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MULTILAYERED PLATING THICKNESS MEASUREMENT

PDO 6984919, Topical Report

R. J. Russell, Project Leader

Project Team:
I. E. Donnell
G. R. Osbourn

Published March 1977

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Bendix Kansas City Division

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Technical Communications Bendix Kansas City Division

MULTILAYERED PLATING THICKNESS MEASUREMENT

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Prepared by R. J. Russell, D/142, under PDO 6984919

A nondestructive method for determining the thickness of a multilayered plating consisting of gold on rhodium on nickel ~~has been~~ *was* developed. Corrections for enhancement effects ~~have been~~ *were* determined and tabulated for X-ray fluorescence measurements.

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SUMMARY

A nondestructive method for measuring multilayered plating thickness has been developed utilizing intensity measurements made with an X-ray fluorescence spectrometer.

A series of multilayered platings was fabricated, and the thickness of each was determined through the use of a Link gage block interferometer. These samples were used in experiments to determine the absorption and enhancement effects that are present when an X-ray fluorescence spectrometer is used to measure the thickness of a multilayered plating.

The results of the investigation indicated that enhancement corrections must be applied to X-ray measurements of gold thickness up to 20 microinches (508 nm). The effect of absorption of the rhodium K-Alpha line in the gold layer was found to have an insignificant effect on the measurement of rhodium thickness; corrections are not needed.

Present technology results in an ability to detect a 1-microinch (25.4 nm) change in gold thickness with an estimated calibration uncertainty (bias) of ±4 microinches (±101.6 nm). The thickness of the rhodium underplate can be determined with an estimated uncertainty of ±10 microinches (254 nm).

Additional work is being performed to establish confidence in the plating-thickness measurement method that has been developed.

DISCUSSION

SCOPE AND PURPOSE

This project was initiated to determine nondestructive techniques for measuring the thickness of the multilayered plating materials on header assemblies for an electrical output switch. Of the available techniques, X-ray fluorescence was chosen for study because it offered the highest probability for success.

By its nature, X-ray fluorescence separates the output from each discrete layer of plating. Once a layer is separated and detected, corrections must be applied for the absorption and enhancement caused by adjacent layers.

This report describes the basic theory, the development of the measurement techniques, and the experiments performed to determine the magnitude of the required corrections.

ACTIVITY

Background

The fabrication of contact header assemblies for an electrical output switch manufactured by the Bendix Kansas City Division requires the application of a multilayered plating. As shown in Figure 1, the plating is composed of 5 microinches (0.127 μm) of gold on 50 microinches (1.270 μm) of rhodium on 200 microinches (5.080 μm) of nickel on a Kovar base.

Because adhesion problems are encountered when the plating process is interrupted, plating-thickness measurements must be made after all of the plating has been deposited. Since the cost of each header is lost when destructively tested after plating, and since the destructive test does not reveal the thickness of the gold, a nondestructive method is needed to determine the thickness of each layer of plating.

The available nondestructive measuring techniques include beta backscatter, X-ray fluorescence, and eddy currents. Of these, X-ray fluorescence was chosen for development.

Basic Theory

When an element absorbs X-rays having energy greater than a critical value, it will emit secondary X-rays which have wavelengths that are characteristic of that particular element. The X-ray fluorescence spectrometer is used to detect characteristic wavelengths from a specific element. The intensity of the characteristic X-rays is a function of the number of atoms of the element

