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ACCURACY OF WEIGHT METHOD USED
TO DETERMINE ALUMINUM FOIL THICKNESS

PDO 6984821, Topical Report

M. C. Lippert, Project Leader

Published August 1976

Prepared for the United States Energy
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**Kansas City
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Department 812

PDO 6984821
Topical Report

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ACCURACY OF WEIGHT METHOD USED TO DETERMINE ALUMINUM FOIL THICKNESS

BDX-613-1245, UNCLASSIFIED Topical Report, Published August 1976

Prepared by M. C. Lippert, D/812, under PDO 6984821

The thickness of 450-microinch ($11.4 \mu\text{m}$) thick aluminum foil, used to make precision circuit laminate, is commonly measured by weighing foil samples. Because the accuracy of the method was not precisely known, tests were conducted to determine its validity. The tests showed that while thickness variations obtained using this method could be in error by a maximum of ± 3 microinches (76.2 nm), the more expected error is ± 1 microinch (25.4 nm).

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SUMMARY

The performance of circuits produced by etching a pattern in thin aluminum foil bonded to a Mylar substrate depends on the uniformity of that aluminum foil. One method of determining this uniformity is to calculate the foil thickness from a specimen's weight and area and substitute it into the equation

$$\text{Thickness} = \frac{\text{Weight}}{\text{Area} \times \text{Density}}.$$

This technique has been used for several years to report foil thickness variations on nonplanar laminates, but the accuracy of the reported values had never been established. To determine this accuracy, the errors resulting from limits on the accuracy of the reported density value, the weighing technique, and the technique of determining specimen area were investigated. After these errors were determined, sample calculations were performed which showed how each of these expected errors affected the thickness value of the foil. Additional calculations showed that the worst case combinations of these errors produces a maximum variation of ± 3 microinches (76.2 nm) in the calculated thickness value, while substitution of these individual errors into the statistically derived equation for the variance of a function of several random variables reveals an expected error of ± 1 microinch (25.4 nm) in the calculated thickness value.

DISCUSSION

SCOPE AND PURPOSE

The performance of circuits produced by etching a pattern in thin aluminum foil bonded to a Mylar substrate depends on the uniformity of that thin aluminum foil. One method of determining this uniformity is to calculate the foil thickness from a specimen's weight and area and substitute it into the equation

$$\text{Thickness} = \frac{\text{Weight}}{\text{Area} \times \text{Density}}.$$

This technique has been used for several years to report foil thickness variations on nonplanar laminates, but the accuracy of the reported values has never been established. To determine this accuracy, the process used to clean the specimen, as well as the errors resulting from limits on the accuracy of the reported density value, the weighing technique, and the technique of determining specimen area were investigated.

PRIOR WORK

The method of determining foil thickness by weighing, and the errors inherent in the process, have been described in ASTM Standard E-252-67. Aluminum foil thicknesses from both formed and unformed laminate have been calculated from weight data for many years and used as evidence in several reports.

ACTIVITY

The material used in the fabrication of formed precision etched circuits is a Rexham Corporation laminate consisting of two layers of 0.005-inch-thick (0.127 mm) electrical grade Mylar bonded with a proprietary adhesive between two layers of 1145-0 aluminum rolled to nominal thicknesses of 0.004 inch (0.102 mm) and 450 microinches (11.4 μm). The performance of the circuit which is etched in the thin aluminum foil is a function of the thickness uniformity of this surface. To determine this uniformity, the areas to be measured are first designated by marking their centers with a felt pen and numbering each location so that the numbers remain on the part after the sample has been removed. After visually centering each mark, a matched die set having a nominal diameter of 0.75 inch (19.05 mm) is used to punch a slug from the laminate.

