.6	4	6	1	2	3	3
	2.0	3	2.63	3480	37.4	32.8	59.2	4	6.5	1	2	3	3
7	0.25	5	1.40*	1760	18.9	16.4	55.4	3	3	4	4	3	2
	1.0	5	2.11	2612	28.1	25.2	56.3	3	5	4	4	3	2
	2.0	5	2.50	3448	37.1	33.0	62.6	3	5.5	4	4	3	2
	0.25	3	1.50	1895	20.4	16.7	52.7	3	3.5	4	4	3	3
	1.0	3	2.25	2715*	29.2*	25.3	53.2	4	5	4	4	3	3
	2.0	3	2.75	3528	38.0	34.6	59.7	4	6	4	4	3	3
8	0.25	5	1.25	1489	16.0	15.6	59.0	3	4	3	3	3	2
	1.0	5	1.89	2438	26.2	24.6	61.5	3	5	4	4	2	2
	2.0	5	2.66	3840	41.3	37.9	67.6	4	6	4	4	2	2
	0.25	3	1.30	1422	15.3	15.6	56.7	3	4.5	4	4	2	3
	1.0	3	1.91	2585	27.8	24.2	60.0	4	5.5	4	4	2	3
	2.0	3	2.64	3982	42.8	36.4	65.4	4	6.5	4	4	2	3

Note: Coating weight iron content and adherence values are averages of three determinations except where indicated as follows:
* - average of two determinations.
** - single determinations.
Alloy thickness values are averages of at least six measurements on single samples.
Ductility and surface appearance ratings are averages of two determinations. For ductility, adherence and surface appearance rating codes, see Table 5 (a).

(continued)

TABLE 6 (Continued)

Average Coating Test Results

Test Number	Immersion Time min	Steel Finish	Coating Wt, oz/sq ft-sheet	Iron Content mg/sq ft	Iron Content g/m^2	Alloy Thickness mm x 10^{-3}	Proportion of Alloy %	Ductility	Adherence	Spangle Size	Spangle Contrast	Brightness	Roughness
9	0.25	5	0.57	260*	2.8*	1.9	15.7	1	1	4	4	2	2
	1.0	5	0.65	353*	3.8*	2.5	18.8	2	1	4	4	2	3
	2.0	5	0.86*	502	5.4	5.0	29.2	1	1	4	4	2	4
	0.25	3	0.54	205	2.2	1.9	18.1	2	1	3	3	2	3
	1.0	3	0.65	279	3.0	2.5	18.4	2	1	3	3	2	3
	2.0	3	0.99	502	5.4	5.0	25.8	2	1.5	4	3	2	4
10	0.25	5	0.53	344	3.7	1.9	17.0	1	1	1	2	1	1
	1.0	5	0.59	363	3.9	2.5	20.5	1	1	1	2	1	4
	2.0	5	0.74*	622*	6.7*	5.0	28.6	2	1	1	2	2	4
	0.25	3	0.48	279	3.0	1.9	16.7	2	1	1	3	2	2
	1.0	3	0.71	474	5.1	3.0	20.0	2	1	1	3	2	3
	2.0	3	1.01	882	9.5	5.0	25.1	3	1	1	3	2	4
11	0.25	5	0.56	325	3.5	1.7	15.8	1	1	3	3	2	1
	1.0	5	0.77	520	5.6	2.8	16.7	1	1	3	3	2	2
	2.0	5	1.18	817	8.8	5.0	20.1	2	1	3	3	2	3
	0.25	3	0.65	242	2.6	1.9	13.8	2	1	4	4	2	2
	1.0	3	1.03	530	5.7	3.0	13.7	2	1.5	4	4	2	3
	2.0	3	1.65	1302	14.0	10.0	28.4	4	2	2	3	2	3
12	0.25	5	0.61	279	3.0	1.9	15.4	1	1	3	2	3	2
	1.0	5	0.83	502	5.4	3.7	21.6	2	1.5	3	2	3	2
	2.0	5	1.53	1720	18.5	10.0	30.9	3	3	3	3	3	3
	0.25	3	0.65	270	2.9	1.9	14.3	2	1	3	2	3	2
	1.0	3	1.02	520	5.6	4.4	20.8	3	1.5	3	2	3	3
	2.0	3	1.76	1180	12.7	10.0	30.5	4	2.5	3	2	3	3
13	0.25	5	0.51	232	2.5	1.3	11.8	1	1	3	3	1	1
	1.0	5	0.59*	335*	3.6*	2.5	17.9	1	1	3	3	1	4
	2.0	5	0.83*	697	7.5	3.7	21.4	1	1	3	3	2	4
	0.25	3	0.54	223	2.4	1.3	11.3	1	1	3	3	1	2
	1.0	3	0.64	288	3.1	2.5	18.4	2	1	4	4	2	3
	2.0	3	0.91	455	4.9	3.7	19.7	1	1.5	4	4	2	4
14	0.25	5	0.62	325*	3.5*	1.3	10.4	1	1	2	1	2	4
	1.0	5	0.66	344	3.7	2.5	17.9	1	1	2	1	1	4
	2.0	5	0.81	595*	6.4	3.7	21.9	1	1	2	1	1	4
	0.25	3	0.58	251	2.7	1.9	15.0	1	1	2	2	1	3
	1.0	3	0.77	372	4.0	3.0	18.9	2	1	2	3	2	4
	2.0	3	1.05	595*	6.4*	5.0	20.7	3	1	2	3	3	4
15	0.25	5	0.51	232	2.5	2.5	23.6	1	1	3	3	2	2
	1.0	5	0.62	279	3.0	3.7	26.4	1	1	3	3	2	2
	2.0	5	0.75	427	4.6	5.0	31.4	1	1	3	3	2	4
	0.25	3	0.54	204	2.2	2.5	22.3	1	1	3	3	2	2
	1.0	3	0.68	279	3.0	3.7	25.4	1	1	4	4	2	3
	2.0	3	0.96	455	4.9	5.0	30.0	1	1	4	4	2	3
16	0.25	5	0.63	530	5.7	3.7	27.8	1	1	3	2	3	1
	1.0	5	1.41	1570**	16.9**	11.9	42.0	3	3.5	2	1	3	1
	2.0	5	2.41	3270*	35.2*	42.5	80.5	4	6	2	1	3	1
	0.25	3	0.56	297*	3.2*	3.0	25.2	2	1	3	3	3	2
	1.0	3	0.92	576	6.2	6.3	33.0	2	2	3	3	3	3
	2.0	3	1.75	1255*	13.5*	15.0	47.5	3	4	3	2	3	4
17	0.25	5	1.49	1922	20.7	17.5	53.6	3	3.5	4	4	3	2
	1.0	5	2.09	3157	34.0	24.2	54.9	3	5.5	4	4	3	3
	2.0	5	2.39	3960	42.6	30.7	60.4	3	6	4	4	3	3
	0.25	3	1.55	2055	22.1	17.1	52.2	3	3.5	4	4	3	2
	1.0	3	2.26	2760	29.7	24.6	53.7	4	5.5	4	4	3	3
	2.0	3	2.67	3460	37.2	30.7	55.0	4	7	4	4	3	3
18	0.25	5	0.53	195	2.1	1.3	11.2	1	1	3	3	1	1
	1.0	5	0.66	288	3.1	1.9	13.7	1	1	3	3	1	4
	2.0	5	0.78	465	5.0	2.9	17.6	1	1	3	3	2	4
	0.25	3	0.61	223	2.4	1.3	10.7	1	1	3	3	1	2
	1.0	3	0.70	353	3.8	2.1	14.2	2	1	3	3	2	2
	2.0	3	0.89	427	4.6	3.3	17.5	2	1	3	3	2	4