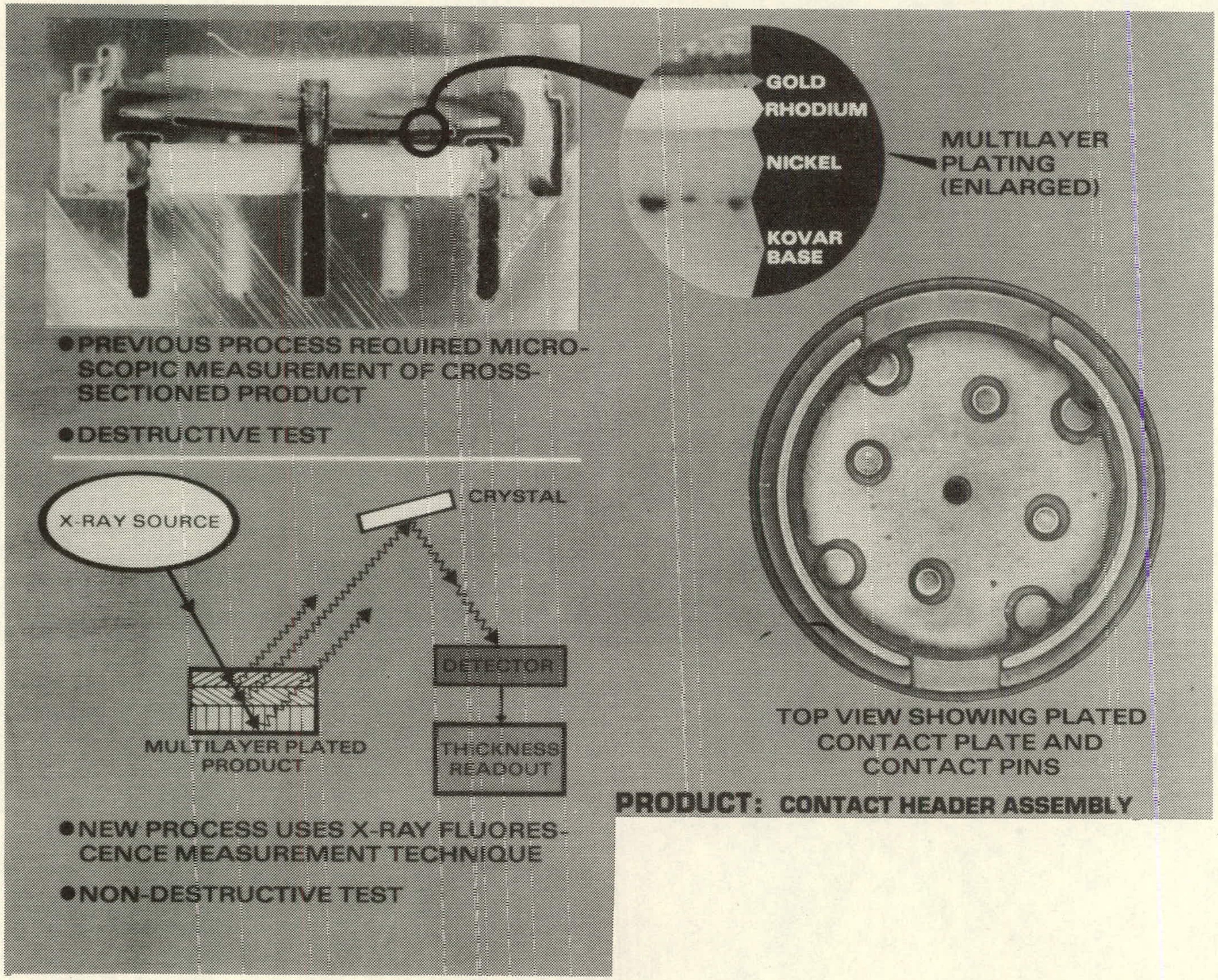

Figure 1. Thickness Measurement of Multilayered Plating

that is present in the sample. Thus if the sample is of uniform density, the intensity increases with an increase in the thickness.

During the measurement of plating thickness, the various elements are located in discrete layers at the surface. When exposed to X-ray bombardment, each layer emits its characteristic wavelengths. The wavelength of the desired element is separated from those of the others, and its intensity is detected by the spectrometer.

The X-rays emitted from a given layer of plating will be attenuated by the absorption in the layers above. In addition, an increase in emitted X-rays may be caused by enhancement (sometimes called secondary fluorescence). This occurs when the fluorescence from one layer has sufficient energy to produce additional fluorescence in the layer under investigation. Because both phenomena can occur in a multilayered plating, the determination of correction factors was made necessary. The proper application of these correction factors makes possible a quantitative determination of the thickness of each layer of a multilayered plating.