This slug is then placed in a correspondingly numbered jar containing Desolve*, a solution which attacks the adhesive and separates the layers of thin aluminum, thick aluminum, and Mylar without harming them. After about one day, the now-separated thin aluminum slug is removed from the Desolve, rinsed, dried, and then put in a correspondingly numbered container where it remains until being weighed. This weight value, the area value based on the 0.75 inch (19.05 mm) diameter, and the handbook density value for 1145 aluminum are then substituted into the equation

$$\text{Thickness} = \frac{\text{Weight}}{\text{Area} \times \text{Density}}$$

This method, which has been used for several years to obtain values to three significant figures, has given remarkably consistent results which agree closely with values obtained using another thickness technique based on electrical resistance measurements. However, when thickness variations in the 10-microinch (0.254 μm) range were suspected of causing circuit behavioral problems, it became imperative to determine the accuracy of the values obtained by the weight method.

To find this accuracy, each of the variables in the thickness equation--density, weight, and area--were investigated. After the expected error was determined for each parameter, sample calculations were performed which showed how this difference by itself affected the thickness value. Sample calculations were also performed to show the maximum amount the thickness value could be affected by a combination of the individual errors, and substitution of these errors into the statistical equation for the variance of a function of several random variables revealed the error expected in the calculated thickness value.

Density Value

The density value for 1145 aluminum used in the thickness calculation is 44.35 g/inch³ (27.06 x 10² kg/m³). According to ASTM Standard E-252-67, this value is accurate to ± 0.3 percent of the true density value. To evaluate how this error affects the thickness value, three sets of calculations were performed using a constant area based on the 0.75 inch (19.05 mm) punch diameter and the lowest, highest, and most commonly occurring weight values. For each of these weights, a change of 0.3 percent in the nominal density value produced a change of 1 microinch (25.4 nm) in the calculated thickness value (Table 1).

*Trademark name of Ram Chemicals, Gardena, California.

Table 1. Effect of 0.3 Percent Change in Density on Calculated Thickness Values.

Density	Thickness if Weight Equals 8.56 mg (microinches) (μm)	Thickness if Weight Equals 8.58 mg (microinches) (μm)	Thickness if Weight Equals 8.6 mg (microinches) (μm)
-0.3 percent error	436 (11.07)	437 (11.1)	438 (11.13)
Nominal Density	437 (11.1)	438 (11.13)	439 (11.15)
+0.3 percent error	438 (11.13)	439 (11.15)	440 (11.18)

Weight Value

Two balances have been used to weigh the punched samples involved in the various thickness studies performed at Bendix Kansas City. When results are required quickly, a Cahn electrobalance capable of weighing to the nearest 0.01 mg is used. However, if the time is available, arrangements are made to have the samples weighed on a Mettler microgram balance which is capable of weighing to the nearest 0.001 mg.

According to the calibration data for the Cahn electrobalance, the weight value is accurate to ± 2 divisions on the 10 milligram scale or ± 0.02 mg. On the other hand, the Mettler microgram balance is accurate to within ± 0.01 mg. Sample calculations performed by using the nominal density and area values show that when the foil thickness is expressed in three significant figures, a weight change of 0.02 mg produces a 1 microinch (25.4 nm) change in the thickness. Therefore, because most of the weight values are obtained from the Cahn electrobalance, each thickness value could be in error by ± 1 microinch (25.4 nm).

A balance capable of weighing to these reported accuracies must use samples which are as clean as possible. Although the aluminum sample is rinsed first in clean Desolve and then in acetone after being separated from the laminate, the possibility exists that some of the adhesive remains on the sample. A scanning electron microscope examination of several aluminum slugs revealed no remaining particles of undissolved adhesive, but it could not show if a film or dissolved adhesive was present.

One method of determining the existence of such a residue, scanning the part with a mass spectrometer, revealed no such impurities. Therefore, samples cleaned in the described manner are assumed free from additional weight because of remaining adhesive.