Note: Coating weight, iron content and adherence values are averages of three determinations except where indicated as follows:
* - average of two determinations.
** - single determinations.
Alloy thickness values are averages of at least six measurements on single samples.
Ductility and surface appearance ratings are averages of two determinations. For ductility, adherence and surface appearance rating codes, see Table 5 (a).

FIGURE I INFLUENCE OF BATH COMPOSITION, IMMERSION TIME AND STEEL SURFACE FINISH ON COATING WEIGHT

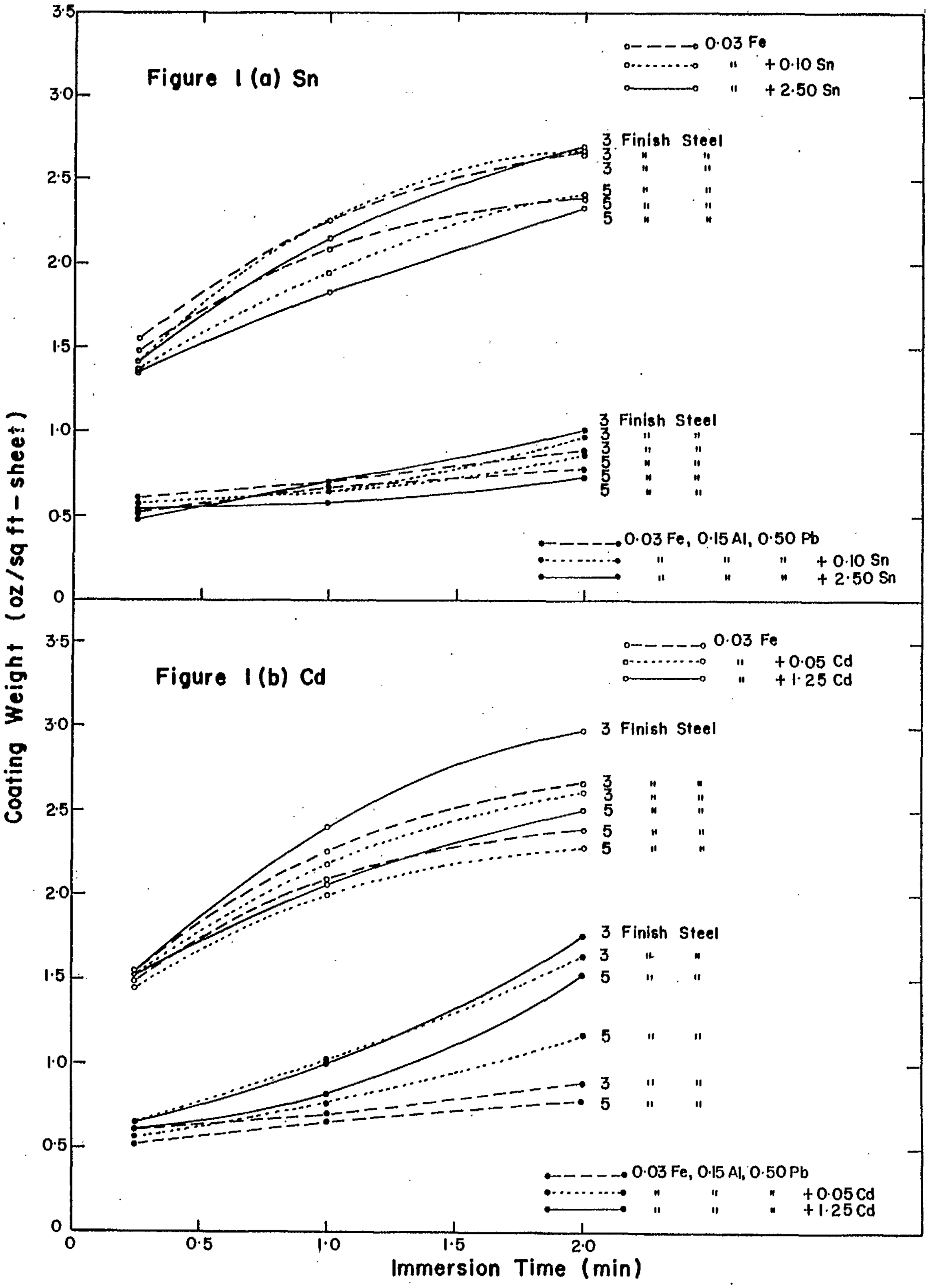


FIGURE 2 INFLUENCE OF BATH COMPOSITION, IMMERSION TIME AND STEEL SURFACE FINISH ON COATING WEIGHT

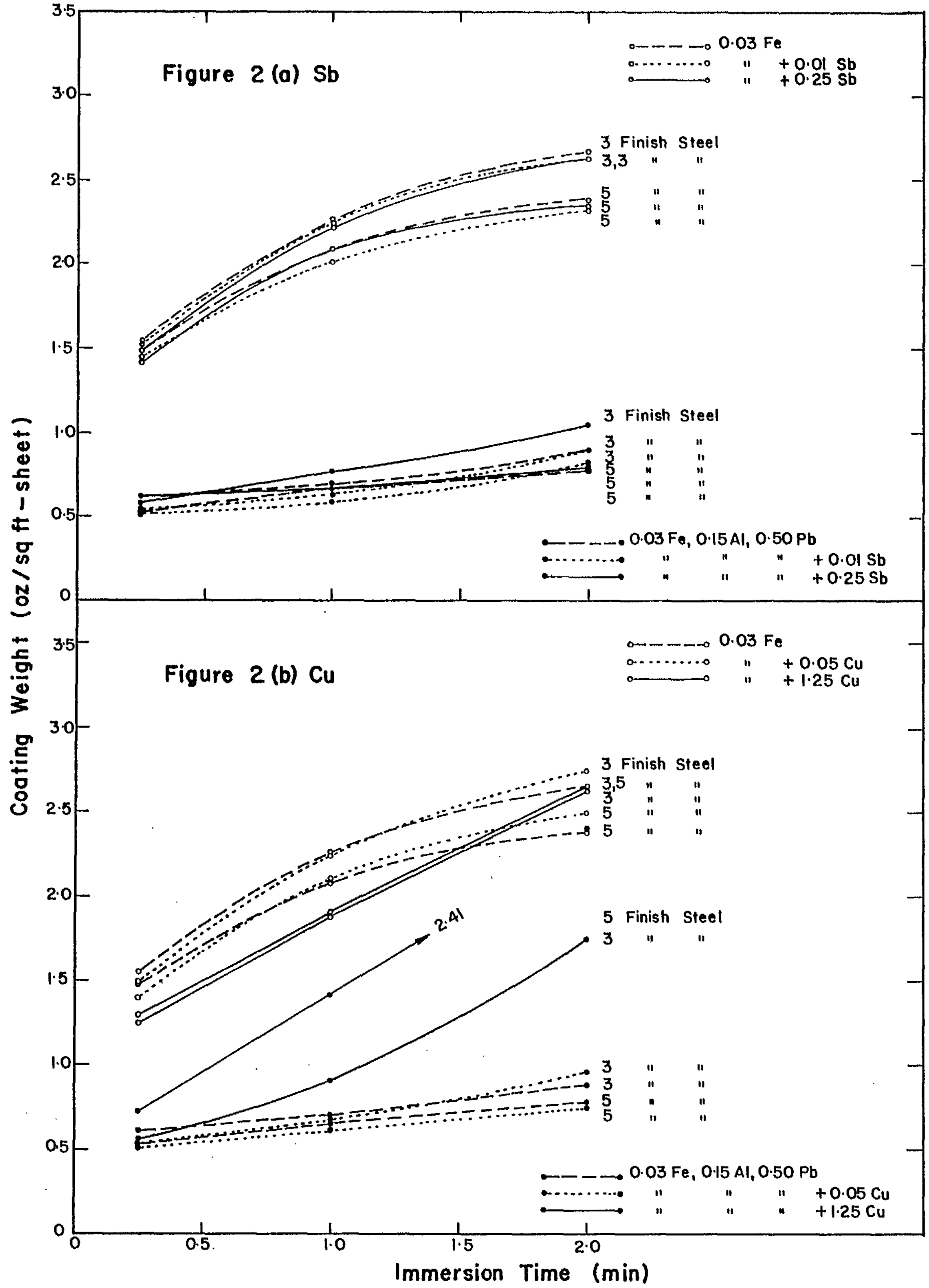
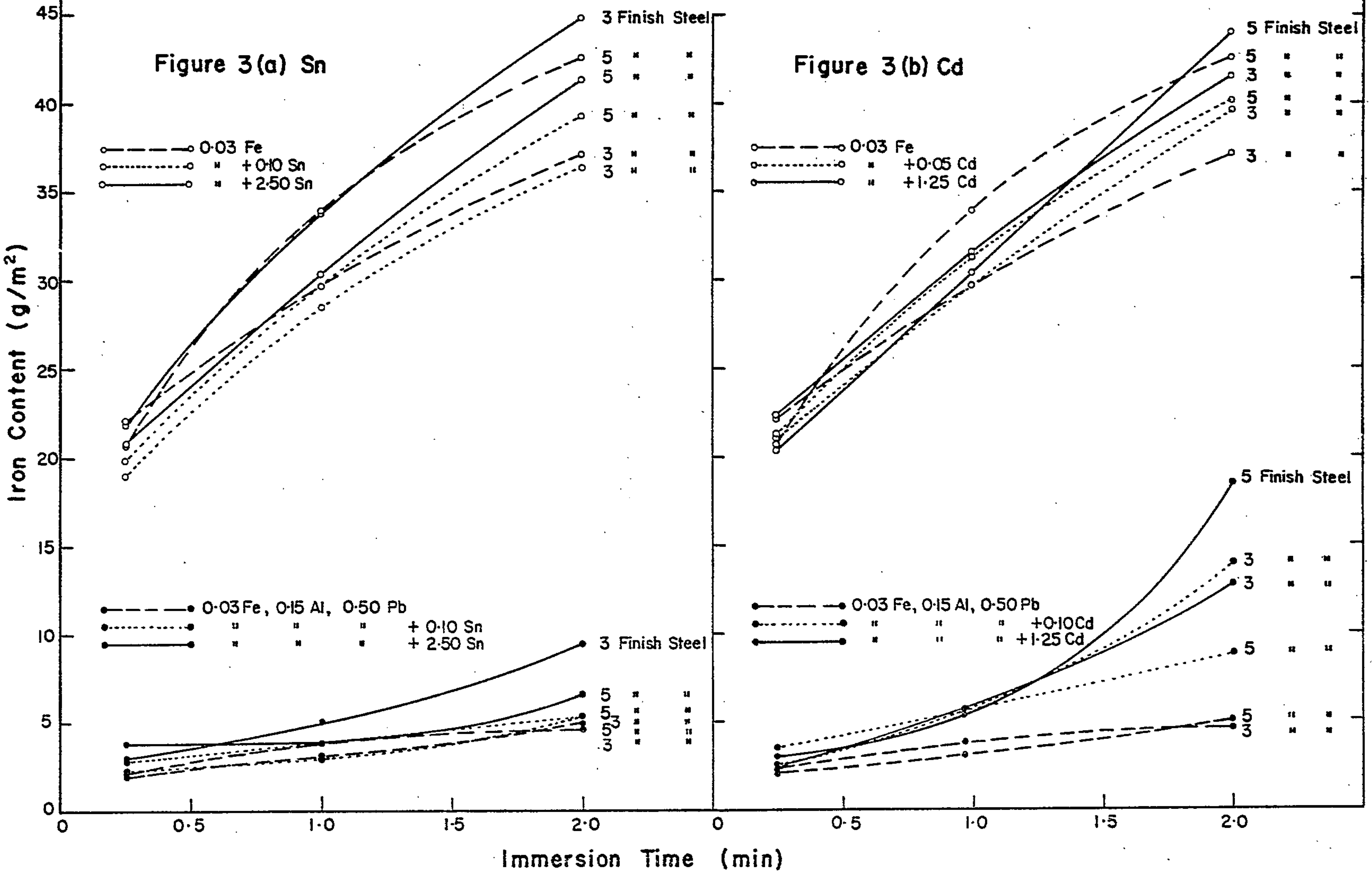