The intensity of the fluorescent radiation can be expressed by the following equation.

$$\frac{I}{I_m} = (1-e^{-kt}), \qquad (1)$$

where

I = observed intensity,

I_m = maximum possible intensity corresponding to an infinite thickness,

k = effective self-absorption coefficient, and

t = sample thickness.

The rhodium K-Alpha line and the gold L-Beta line were chosen for use because of their intensity and low self-absorption.

Standards

The measurement of plating thickness is performed using a comparative instrument which requires a set of reference thickness standards. These standards should provide a traceable reference which can be reproduced at a later time. The basic gold-on-nickel and rhodium-on-nickel references were purchased from a commercial source and were calibrated by the Bendix Kansas City Metrology Laboratory (Figure 2).

Figure 2. Gold-On-Nickel Plating Standards

References for the enhancement and absorption study were fabricated and measured at Bendix Kansas City. The basic structure of each reference consisted of a Kovar substrate whose faces had been lapped flat and parallel within 2 microinches (50.8 nm). The thickness of the substrate was determined using a Link gage block interferometer. A series of platings, identified later in this report, was deposited on each substrate and the thickness of each was determined with the interferometer. These references provide an effective means of evaluating enhancement and absorption effects (Table 1).

Correlation of Thickness Measurements of Rhodium Plating Under Gold

The presence of a thin gold overplate is known to change the apparent rhodium thickness measured with an X-ray fluorescence spectrometer. The object of this experiment was to determine the amount of the change.

The X-ray spectrometer was calibrated (X-ray intensity versus thickness) using the rhodium-on-nickel references. The apparent rhodium thickness of the Kovar-based reference parts was determined before and after each gold overplate (Table 2 and Figure 3). The experiment showed the magnitude of the absorption effect to be less than 5 microinches (127.0 nm) for gold overplates up to 10 microinches (254.0 nm) in thickness. This error is insignificant for the intended application and will be ignored.

Correlation of Thickness Measurements of Gold Plating

Because there is a possibility that the fluorescent radiation emitted from the gold layer will be enhanced by the radiation emitted from the underlying rhodium layer, the following experiment was performed to determine the magnitude of error in the determination of the gold thickness.

The measurement technique and calibration procedure used in this experiment were identical to those used in the previously described experiment except that gold-on-nickel calibration standards were employed. The object of the experiment was to determine the amount of change in the apparent gold thickness that resulted from the rhodium underplate (Table 3). The effect of four different thicknesses of rhodium (Samples 1, 2, 4, and 10) on the X-ray thickness measurement is shown in Table 4. The data show that the enhancement effect caused by the four different thicknesses of rhodium underplate was constant.

Figure 4 was developed to show the relationship between the gold intensity ratio and the gold thickness. This information reveals that the enhancement correction is dependent upon the gold thickness and, for practical purposes, is independent of the rhodium thickness. Corrections for measuring the thickness of gold over rhodium were determined (Table 5). This information is plotted in Figure 5.

By using the method described and applying the derived corrections, the estimated uncertainty in the measurement of gold having a

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Table 1. Sample Plating Thicknesses Determined Using Link Interferometer

Sample	Woods Nickel Thickness (Microinches)*	Rhodium Thickness (Microinches)	Gold Thickness for Overplate Indicated: Number 1 (Microinches)	Number 2 (Microinches)	Number 3 (Microinches)	Number 4 (Microinches)
1		28	1	5	7	13
2		18	1	6	9	23
4		44	1	4	7	18
5	2					
8	2					
10		75	1	6	9	15

*1 microinch = 25.4 nm.

