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MULTILAYERED PLATING THICKNESS

Final Report

R. J. Russell, Project Leader

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

Published August 1978

Prepared for the United States Department of Energy
Under Contract Number EY-76-C-04-0613.

**Kansas City
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MULTILAYERED PLATING THICKNESS

BDX-613-1795 (Rev.), UNCLASSIFIED Final Report, Published August 1978

Prepared by R. J. Russell

A nondestructive technique utilizing the X-ray fluorescence spectrometer has been developed for measuring the thickness of both single and multiple layers of plating. Thickness standards and calibration curves also have been developed to support the measurements. Mathematical models for equating the intensity of the fluorescent radiation to the plating thickness are presented.

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The Bendix Corporation
Kansas City Division
P. O. Box 1159
Kansas City, Missouri 64141

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SUMMARY

This project was initiated to develop a method for determining nondestructively the thickness of multilayered platings on production parts. The thickness of the gold plating was suspected of causing torque problems in the final assembly of an output switch. The existing measurement technique had destroyed a \$120.00 part while failing to measure the thickness of the gold surface-plating.

A technique utilizing an X-ray fluorescence spectrometer was developed to measure the thickness of both the gold and rhodium platings on the part. Standards were developed to support the measurement technique, which is analytically and experimentally evaluated in this report. The employment of a mathematical model simplifies the thickness measurement by requiring the use of only one calibration standard, thereby reducing the calibration time.

Methods for measuring the thickness of the nickel underplate also are evaluated.

This project is being terminated; the developed technology will not be implemented at the present time for the following reasons:

- The availability of low-cost sample parts now makes destructive testing economically acceptable; and
- The thickness of the gold plating on the part is no longer specified.

DISCUSSION

SCOPE AND PURPOSE

The fabrication of A-contact-header assemblies for electrical output switches manufactured by Bendix Kansas City requires the application of a multilayered plating consisting of $0.127\text{ }\mu\text{m}$ (5 microinches) of gold on $1.27\text{ }\mu\text{m}$ (50 microinches) of rhodium on $5.08\text{ }\mu\text{m}$ (200 microinches) of nickel on a Kovar base. Sample parts valued at \$120.00 each are sectioned to measure the thickness of the various layers of plating under a microscope. Only limited data are obtained from this measurement since the thickness of the gold layer, which has been blamed for torque problems in the final assembly, cannot be determined by the method used.

This project was initiated to minimize the cost of product inspection by providing a nondestructive measurement technique which also would be capable of measuring the thickness of the gold. The objective was to implement the developed technology in the production area by acquiring or fabricating the necessary equipment and providing the required training for personnel.

PRIOR WORK

Beta-backscatter techniques were briefly investigated for measuring the thickness of the gold plating on A-contact-header assemblies, but they subsequently were abandoned because of their inability to differentiate between gold and rhodium.

ACTIVITY

As previously reported, a technique utilizing X-ray fluorescence spectrometry has been developed to measure the thickness of the gold- and rhodium-plating layers.¹ This method, illustrated in Figure 1, requires excitation of the characteristic X-rays of the plating and a measurement of their intensity. An X-ray tube is used to fluoresce the specimen, and the fluorescent radiation is separated and measured with an X-ray spectrometer.

The following described activity has been completed since February, 1976.

Mathematical Model

A mathematical expression for plating thickness has been developed for use with the X-ray fluorescence spectrometer. The equation uses the intensity of the fluorescent radiation from a pure slug of plating material and from the sample under evaluation to calculate thickness.

Figure 1. Thickness Measurement of Multilayered Plating

The following equation is used for determining the thickness of a single layer of plating; its derivation has been previously reported:

$$\frac{F_t}{F_{t\infty}} = 1 - e^{-\left(\frac{\mu_1}{\sin \theta_1} + \frac{\mu_2}{\sin \theta_2}\right)t}, \quad (1)$$

where, as illustrated by Figure 2,

F_t = fluorescent-radiation intensity from the sample,

$F_{t\infty}$ = fluorescent-radiation intensity from a pure slug of the plating material,

μ_1 = mass-absorption coefficient of the incident X-ray beam in the plating,

μ_2 = mass-absorption coefficient of the fluorescent radiation in the plating,

θ_1 = angle between the incident X-ray beam and the plating,

θ_2 = angle between the fluorescent radiation and the plating, and

t = plating thickness (cm).