FIGURE 3 INFLUENCE OF BATH COMPOSITION, IMMERSION TIME AND STEEL SURFACE FINISH ON IRON CONTENT OF COATING



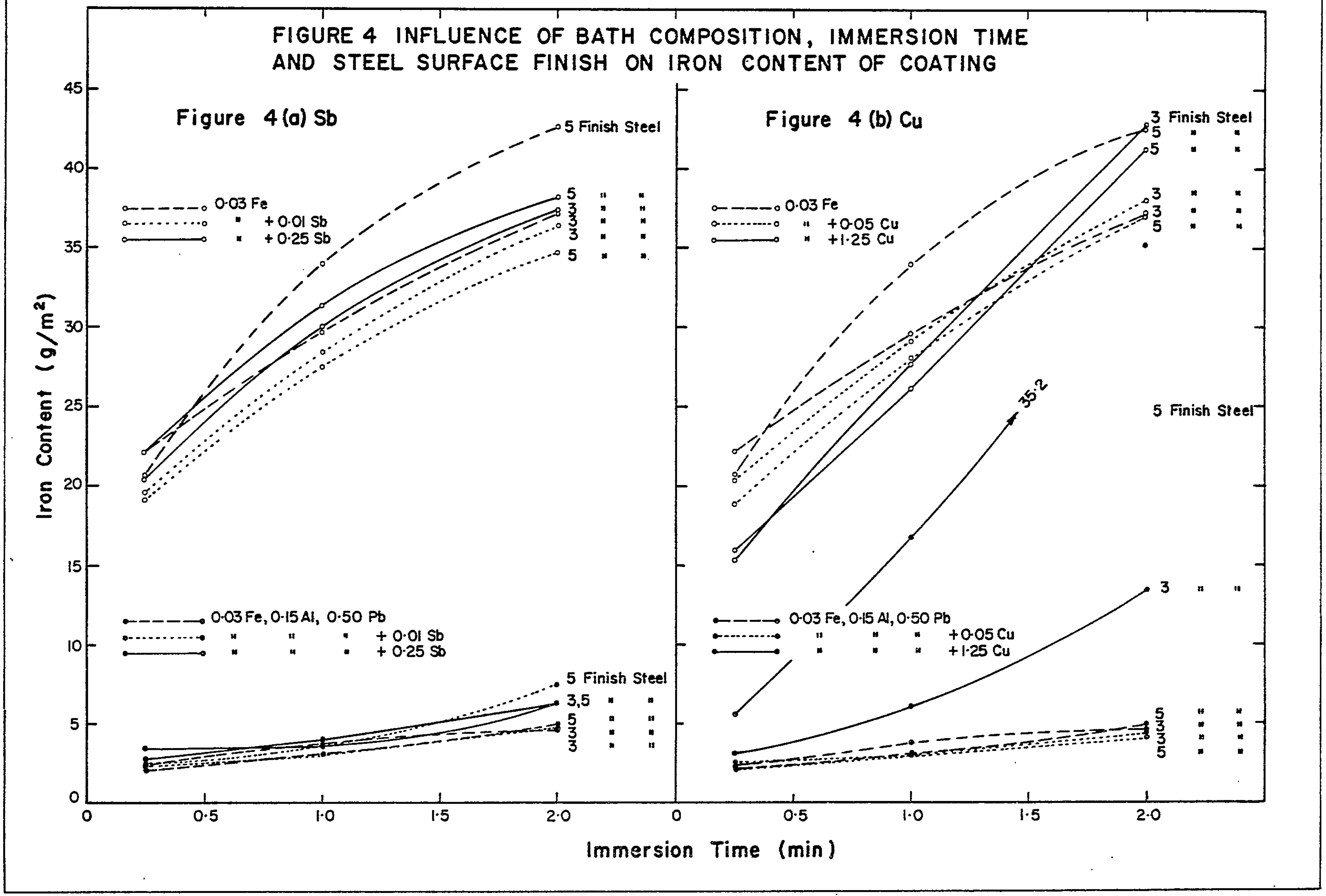


FIGURE 4 INFLUENCE OF BATH COMPOSITION, IMMERSION TIME
AND STEEL SURFACE FINISH ON IRON CONTENT OF COATING
Figure 4 (a) Sb
Figure 4 (b) Cu
Iron Content (g/m²)
Immersion Time (min)
0·03 Fe
" + 0·01 Sb
" + 0·25 Sb
0·03 Fe, 0·15 Al, 0·50 Pb
" " " + 0·01 Sb
" " " + 0·25 Sb
5 Finish Steel
3,5 " "
0·03 Fe
" + 0·05 Cu
" + 1·25 Cu
0·03 Fe, 0·15 Al, 0·50 Pb
" " " + 0·05 Cu
" " " + 1·25 Cu
3 Finish Steel
35·2
5 Finish Steel