Table 2. Rhodium Data, Before and After Application of Gold Plating, Using X-Ray Spectrometer

Sample	Gold Thickness (Micro-inches)*	Rhodium Thickness (Micro-inches)	Corrected Standard Intensity (A) (Counts/Sec.)	Corrected Rhodium Count (B)	Ratio (B/A)	Apparent Thickness (Micro-inches)
1	0	28	30067	1641	0.055	28
1	1	28	30209	1849	0.061	32
1	4	28	29800	1731	0.058	30
1	9	28	29461	1484	0.050	29
1	13	28	29947	1587	0.053	29
2	0	18	30067	930	0.031	18
2	1	18	30209	1053	0.035	19
2	6	18	29800	983	0.033	18
2	8	18	29461	879	0.030	17
2	23	18	29947	920	0.031	18
4	0	44	30067	2668	0.089	44
4	1	44	30209	2975	0.098	50
4	4	44	29800	2789	0.094	47
4	9	44	29461	2498	0.085	46
4	18	44	29947	2588	0.086	44
10	0	70	30067	4234	0.141	70
10	1	70	30209	4779	0.158	80
10	6	70	29800	4422	0.148	75
10	9	70	29461	3838	0.130	68
10	15	70	29947	4142	0.138	70

*1 microinch = 25.4 nm.