Equation 1, when arranged to calculate plating thickness, becomes

$$t = \frac{1}{\frac{\mu_1}{\sin \theta_1} + \frac{\mu_2}{\sin \theta_2}} \log_e \left(1 - \frac{F_t}{F_{t\infty}} \right). \quad (2)$$

When a two-layer composite plating is to be measured, as illustrated in Figure 3, the thickness of the overplate can be determined through the use of Equation 2. The thickness of the underplate can be determined through the use of Equation 3; the derivation of this equation is shown in the Appendix of this report:

$$x = - \frac{1}{\frac{\mu_3}{\sin \theta_1} + \frac{\mu_4}{\sin \theta_2}} \log_e \left[1 - \frac{F_x}{F_{x\infty} \left(1 - \frac{F_t}{F_{t\infty}} \right)^{\frac{\mu_5}{\mu_1}}} \right], \quad (3)$$

Figure 2. Determination of Single-Layer Plating Thickness

Figure 3. Determination of Underplate Thickness

where

x = thickness of the underplate (cm),

μ_3 = mass-absorption coefficient of the incident X-ray beam in the underplate,

μ_4 = mass-absorption coefficient of the fluorescent radiation in the underplate,

θ_1 = angle between the incident X-ray beam and the plating,

θ_2 = angle between the fluorescent radiation and the plating,

F_x = fluorescent-radiation intensity from the underplate,

$F_{x\infty}$ = fluorescent-radiation intensity from a pure slug of the underplate material,

F_t = fluorescent-radiation intensity from the overplate,

$F_{t\infty}$ = fluorescent-radiation intensity from a pure slug of the overplate material,

μ_1 = mass-absorption coefficient of the incident X-ray beam in the overplate, and

μ_5 = mass-absorption coefficient of the underplate's fluorescent radiation in the overplate.

Evaluation of Mathematical Model for Calculating Thickness of Overplate

The mathematical model was evaluated by comparing the results of its performance with those obtained using a set of reference plating-thickness standards. Both techniques involve equating the measured intensity of the fluorescent radiation from the samples to the thickness. The two techniques differ in the methods used for calibration and in the time required to obtain thickness values.

The technique now in use requires a set of six plating-thickness standards for calibration. A curve is plotted through the data points to provide a relationship between the counts/second and the thickness. The thickness of a sample part is determined by measuring the intensity in counts/second of the fluoresced radiation with an X-ray spectrometer and converting the value obtained to thickness through use of the calibration curve.

Use of the mathematical model requires a less rigorous calibration technique. A slug of plating material which appears infinitely thick to the incident X-ray beam constitutes the required standard. The measured intensity (in counts/second) from both the slug and the sample part is substituted in the mathematical model to determine the thickness. Parameters such as the spectrometer geometry and the mass-absorption coefficients must be defined for the equipment and materials involved.

Using the present equipment, the following parameters were determined:

- $\theta_1 = 75$ degrees;
- $\theta_2 = 30$ degrees;
- $\mu_1 = 1515 \text{ cm}^{-1}$ (determined from Table 1); and
- $\mu_2 = 1698 \text{ cm}^{-1}$ (from Reference 3).

These parameters were used with Equation 2 to determine gold-plating thickness on sample parts. The reported thickness is the average thickness of the gold plating on the face of the sample part, assuming a density of 19.3 g/cm^3 .

Several sample parts, each having a different gold-plating thickness, were fabricated for the purpose of evaluating the similarity of the results obtained from the two different calibration techniques. Table 2 shows the thickness values from the two techniques to be virtually identical. Either technique therefore should be capable of determining the thickness of sample parts with similar precision.

Because of the thin plating and the complex geometry of the sample parts, fabricating a set of look-alike standards that could be measured dimensionally to the required accuracy was virtually impossible. To alleviate this problem, a set of flat plating-thickness standards consisting of various thicknesses of gold was fabricated, and the thickness of each standard was determined by the weight-gain method. (The estimated uncertainty in this determination of gold thickness does not exceed ± 5 percent.) Each standard then was measured with an X-ray spectrometer, and the output was converted to thickness by use of the mathematical model.