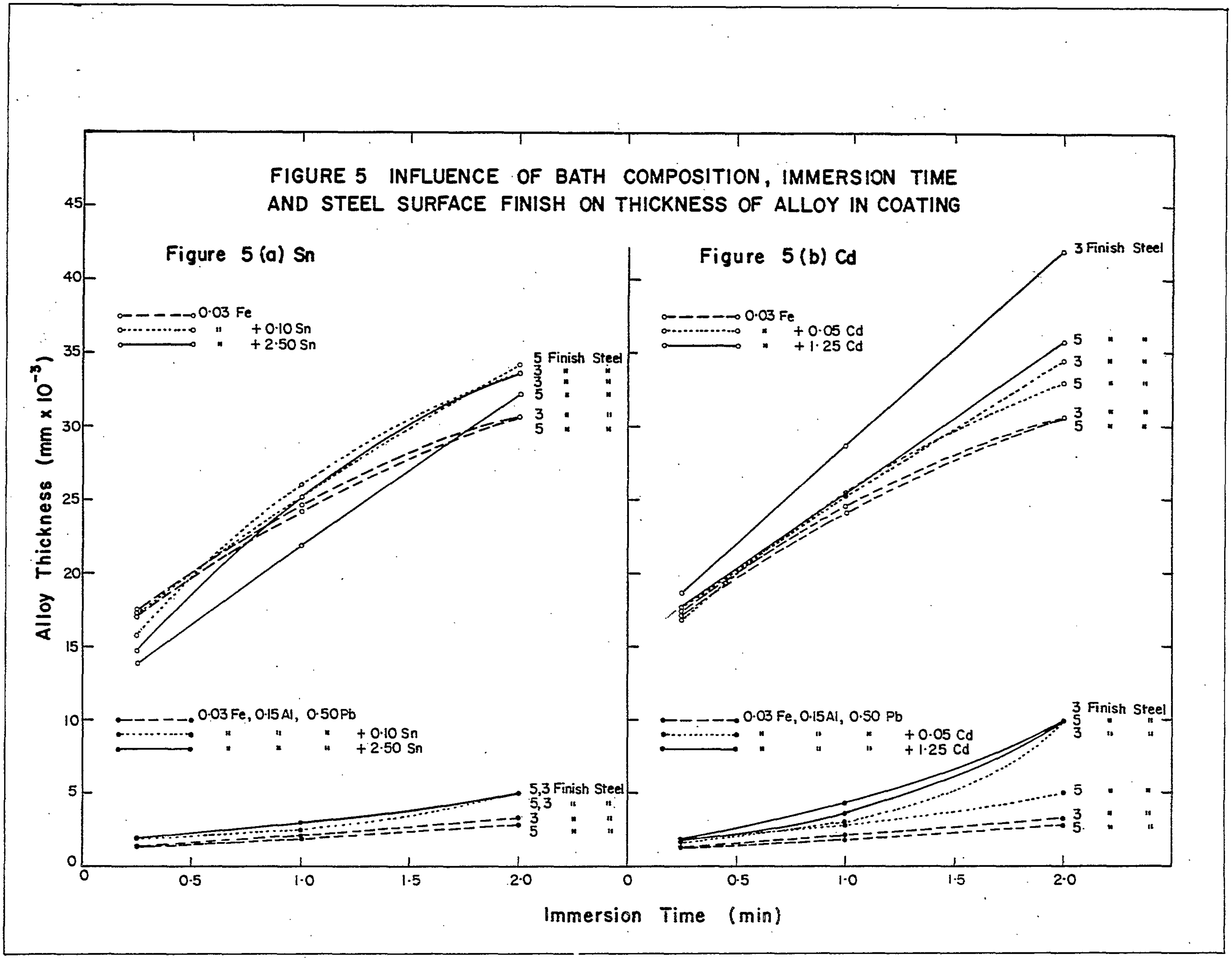


FIGURE 5 INFLUENCE OF BATH COMPOSITION, IMMERSION TIME AND STEEL SURFACE FINISH ON THICKNESS OF ALLOY IN COATING
Figure 5 (a) Sn
Figure 5 (b) Cd
0·03 Fe
" + 0·10 Sn
" + 2·50 Sn
0·03 Fe
" + 0·05 Cd
" + 1·25 Cd
0·03 Fe, 0·15 Al, 0·50 Pb
" " " + 0·10 Sn
" " " + 2·50 Sn
0·03 Fe, 0·15 Al, 0·50 Pb
" " " + 0·05 Cd
" " " + 1·25 Cd
5 Finish Steel
3 Finish Steel
5,3 Finish Steel
Alloy Thickness (mm x 10⁻³)
Immersion Time (min)