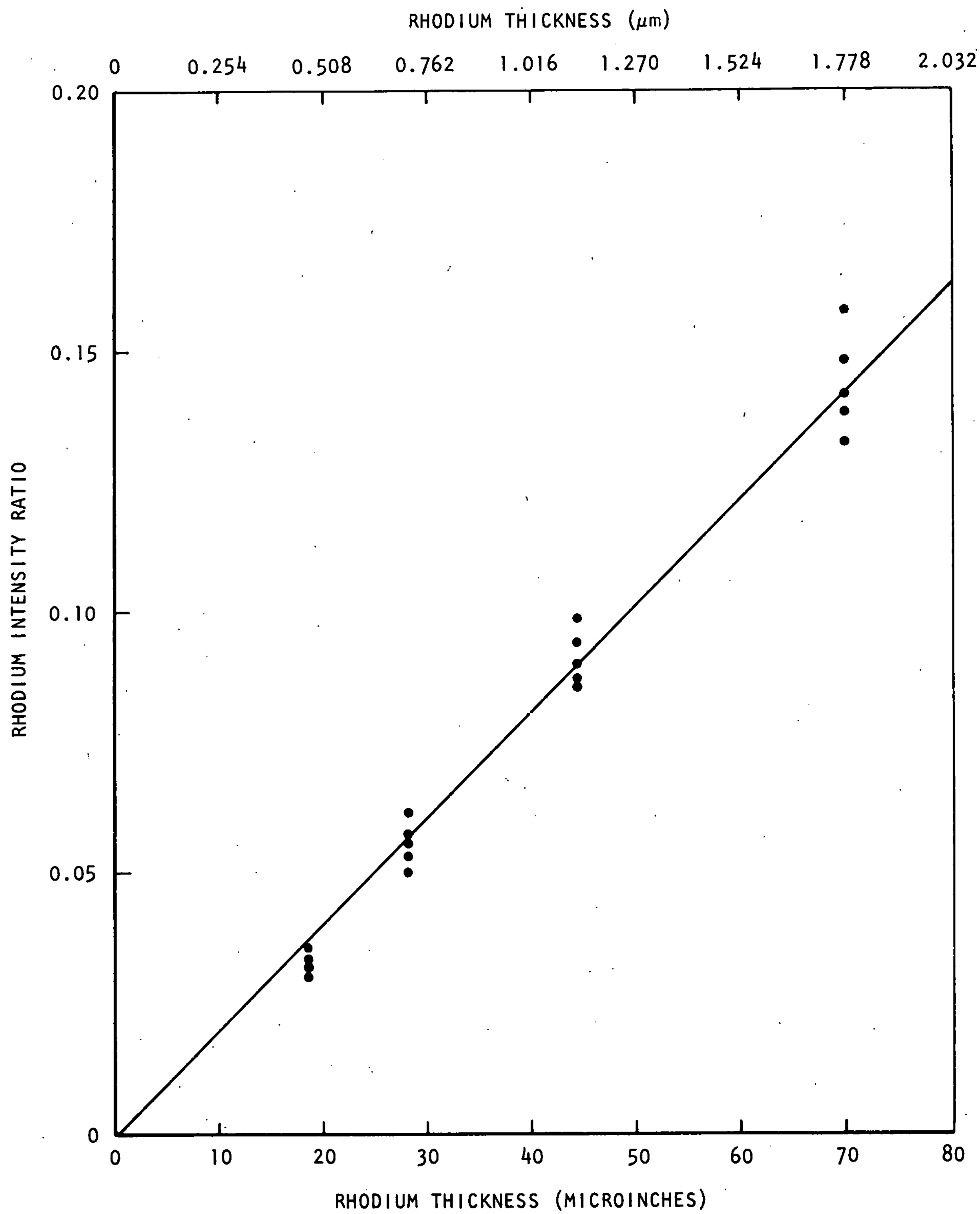

Figure 3. Rhodium Intensity Ratio Versus Rhodium Thickness (Samples include rhodium substrate and four gold overplates.)

Table 3. Gold Thicknesses Determined Using X-Ray Spectrometer

Sample	Rhodium Underplate Thickness (Microinches)*	Gold Thickness for Overplate Indicated			
		Number 1 (Micro-inches)	Number 2 (Micro-inches)	Number 3 (Micro-inches)	Number 4 (Micro-inches)
1	28	2	9	13	20
2	18	2	8	12	19
4	44	3	9	13	20
10	70	2	9	13	20

*1 microinch = 25.4 nm.

thickness up to 10 microinches (254 nm) is ±4 microinches (±101.6 nm). A variation of 1 microinch (25.4 nm) in gold thickness can be detected by this method.

Measurements

The measurement of the gold and rhodium plating thickness on contact headers is performed by comparison through the use of an X-ray spectrometer. The following typical sequence of events occurs during a measurement.

The X-ray spectrometer is adjusted to detect the rhodium K-Alpha line. Rhodium-on-nickel standards are used to establish a relationship between the rhodium thickness and the X-ray intensity (count relationship). The part (contact header) is placed on the instrument and measured, and the thickness-versus-count relationship is applied to the result to determine the rhodium thickness.

The X-ray spectrometer then is adjusted to detect the gold L-Beta line, and gold-on-nickel standards are used to establish a gold-thickness versus X-ray-intensity (count) relationship. The part (contact header) is placed in the instrument and measured and, using the established relationship, the X-ray intensity data from the part are converted to gold thickness.

Gold-thickness values of less than 20 microinches (508 nm) require enhancement corrections; the graph shown in Figure 5 should be consulted for the magnitude of the correction factor. The actual gold thickness is determined by multiplying the uncorrected gold-thickness value by the correction factor.

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Table 4. Gold Data, for Gold Deposited on Rhodium

Sample	Gold Thickness (Microinches)*	Rhodium Thickness (Microinches)	Corrected Standard Intensity (Counts/Sec.)	Corrected Gold Count	Ratio
1	1	28	7731	166	0.022
1	4	28	7631	852	0.112
1	9	28	7665	1094	0.143
1	13	28	7725	1771	0.229
2	1	18	7731	141	0.018
2	6	18	7631	770	0.101
2	8	18	7665	1047	0.137
2	23	18	7725	1673	0.217
4	1	44	7731	249	0.032
4	4	44	7631	858	0.112
4	9	44	7665	1079	0.141
4	18	44	7725	1756	0.227
10	1	70	7731	187	0.024
10	6	70	7631	840	0.110
10	9	70	7665	1085	0.142
10	15	70	7725	1797	0.233

*1 microinch = 25.4 nm.