Table 3 indicates a maximum difference of 5.6 percent in the results obtained from the two independent methods for a gold-plating thickness of $2.2 \text{ }\mu\text{m}$. These results are very encouraging,

Table 1. Determination of Mass-Absorption Coefficient μ_1

Certified Gold Thickness (μm)	F_t (Counts)	$F_{t\infty}$ (Counts)	$\log_e \left(1 - \frac{F_t}{F_{t\infty}} \right)$	μ_1 , Calculated* (cm^{-1})
0.279	7694	60030	-0.1372	1468
0.483	12788	60030	-0.2396	1511
0.813	19267	60030	-0.3871	1318
1.372	28952	60030	-0.6583	1354
1.956	37691	60030	-0.9885	1601
2.210	41210	60030	-1.1600	1789
0.483	12234	59380	-0.2307	1334
0.813	18843	59380	-0.3817	1254
1.372	29022	59380	-0.6709	1443
1.956	37685	59380	-1.0068	1692
2.210	41312	59380	-1.1898	1920
				1516 Average

*Calculations were made from Equation 4:

$$\mu_1 = \sin \theta_1 \left[\frac{-\log_e \left(1 - \frac{F_t}{F_{t\infty}} \right)}{t} \right] - \frac{\mu_2}{\sin \theta_2} \quad (4)$$

since a precision of ± 20 percent would be acceptable for the determination of the gold thickness on a part. Enhancement corrections should be consulted when less than $0.5 \mu\text{m}$ of gold on rhodium is measured.

If the average thickness of the plating on the total face area of the part is an acceptable criterion for evaluating the plating thickness, the technique described should provide excellent results.

Table 2. Comparison of Gold-Thickness Measurements
Obtained Using Different Calibration
Techniques

Sample	Position	Measured Thickness, Using Calibration Technique Indicated	
		Standard Comparison (μm)	Mathematical Model (μm)
1	1	0.23	0.22
	2	0.24	0.22
2	1	0.06	0.05
	2	0.06	0.05
3	1	0.11	0.11
	2	0.11	0.10
4	1	0.11	0.10
	2	0.11	0.10
5	1	0.11	0.11
	2	0.12	0.11
6	1	0.18	0.17
	2	0.18	0.17

Evaluation of Mathematical Model for Calculating Thickness of Underplate

Equation 3 can be used with the X-ray spectrometer data to determine the thickness of the underplate. Except for the use of both rhodium and gold calibration standards, the collection of data and the calibration were performed as previously described. Using the present equipment, the following parameters were determined:

- $\theta_1 = 75$ degrees;
- $\theta_2 = 30$ degrees;
- $\mu_3 = 542 \text{ cm}^{-1}$ (determined from Table 4);
- $\mu_4 = 199 \text{ cm}^{-1}$ (from Reference 2); and
- $\mu_5/\mu = 0.988$.

Table 3. Comparison of Gold-Thickness Measurements Determined by Weight-Gain and Mathematical-Model Methods

Gold Thickness Determined by Method Indicated*			
Weight-Gain (μm)	Mathematical Model (μm)	Difference	
		μm	Percent
0.279	0.277	0.002	0.7
0.483	0.484	0.001	0.2
0.813	0.781	0.032	4.1
1.372	1.328	0.044	3.3
1.956	1.995	0.039	1.9
2.210	2.341	0.131	5.6

*Plating-thickness standards consisted of gold on nickel.

These parameters were used with Equation 3 to determine rhodium-plating thickness on sample parts. The thickness reported in Table 5 is the average thickness of the rhodium plating on the face of the sample part. The rhodium thickness also was determined by the standard-comparison method and the results are shown in Table 5.

A bias of approximately 14 percent was found between the two methods of measuring rhodium thickness. One explanation of this discrepancy could be an error in the value of μ_3 . The value used was experimentally determined from rhodium-on-nickel plating-thickness standards without a gold overplate. The gold overplate may possibly increase the effective mass-absorption coefficient of the rhodium layer.

If μ_3 is assigned a value of 672 cm^{-1} , the two methods show agreement, as indicated in Table 6; the maximum difference between the two techniques is 10 percent, and the average difference is 0.3 percent. If, by further investigation, μ_3 can be proven to equal 672 cm^{-1} , the mathematical-model technique should be useful for determining rhodium thickness on a production basis.