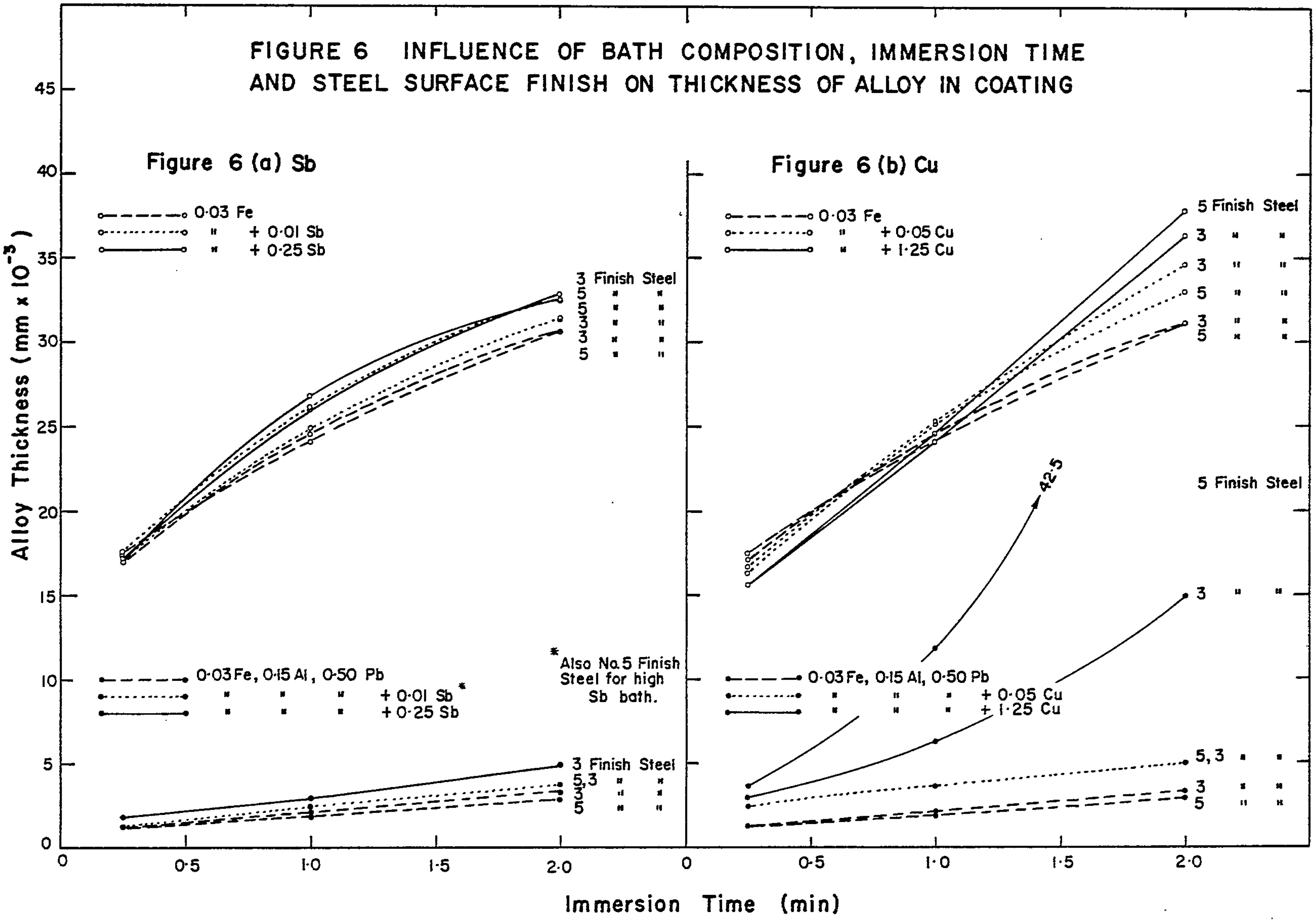


FIGURE 6 INFLUENCE OF BATH COMPOSITION, IMMERSION TIME AND STEEL SURFACE FINISH ON THICKNESS OF ALLOY IN COATING
Figure 6 (a) Sb
Figure 6 (b) Cu
Alloy Thickness (mm x 10^-3)
Immersion Time (min)
0·03 Fe
" + 0·01 Sb
" + 0·25 Sb
0·03 Fe, 0·15 Al, 0·50 Pb
" " " + 0·01 Sb*
" " " + 0·25 Sb
*Also No. 5 Finish Steel for high Sb bath.