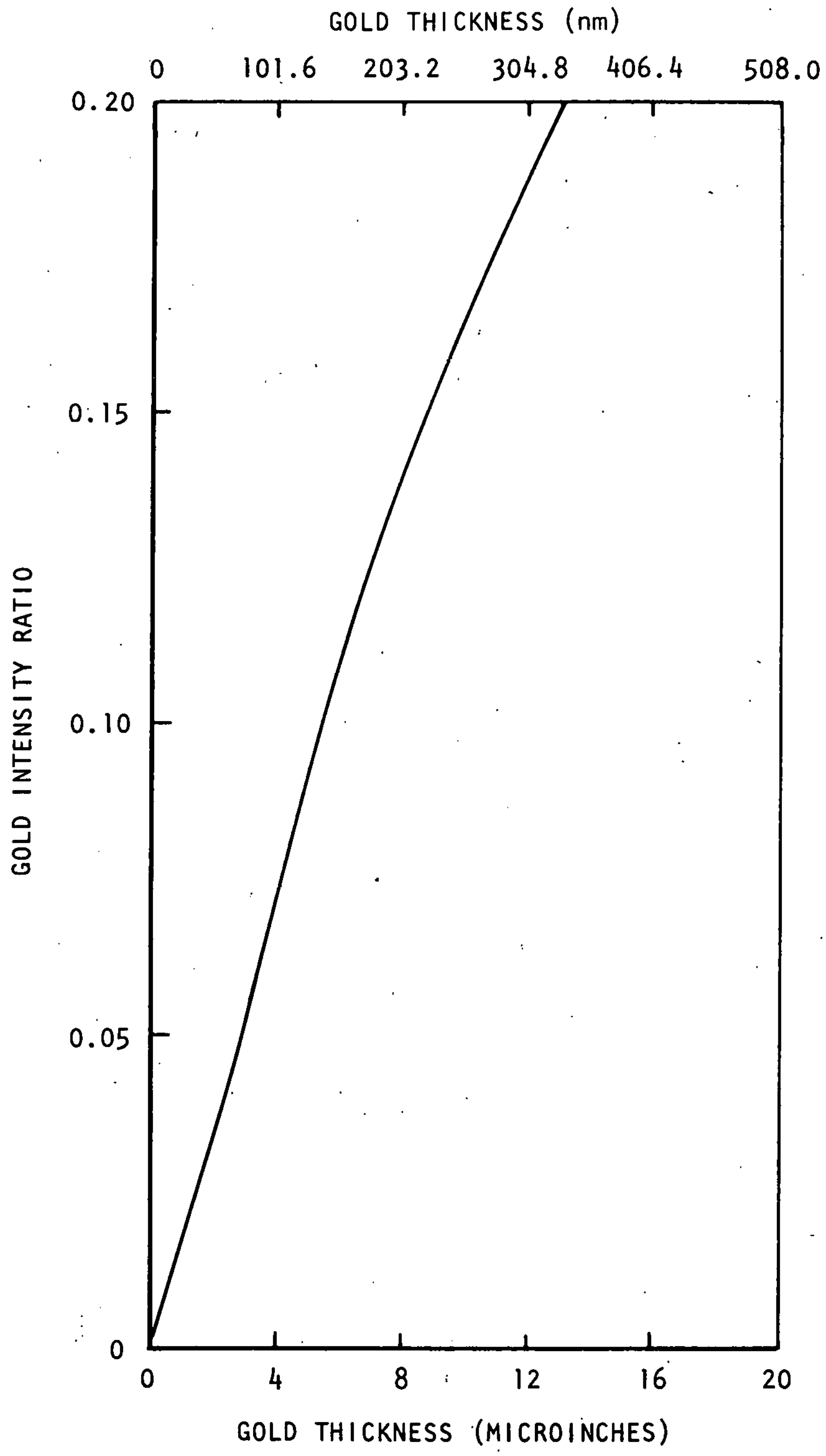

Figure 4. Gold Intensity Ratio Versus Gold Thickness

Table 5. Correction Data for Measuring Gold Thickness Over Rhodium Plating

Sample	Corr. Standard Intensity (Counts/Sec.)	Corr. Gold Sample (Counts/Sec.)	Ratio	Link Thickness (Microinches)*	X-Ray Thickness (Microinches)	X-Ray Corr. Factor	X-Ray Corr. Thickness (Microinches)
1	7731	166	0.022	1	2	0.5	1.0
2	7731	141	0.018	1	2	0.5	1.0
4	7731	249	0.032	1	3	0.5	1.5
10	7731	187	0.024	1	2	0.5	1.0
1	7631	852	0.112	4	9	0.6	5
2	7631	770	0.101	6	8	0.6	5
4	7631	858	0.112	4	9	0.6	5
10	7631	840	0.110	6	9	0.6	5
1	7665	1094	0.143	9	13	0.7	9
2	7665	1047	0.137	8	12	0.7	8
4	7665	1079	0.141	9	13	0.7	9
10	7665	1085	0.142	9	13	0.7	9
1	7725	1771	0.229	13	20	0.9	18
2	7725	1673	0.217	23	19	0.9	17
4	7725	1756	0.227	19	20	0.9	18
10	7725	1797	0.233	15	20	0.9	18