Area Value

The area of the punched slug was assumed to be that of a circle having a diameter equal to the nominal punch diameter, 0.75 inch (19.05 mm). Because the error involved in determining the area can be directly associated with the difference between the actual slug diameter and the assumed 0.75 inch (19.05 mm), efforts were made to measure the diameter of a number of slugs by examining those slugs with a Zeiss measuring microscope at 40X.

Such measurement is difficult. Under magnification, the edge of the slug did not appear to be a sharply smooth line but rather to be rough and jagged (Figure 1). These peaks and valleys made it extremely difficult to define the sample edge. In addition, the periphery of the punch, die, and an aluminum/Mylar laminate slug were traced with the stylus of a Taylor-Hobson talyrond.

Figure 1. Jagged Edges of Aluminum Foil Sample Which Make Area Calculations Difficult, 40X and 120X

These traces revealed that the samples were egg shaped rather than circular (Figure 2). The jaggedness of the slug edge was reflected in the tracings, but the radius on the stylus prohibited a true representation. Therefore, although the tracings provided a generalized representation of the slug's shape, no numerical definition of the diameter or peak-to-valley distance could be recorded.

SCALE: 5 MICROINCHES (127 nm)/DIVISION

Figure 2. Taylor-Hobson Talysond Traces of Die (Top Left), Punch (Top Right), and Punched Sample (Bottom). Die and Punch Nominal Diameter is 0.75 Inch (19.05 mm)

To account for this jaggedness and noncircular shape, 18 diameters on each of 5 slugs were measured with a Zeiss measuring microscope by flattening the samples between two glass slides and rotating them 10 degrees between readings. The range, mean, and standard deviations were calculated for each slug as shown in Table 2.

Diameter readings of a slug that is assumed to have a constant diameter can vary as much as 0.0072 inch (0.183 mm). In light of this data, it was assumed that a more accurate thickness value could be calculated by assuming that the slug diameter is better represented by the average of the 18 diameters. Based on this sample of 5 slugs, it seems that the 0.75 inch (19.05 mm) diameter value should be replaced by 0.7513 inch (19.08 mm). This substitution would decrease by 1 microinch (25.4 nm) those thickness values previously obtained using the 0.75 inch (19.05 mm) diameter, but it is not known how much the error between the calculated thickness and the absolute thickness would be improved.

Further analysis of this data revealed that when the diameters of each slug were constructed into histograms, the distribution of diameter measurements was remarkably similar (Figure 3). Because this similarity suggested a closer area relationship between slugs than between a slug and an absolute area value, the areas of a number of slugs were compared.

Table 2. Mean Range and Standard Deviation of 18 Diameter Measurements for Each of 5 Samples

Sample Number	Mean (inch) (mm)	Range (inch) (mm)	Standard Deviation (mil) (μ m)
1	0.7513 (19.083)	0.7485 to 0.7557 (19.01 to 19.19)	1.69 (42.9)
2	0.7517 (19.09)	0.7498 to 0.7545 (19.04 to 19.16)	1.16 (29.5)
3	0.7513 (19.083)	0.7480 to 0.7541 (18.99 to 19.15)	1.63 (41.4)
4	0.7514 (19.085)	0.7494 to 0.7528 (19.03 to 19.12)	0.9 (22.9)
5	0.7507 (19.06)	0.7490 to 0.7530 (19.02 to 19.13)	1.02 (25.9)
Average	0.7513 (19.093)

Figure 3. Distribution of Diameter Measurements

The areas of 25 discs were compared on an Image Analyzer which has the capability of projecting the measured object's image on a screen and directly indicating the amount of screen space occupied by that object. This method cannot produce an absolute area value but does compare areas to a standard value. The area of the first slug measured was defined as 1000, and the remaining slugs were compared to this sample. After examination of the 25 slugs, the greatest area difference was found to be 1.5 percent. Because this large difference was suspect, five of these slugs were reexamined on the image analyzer. This measurement did not reveal the same area differences between slugs as the first set of measurements. In addition, a comparison of standard deviations detected a significant difference between the separate