3 Finish Steel
" + 0·05 Cu
" + 1·25 Cu
" " " + 0·05 Cu
" " " + 1·25 Cu
5 Finish Steel
42·5

FIGURE 7 INFLUENCE OF BATH COMPOSITION, IMMERSION TIME AND STEEL SURFACE FINISH ON PROPORTION OF ALLOY IN COATING

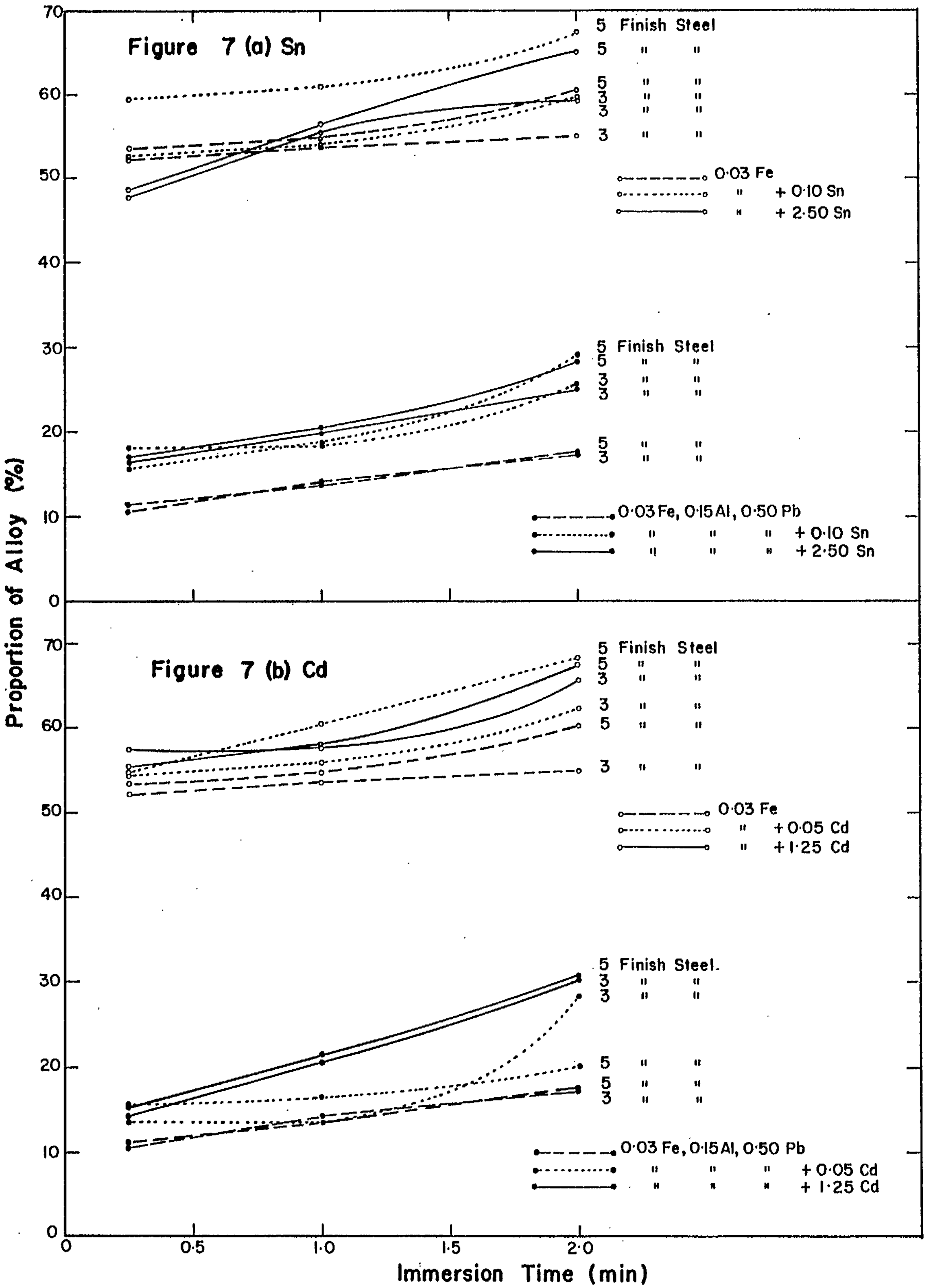
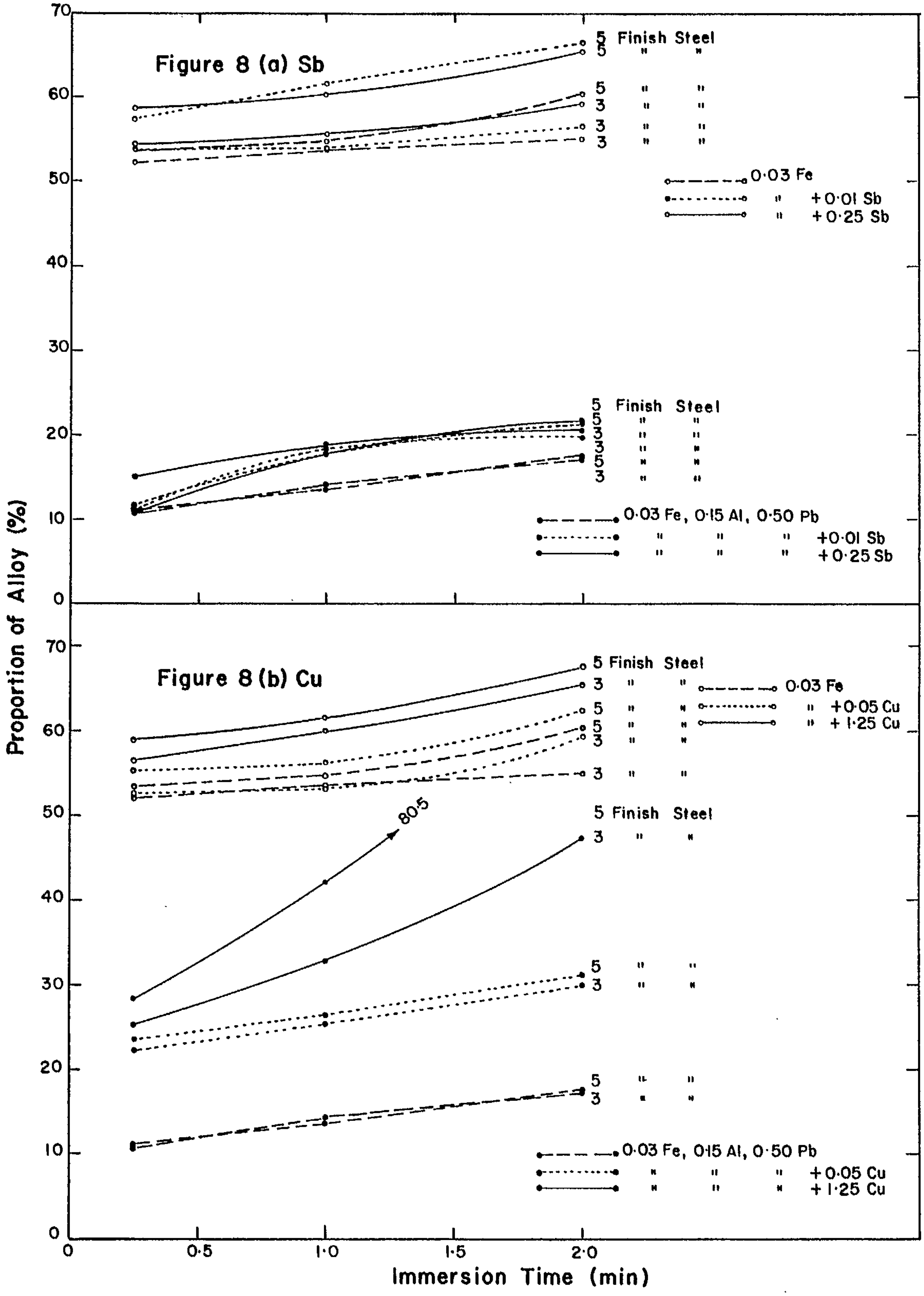
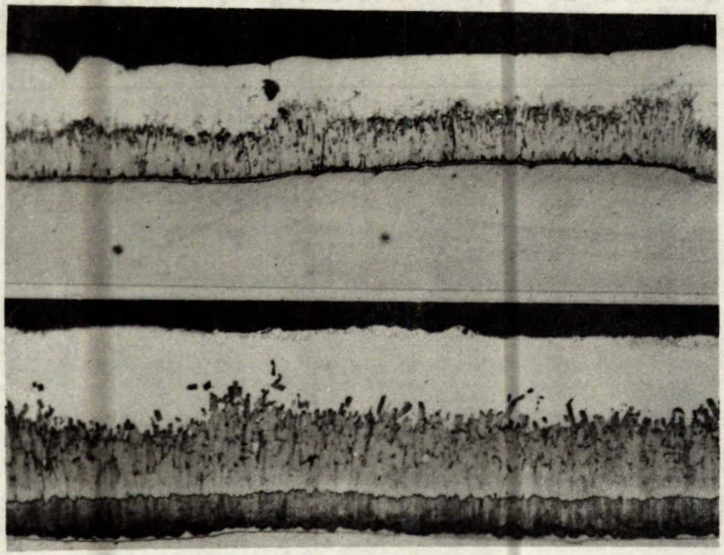