*1 microinch = 25.4 nm.

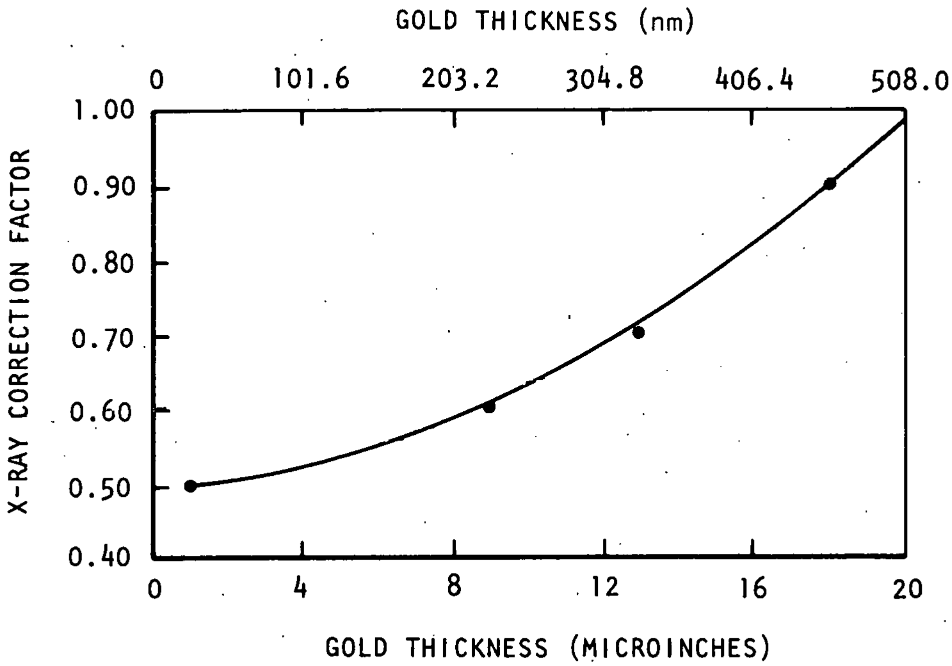

Figure 5. Graph for Determination of X-Ray Correction Factor for Gold-Over-Rhodium Plating

When this method is used, the density must remain constant since the measured parameter is mass/area. The value obtained is converted to thickness by the use of calibration standards having platings that approach the theoretical density.

Gold-thickness variations of 1 microinch (25.4 nm) can be detected through the use of the described method. The estimated uncertainty in the measurement of gold thickness is ±4 microinches (±101.6 nm). The estimated uncertainty in the measurement of rhodium-underplate thickness is ±10 microinches (±254 nm).

ACCOMPLISHMENTS

A technique has been developed to determine the thickness of the gold and rhodium plating on contact headers. Standards to support this measurement have been developed and are now in service.

The method described requires the application of correction curves in determining the gold thickness. The curves have been generated by a small amount of data, and additional documentation is necessary to provide confidence in their accuracy.

FUTURE WORK

Future work on this project will consist of the following endeavors.

- The effect of angular orientation of the contact header in the X-ray fixture will be evaluated.
- A mathematical model will be developed to allow the use of an infinite-thickness specimen as a reference.
- The effect of surface texture on thickness measurements will be determined.
- A capability study of the measurement system will be made using typical sample parts.
- The accuracy of the correction curve for gold-over-rhodium versus X-ray thickness (Figure 5) will be verified.
- A nondestructive method of determining nickel thickness will be developed.

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