Table 4. Determination of Mass-Absorption Coefficient μ_3
Using Rhodium-On-Nickel Plating-Thickness Standards

Certified Rhodium Thickness (μm)	F_x (Counts)	$F_{x\infty}$ (Counts)	$\log_e \left(1 - \frac{F_x}{F_{x\infty}} \right)$	μ_3 , Calculated* (cm^{-1})
0.42	6703	167886	-0.0407	551
0.91	15192	167886	-0.0948	621
1.42	21368	167886	-0.1361	541
2.49	34202	167886	-0.2278	499
2.95	41313	167886	-0.2825	540
0.42	7279	180299	-0.0412	563
0.91	15980	180299	-0.0928	600
1.42	22454	180299	-0.1330	520
2.49	35126	180299	-0.2167	456
2.95	43909	180299	-0.2791	529
				542 Average

*Error may exist in μ_3 because the standards used did not have a gold overplate. Calculations were made from Equation 5:

$$\mu_3 = \sin \theta_1 \left[\frac{-\log_e \left(1 - \frac{F_x}{F_{x\infty}} \right)}{x} \right] - \frac{\mu_4}{\sin \theta_2} \quad (5)$$

Measurement of Thickness of Nickel on Kovar

A nondestructive nickel-thickness-measuring system was evaluated to determine its capability for measuring the thickness of nickel plating on contact-header assemblies.

Contact headers receive two layers of nickel plating. The first layer is deposited by the vendor to increase adherence during a subsequent brazing operation. After brazing, the part is sent to Bendix Kansas City where the annealed nickel plating receives a second layer of nickel. After the second plating, the thickness of the nickel is then measured.

Table 5. Comparison of Rhodium-Underplate-Thickness Measurements
Determined by Standard-Comparison and Mathematical-Model Methods

Sample	Gold Overplate Thickness (μm)	Rhodium Thickness Determined by Method Indicated		Difference	
		Standard Comparison (μm)	Mathematical Model* (μm)	μm	Percent
1	0.23	1.45	1.73	0.28	19
2	0.24	1.40	1.67	0.27	19
3	0.05	1.20	1.29	0.09	8
4	0.05	0.89	0.94	0.05	6
5	0.11	0.32	0.37	0.05	16
6	0.11	0.43	0.50	0.07	16
7	0.11	1.52	1.70	0.18	12
8	0.11	2.13	2.19	0.06	3
9	0.11	0.61	0.71	0.10	16
10	0.12	0.60	0.68	0.08	13
11	0.19	0.94	1.13	0.19	20
12	0.18	0.93	1.12	0.19	20
					14 Average
* $\mu_3 = 542 \text{ cm}^{-1}$; $\mu_4 = 199 \text{ cm}^{-1}$.					

Table 6. Comparison of Rhodium-Underplate-Thickness Measurements Determined by Standard-Comparison and Mathematical-Model Methods When $\mu_3 = 672 \text{ cm}^{-1}$

Sample	Gold Overplate Thickness (μm)	Rhodium Thickness Determined by Method Indicated		Difference	
		Standard Comparison (μm)	Mathematical Model* (μm)	μm	Percent
1	0.23	1.45	1.52	0.07	5
2	0.24	1.40	1.46	0.06	4
3	0.05	1.20	1.13	0.07	-6
4	0.05	0.89	0.82	0.07	-8
5	0.11	0.32	0.32	0.00	0
6	0.11	0.43	0.44	0.01	2
7	0.11	1.52	1.49	0.03	-2
8	0.11	2.13	1.92	0.21	-10
9	0.11	0.61	0.62	0.01	2
10	0.12	0.60	0.60	0.00	0
11	0.19	0.94	0.99	0.05	5
12	0.18	0.93	0.98	0.05	5
					0.3 Average

* $\mu_3 = 672 \text{ cm}^{-1}$; $\mu_4 = 199 \text{ cm}^{-1}$.

Each layer of the nickel plating exhibits differences in magnetic properties caused by variables in the conditions under which the part was plated, the type and composition of the plating, and the stress that is present. Manufacturing all of the standards that would be required to support the thickness measurement would be virtually impossible.