FIGURE 8 INFLUENCE OF BATH COMPOSITION, IMMERSION TIME AND STEEL SURFACE FINISH ON PROPORTION OF ALLOY IN COATING

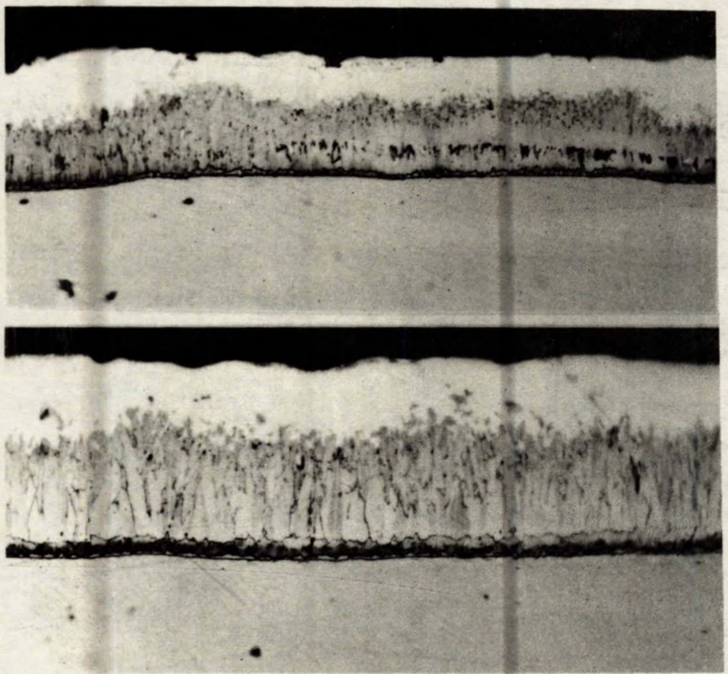




(a) 0.25 min dip, sample 1-3.

(b) 2 min dip, sample 1-7.