measurements. Because direct measurement of the slug area did not yield reliable results, the 18 diameter measurements performed on each of the five slugs were statistically examined with analysis of variance techniques. This examination revealed that while the distribution of diameters differed from one slug to the next, the slug areas did not vary significantly. In fact, the area standard deviation between samples was shown to be only $252 \times 10^{-6} \text{ in.}^2$ (0.163 mm^2). For a sample of five in a population normally distributed about a mean value, the standard deviation must be multiplied by 5.079 to determine the range within which one may be 95 percent confident that 95 percent of the readings will fall. Therefore, it is reasonable to expect that the actual areas will not vary by more than 1.28×10^{-3} square inches (0.826 mm^2) from the new nominal area value, which previous studies have shown to be the mean. If sample calculations are performed by varying the area value $640 \times 10^{-6} \text{ in.}^2$ (0.413 mm^2) from the value calculated by assuming a diameter of 0.7513 inch (19.08 mm), the change in thickness is shown to be 0.6 microinch (15.2 nm). It should be pointed out, however, that this variation between thicknesses is directly related to an uncertainty which is based on a sample of only five slugs. Because it is risky to base conclusions on such a small sample size, the standard deviation must be multiplied by a rather large number to obtain the 95 percent confidence limits. It is felt that examination of more slugs would reveal the same small standard deviation but that the multiplication factor would be reduced because of sample size. Therefore, the area uncertainty range would be reduced. It should also be pointed out that the disagreement between the calculated and actual thickness values depends on how accurately the area calculation, based on the 0.7513 inch (19.08 mm) diameter, approximates the actual area. Although it is known that this new value is more representative of the actual value, the degree of improvement is unknown.

Total Error

Analysis of the potential errors in the three parameters has shown that the variation in the density value is ± 0.3 percent, that the error in the weight value is $\pm 0.02 \text{ mg}$, and that the area difference between slugs will be no more than $640 \times 10^{-6} \text{ in.}^2$ (0.413 mm^2) from the nominal value. Although sample calculations have shown how a change in each parameter by itself affects the calculated thickness value, the total effect of these errors has not yet been shown. The maximum error in the thickness value is shown by sample calculations comparing the combination of nominal parameters to the worst case combinations. The expected error in the thickness value is shown by

substituting the parameter errors into the statistical equation for determining the variance of a function of several random variables (Appendix).

The nominal/worst case comparison was conducted by calculating the thickness for the lowest weight obtained in previous studies, the highest weight obtained, and the weight obtained most often. The nominal thickness was calculated by dividing the weight value indicated on the balance by the book value for density and the area of a 0.7513 inch diameter (19.08 mm) slug. The most this nominal value could be in error is determined by assuming that the minimum worst case exists when the slug actually weighs 0.02 mg less than the balance value, the density is actually 0.3 percent greater than the book value, and the area is actually $640 \times 10^{-6} \text{ in.}^2$ (0.413 mm^2) greater than the area for a 0.7513 inch diameter (19.08 mm) slug. The maximum worst case is the same error in magnitude but in the opposite direction for each parameter. The results of these calculations,, as shown in Table 3, indicate that the calculated thickness value should have a maximum error of ± 3 microinches (76.2 nm) for a given height.

Table 3. Nominal/Worst Case Comparison Showing Maximum Error in Thickness Calculation

Sample Weight	Nominal Case (μin) (μm)	Minimum Worst Case (μin) (μm)	Maximum Worst Case (μin) (μm)
8.1 mg	412.3 10.47	409.5 10.40	415.1 11.54
8.65 mg	440.3 11.18	437.4 11.11	443.2 11.26
9.1 mg	463.2 11.77	460.2 11.69	466.2 11.84

When slugs are punched from the same laminate sheet it is reasonable to assume that the density is the same for each slug. If this situation exists, the minimum worst case consists of the nominal weight minus 0.02 mg divided by the nominal density and the nominal area plus $640 \times 10^{-6} \text{ in.}^2$ (0.413 mm^2). In the maximum worst case, the density again remains at the nominal value but the weight is 0.02 mg greater than the nominal weight while the area is $640 \times 10^{-6} \text{ in.}^2$ (0.413 mm^2) less than the nominal weight. As shown in Table 4, the maximum error in thickness values for slugs taken from the same laminate sheet is only ± 1.6 microinches (40.6 nm).