Evaluation of Microscope for Nickel-Thickness Determination

Five gold-on-nickel plating-thickness standards were sectioned and mounted. The nominal thicknesses shown in Table 7 were determined by beta-backscatter readings referenced to thickness standards supplied by the National Bureau of Standards (NBS). Five thickness measurements were made from each standard using a Leitz Metallux metallurgical microscope.

The data obtained from this evaluation are shown in Table 7. As determined from the data, and including resolution problems caused by diffraction, an uncertainty of $0.76\text{ }\mu\text{m}$ can be anticipated when this microscope is used to make plating-thickness measurements. The resolution limit can be calculated from the following equation:

$$d = \frac{\lambda}{NA_{\text{obj}} + NA_{\text{cond}}}; \quad (6)$$

where

d = the smallest resolvable separation between two object points;

λ = the wavelength of the light used for observation;

NA_{obj} = numerical aperture of the objective; and

NA_{cond} = numerical aperture of the condenser.

The resolution limit (d) was found to be $0.28\text{ }\mu\text{m}$ for the 100X objective, and $0.33\text{ }\mu\text{m}$ for the 50X objective. As problems of surface roughness become more apparent, the estimated uncertainty will increase.

ACCOMPLISHMENTS

A technique has been developed to determine the thickness of the gold and rhodium plating on A contact headers. Standards to support this measurement have been developed, and they are now in service. Calibration curves, required for determining the gold thickness, have been generated.

Table 7. Results of Evaluation of Microscope for Determining Nickel Thickness*

Magnification	Nominal Thickness** (μm)	Thickness Determined by Sectioning*** (μm)	Standard Deviation*** (μm)
100X	2.13	1.63	0.05
	1.40	1.32	0.05
	0.91	1.52	0.08
	0.56	0.61	0.08
	0.30	0.51	0.08
50X	2.13	1.63	0.05
	1.40	1.40	0.08
	0.91	1.04	0.13
	0.56	0.66	0.10
	0.30	0.74	0.10
	2.13****	1.93	0.08

*Leitz Metallux metallurgical microscope used.

**Beta-backscatter readings referenced to NBS thickness standards.

***Values shown are averages of five readings.

****Change in operator.

A mathematical model for the X-ray fluorescence spectrometer thickness measurement of a two-layer plating has been developed. The model is applicable to the plating-thickness measurement of an electronic assembly fabricated at this division and may prove to be useful for numerous other applications.

Equipment for measuring the thickness of nickel plating was evaluated, and the estimated uncertainty of the measurements was determined.

FUTURE WORK

Although the original plan was to utilize an X-ray fluorescence spectrometer in the production-inspection area for measuring

plating thickness, this project is now being terminated. The developed technology will not be implemented since the availability of low-cost sample parts now makes destructive testing economically acceptable and because the thickness of the gold plating on the part is no longer specified.

REFERENCES

- ¹R. J. Russell, *Multilayered Plating Thickness Measurement* (Topical Report). UNCLASSIFIED. Bendix Kansas City: BDX-613-1632, Revision, November, 1976. (Available from NTIS).
- ²*Noreleco Reporter*, 1960.

Appendix

DERIVATION OF EQUATION 3

The equation for determining the thickness of the underplate in a two-layer plating system was derived by G. R. Osbourn in September, 1976. The following material shows its derivation. (Refer to Figure 3 of this report.)

A beam of monochromatic X-rays having an intensity of I_0 is incident on the surface of the sample at an angle θ_1 . The radiation absorbed, dI , by a layer of thickness dt is proportional to the radiation, I , at dt and the thickness of the beam $dt/\sin \theta_1$. Therefore,

$$dI = \mu_1 I \left(\frac{dt}{\sin \theta_1} \right); \quad (A-1)$$

$$\frac{dI}{I} = - \left(\frac{\mu_1}{\sin \theta_1} \right) dt; \quad (A-2)$$

and

$$\log_e I \Big|_{I_0}^I = - \frac{\mu_1 t}{\sin \theta_1} \Big|_0^t. \quad (A-3)$$

After the beam has passed through a thickness t , its intensity is represented by the following equation:

$$I = I_0 e^{- \left(\frac{\mu_1}{\sin \theta_1} \right) t}; \quad (A-4)$$

where μ_1 is the mass-absorption coefficient.