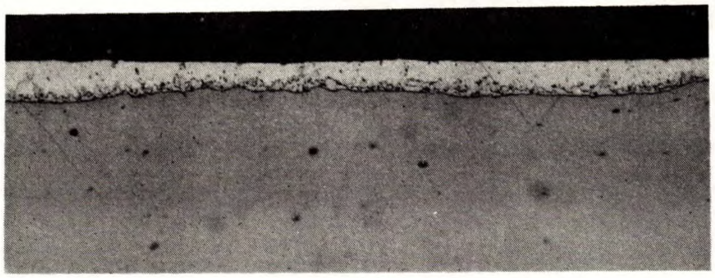
Fig. 9. - Typical coating microstructures on No. 5 finish steel with aluminum-free, low tin bath (0.03% Fe, 0.10% Sn). X500, nitramyl and picral etch.



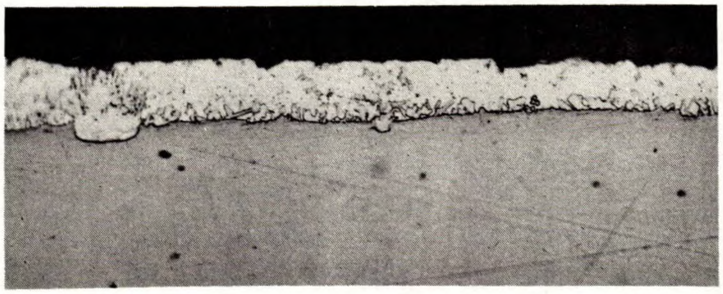
(a) 0.25 min dip, sample 2-1.

(b) 2 min dip, sample 2-7.

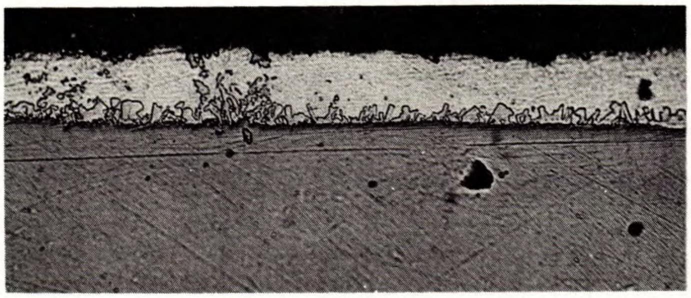
Fig. 10. - Typical coating microstructures on No. 5 finish steel with aluminum-free, high tin bath (0.03% Fe, 2.50% Sn). X500, nitramyl and picral etch.



(a) 0.25 min dip, sample 9-1. (0.03% Fe, 0.15% Al, 0.50% Pb, 0.10% Sn).

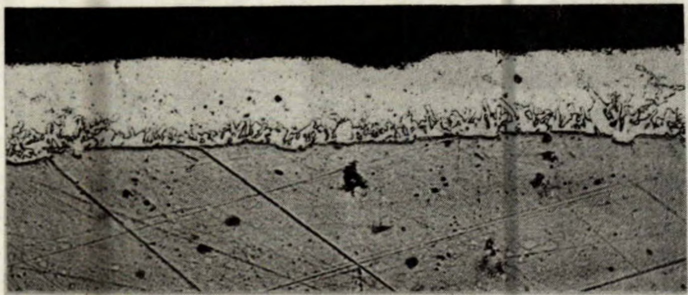


(b) 2 min dip, sample 9-7. (0.03% Fe, 0.15% Al, 0.50% Pb, 0.10% Sn).

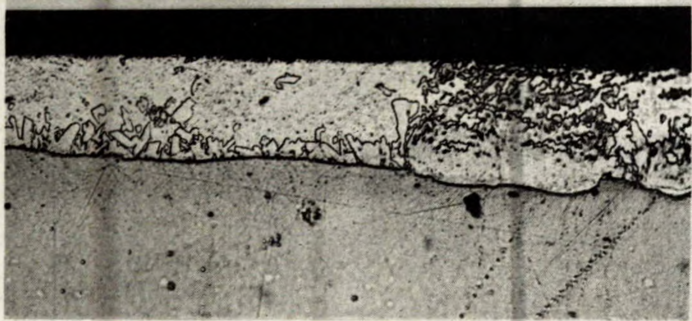


(c) 2 min dip, sample 10-7. (0.03% Fe, 0.15% Al, 0.50% Pb, 2.50% Sn).

Fig. 11. - Typical coating microstructures on No. 5 finish steel with aluminum-containing baths having low tin (a) (b) and high tin (c) content. X500, nitramyl etch.

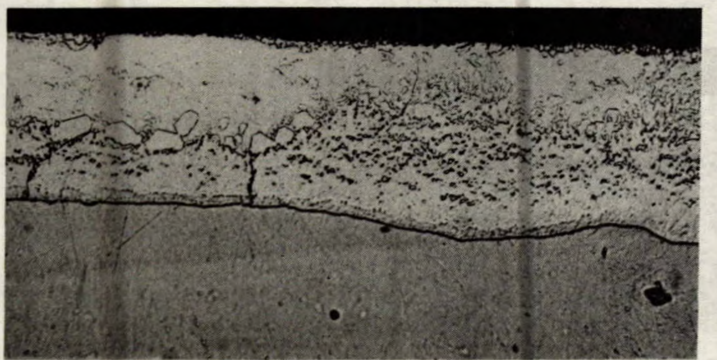


(a) 2 min dip, sample 11-7. (0.03% Fe, 0.15% Al, 0.50% Pb, 0.05% Cd).

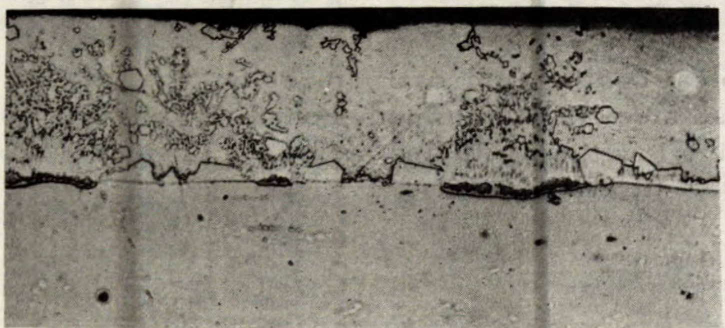


(b) 2 min dip, sample 12-7. (0.03% Fe, 0.15% Al, 0.50% Pb, 1.25% Cd).

Fig. 12. - Typical coating microstructures on No. 5 finish steel with aluminum-containing baths having low cadmium (a) and high cadmium (b) content.
X500, nitramyl etch.



(a) 2 min dip, sample 16-7.



(b) Another field on sample 16-7.

Fig. 13. - Typical coating microstructures on No. 5 finish steel with aluminum-containing, high-copper bath (0.03% Fe, 0.15% Al, 0.50% Pb, 1.25% Cu).
X500, nitramyl etch.