Table 4. Nominal/Worst Case Comparison for Slugs Taken From the Same Laminate Sheet

Sample Weight	Nominal Case (μ in) (μ m)	Minimum Worst Case (μ in) (μ m)	Maximum Worst Case (μ in) (μ m)
8.1 mg	412.3 10.47	410.7 10.43	413.7 10.51
8.65 mg	440.3 11.18	438.7 11.14	441.9 11.22
9.1 mg	463.2 11.77	461.5 11.72	464.8 11.81

Since it is extremely unlikely that all three parameters will be in maximum error and in the worst case conditions, a more reasonable approximation of the accuracy of the calculated thickness value is obtained by substituting these parameter errors into the equation for determining the variance of a function of several random variables. When this relationship is derived for the thickness parameters, the following equation for the standard deviation expected in the thickness value results:

$$\sigma_{\Delta T}^2 = 1/(AD)^2 (\sigma_{WS}^2 - \sigma_{WP}^2) + (W^2/A^4 D^2) \sigma_{Ap} - (2W^2/A^3 D^3) \sigma_{Ap} \sigma_{Dp} + (W^2/A^2 D^4) \sigma_{Dp}^2$$

In this equation the "A" and "D" values are the average area and nominal density values while the "W" value is 8.65 mg which is the most commonly occurring weight measurement. The parameter errors previously determined are assumed to be equal to 3σ since they are the maximum error. Therefore, the density standard deviation (σ_{Dp}) equals $0.003/3$ and $\sigma_{WS}^2 - \sigma_{WP}^2$ which is the

variance of error in measuring weight $(0.02 \text{ mg}/3)^2$. The analysis of variance performed on the 18 diameters measured on each of five slugs revealed that the standard deviation between slugs was $252 \times 10^{-6} \text{ in.}^2$ ($0.163 \mu\text{m}^2$).

When these values are substituted into the equation, the expected error which is considered to be $2\sigma_{\Delta T}$ is ± 1.044 microinches (26.5 nm). If the density is considered constant, the expected error becomes ± 0.8 microinch (20.3 nm).

ACCOMPLISHMENTS

This accuracy investigation indicates that the error in the thickness values calculated from weight data for foil which is nominally 450 microinches ($11.4\ \mu\text{m}$) thick is expected to be ± 1 microinch ($25.4\ \text{nm}$) with a maximum error of ± 3 microinches ($76.2\ \text{nm}$). If the slugs are taken from the same laminate sheet and the density is considered constant, the expected error reduces to ± 0.8 microinch ($20.3\ \text{nm}$) while the maximum error becomes ± 1.6 microinch ($40.6\ \text{nm}$).

FUTURE WORK

Because the weight method has been shown to yield consistent results and because it consumes so little time, it will continue to be used in the evaluation of thin foil thickness. However, the results will no longer be considered absolute and will be used more as a means of process control. Efforts will be made to improve this process by developing an improved punching technique or an etching process used to better control the sample area. In addition, two different methods using electrical resistance and beta backscatter will be evaluated for their ability to yield absolute thickness values.

Appendix

EVALUATION OF MEASUREMENT ERROR OF CIRCULAR DISCS

The expression for thickness of the disc in question is $T = W/AD$, where T = Thickness, W = Weight, A = Area, and D = Density.