For a two-layer plating, Equation A-5 represents the intensity of the X-ray beam after it has passed through the overplate having a thickness t :

$$I_1 = I_0 e^{- \left(\frac{\mu_1}{\sin \theta_1} \right) t}; \quad (A-5)$$

and Equation A-6 represents the intensity of the X-ray beam after it has passed through the underplate having a thickness x :

$$I_2 = I_1 e^{-\left(\frac{\mu_3}{\sin \theta_1}\right)x} \quad (A-6)$$

Recalling that the fluorescent radiation is proportional to the incident radiation, the intensity of the fluorescent radiation from the underplate is expressed by Equation A-7:

$$I_{2F_x} = kI_2 e^{-\left(\frac{\mu_4}{\sin \theta_2}\right)x}; \quad (A-7)$$

and the intensity of the fluorescent radiation from the underplate, as received by the detector, is expressed by Equation A-8:

$$I_{F_t} = I_{2F_x} e^{-\left(\frac{\mu_2}{\sin \theta_2}\right)t} \quad (A-8)$$

By combining these equations, an equation is derived for I_{F_t} as a function of I_0 :

$$I_2 = I_0 \left[e^{-\left(\frac{\mu_1}{\sin \theta_1}\right)t} \right] \left[e^{-\left(\frac{\mu_3}{\sin \theta_1}\right)x} \right]; \quad (A-9)$$

$$I_{2F_x} = kI_0 \left[e^{-\left(\frac{\mu_1}{\sin \theta_1}\right)t} \right] \left[e^{-\left(\frac{\mu_3}{\sin \theta_1}\right)x} \right] \left[e^{-\left(\frac{\mu_4}{\sin \theta_2}\right)x} \right]; \quad (A-10)$$

$$I_{F_t} = kI_0 \left[e^{-\left(\frac{\mu_1}{\sin \theta_1}\right)t} \right] \left[e^{-\left(\frac{\mu_3}{\sin \theta_1}\right)x} \right] \left[e^{-\left(\frac{\mu_4}{\sin \theta_2}\right)x} \right] \left[e^{-\left(\frac{\mu_2}{\sin \theta_2}\right)t} \right]; \quad (A-11)$$

and

$$I_{F_t} = kI_o e^{-\left[\left(\frac{\mu_1}{\sin \theta_1}\right)t + \left(\frac{\mu_3}{\sin \theta_1}\right)x + \left(\frac{\mu_4}{\sin \theta_2}\right)x + \left(\frac{\mu_2}{\sin \theta_2}\right)t\right]}. \quad (A-12)$$

The total fluorescent intensity that is received by the detector is the sum of dF 's, where dF is the fluorescent-radiation absorption by a thickness dT :

$$F_x = \int_0^t (I_{F_t}) dx; \quad (A-13)$$

$$F_x = \int_0^t kI_o \left[e^{-\left(\frac{\mu_1}{\sin \theta_1} + \frac{\mu_2}{\sin \theta_2}\right)t} \right] \left[e^{-\left(\frac{\mu_3}{\sin \theta_1} + \frac{\mu_4}{\sin \theta_2}\right)x} \right] dx; \quad (A-14)$$

and

$$F_x = \left[kI_o e^{-\left(\frac{\mu_1}{\sin \theta_1} + \frac{\mu_2}{\sin \theta_2}\right)t} \right] \int_0^t \left[e^{-\left(\frac{\mu_3}{\sin \theta_1} + \frac{\mu_4}{\sin \theta_2}\right)x} \right] dx. \quad (A-15)$$

Using the equation

$$\int e^{ax} = \frac{1}{a}(e^{ax}), \quad (A-16)$$

$$F_x = \left[\frac{kI_o e^{-\left(\frac{\mu_1}{\sin \theta_1} + \frac{\mu_2}{\sin \theta_2}\right)t}}{-\left(\frac{\mu_3}{\sin \theta_1} + \frac{\mu_4}{\sin \theta_2}\right)} \right] e^{-\left(\frac{\mu_3}{\sin \theta_1} + \frac{\mu_4}{\sin \theta_2}\right)x} \Bigg|_0^x, \quad (A-17)$$

$$F_x = \frac{kI_o e^{-\left(\frac{\mu_1}{\sin \theta_1} + \frac{\mu_2}{\sin \theta_2}\right)t}}{-\left(\frac{\mu_3}{\sin \theta_1} + \frac{\mu_4}{\sin \theta_2}\right)} \left[e^{-\left(\frac{\mu_3}{\sin \theta_1} + \frac{\mu_4}{\sin \theta_2}\right)x} - e^0 \right], \quad (A-18)$$

$$F_x = \frac{kI_0 e^{-\left(\frac{\mu_1}{\sin \theta_1} + \frac{\mu_2}{\sin \theta_2}\right)t}}{\frac{\mu_3}{\sin \theta_1} + \frac{\mu_4}{\sin \theta_2}} \left[1 - e^{-\left(\frac{\mu_3}{\sin \theta_1} + \frac{\mu_4}{\sin \theta_2}\right)x} \right], \quad (A-19)$$

and

$$e^{-\left(\frac{\mu_1}{\sin \theta_1} + \frac{\mu_2}{\sin \theta_2}\right)t} = e^{-\left(\frac{\mu_1}{\sin \theta_1} + \frac{\mu_1}{\sin \theta_2}\right)t} \quad (A-20)$$

Assuming that $\mu_1 = \mu_2 = \mu$, and using the form of Equation 13 in Reference 2:

$$\frac{F_t}{F_{t\infty}} = 1 - e^{-\left(\frac{\mu}{\sin \theta_1} + \frac{\mu}{\sin \theta_2}\right)t}; \quad (A-21)$$

$$e^{-\left(\frac{\mu}{\sin \theta_1} + \frac{\mu}{\sin \theta_2}\right)t} = 1 - \frac{F_t}{F_{t\infty}}; \quad (A-22)$$

$$e^{-\frac{\mu}{\mu_5} \left(\frac{\mu_5}{\sin \theta_1} + \frac{\mu_5}{\sin \theta_2}\right)t} = 1 - \frac{F_t}{F_{t\infty}}; \quad (A-23)$$

or

$$e^{-\left(\frac{\mu_5}{\sin \theta_1} + \frac{\mu_5}{\sin \theta_2}\right)t} = \left(1 - \frac{F_t}{F_{t\infty}}\right)^{\frac{\mu_5}{\mu}}; \quad (A-24)$$

where

μ_5 = the mass-absorption coefficient of the rhodium $K\alpha_1$ line in gold.

Substituting in Equation A-19:

$$F_x = \frac{kI_o}{\frac{\mu_3}{\sin \theta_1} + \frac{\mu_4}{\sin \theta_2}} \left(1 - \frac{F_t}{F_{t\infty}}\right)^{\frac{\mu_5}{\mu_1}} \left[1 - e^{-\left(\frac{\mu_3}{\sin \theta_1} + \frac{\mu_4}{\sin \theta_2}\right)x}\right]; \quad (A-25)$$

$$F_x = F_{x\infty} \left(1 - \frac{F_t}{F_{t\infty}}\right)^{\frac{\mu_5}{\mu_1}} \left[1 - e^{-\left(\frac{\mu_3}{\sin \theta_1} + \frac{\mu_4}{\sin \theta_2}\right)x}\right]; \quad (A-26)$$

and

$$\frac{F_x}{F_{x\infty} \left(1 - \frac{F_t}{F_{t\infty}}\right)^{\frac{\mu_5}{\mu_1}}} = 1 - e^{-\left(\frac{\mu_3}{\sin \theta_1} + \frac{\mu_4}{\sin \theta_2}\right)x} \quad (A-27)$$

Recalling that $\mu = \mu_1$, and taking the log_e of each side:

$$\log_e \left[\frac{1 - \frac{F_x}{F_{x\infty} \left(1 - \frac{F_t}{F_{t\infty}}\right)^{\frac{\mu_5}{\mu_1}}}}{1} \right] = -\left(\frac{\mu_3}{\sin \theta_1} + \frac{\mu_4}{\sin \theta_2}\right)x; \quad (A-28)$$

and

$$x = -\frac{1}{\frac{\mu_3}{\sin \theta_1} + \frac{\mu_4}{\sin \theta_2}} \log_e \left[\frac{1 - \frac{F_x}{F_{x\infty} \left(1 - \frac{F_t}{F_{t\infty}}\right)^{\frac{\mu_5}{\mu_1}}}}{1} \right]. \quad (3)$$

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