The measurement error for a particular disc is $\Delta T = T_s - T_p = (W_s/A_c D_c) - (W_p/A_p D_p)$, where subscript s refers to a measured value, subscript c refers to a constant value, subscript p refers to the true value.

Using standard statistical techniques a ± 2 sigma estimate of the error in measuring thickness is approximately $\pm .000001044$.

Assumptions include the following:

$$\bar{A}_p = A_c = y_4 \pi (.7513)^2 = .44332 \text{ inch}^2 \text{ (Based on previous study)}$$

$$\bar{D}_p = D_c = 44.35 \text{ grams/inch}^3 \text{ (Based on handbook value)}$$

$$W_p = .0086348 \text{ gram (Based on lot 5858)}$$

$$\sigma_{Dp}^2 = ((.0030/3) (44.35))^2 = (.4435 \text{ gram/inch}^3)^2 \text{ (based on the assumption that variability in true density from disc to disc is about the same as the variability that would be involved in measuring the density in one disc by a recommended ASTM method)}$$

$$(\sigma_{Ws}^2 - \sigma_{Wp}^2) = (.00002/3)^2 \text{ (Based on estimate that weight can be measured within } \pm .00002 \text{ grams)}$$

Correlation Coefficients

	W_m	W_p	A_p	D_p
W_m	1	0	0	0
W_p		1	0	0
A_p			1	-1
D_p				1

where W_m is the error in measuring weight ($W_s = W_m + W_p$).

The expression used for the variance of measurement error for thickness is:

$$\sigma_{\Delta T}^2 = 1/(AD)^2 (\sigma_{W_s}^2 - \sigma_{W_p}^2) + (W^2/A^4 D^2) \sigma_{A_p}^2 - (2W^2/A^3 D^3) \sigma_{A_p} \sigma_{D_p} + (W^2/A^2 D^4) \sigma_{D_p}^2$$

The expression is obtained by standard techniques as described in Bennet, C. A., and Franklin, N. L., Statistical Analysis in Chemistry and the Chemical Industry, pp. 51 - 54 (copy attached). That is, if $z = f(x_1, x_2, x_3, \dots, x_n)$.

$$\sigma_z^2 = \sum_{i=1}^n X_i^2 (\mu_{x_1}, \dots, x_n) \sigma_{x_i}^2 + 2 \sum_{i \neq j} f_{x_i}(x_1, \dots, x_n) f_{x_j} \sigma_{x_{ij}}$$

The partial derivatives are straight forward. The covariances, $\sigma_{x_{ij}}$, are as follows:

	Covariance			
	W_s	W_p	A_p	D_p
W_s	$\sigma_{W_s}^2$	$\sigma_{W_p}^2$	$\sigma_{W_p} \sigma_{A_p}$	$\sigma_{W_p} \sigma_{D_p}$
W_p		$\sigma_{W_p}^2$	$\sigma_{W_p} \sigma_{A_p}$	$\sigma_{W_p} \sigma_{D_p}$
A_p			$\sigma_{A_p}^2$	$\sigma_{A_p} \sigma_{D_p}$
D_p				$\sigma_{D_p}^2$

For example, to obtain the covariance of W_s and D_p , the correlation coefficient

$$\begin{aligned} \rho_{W_s D_p} &= (E(W_s - \mu_{W_s})(D_p - \mu_{D_p})) / \sigma_{W_s} \sigma_{D_p} \\ &= (E(W_s D_p) - \mu_{W_s} \mu_{D_p} - \mu_{W_s} \mu_{D_p} + \mu_{W_s} \mu_{D_p}) / \sigma_{W_s} \sigma_{D_p} \\ &= (E(W_m + W_p) D_p - \mu_{W_p} \mu_{D_p}) / \sigma_{W_s} \sigma_{D_p} \\ &= (E(W_m D_p) + E D_p) - \mu_{W_p} \mu_{D_p} / \sigma_{W_s} \sigma_{D_p} \end{aligned}$$

Now:

$$\begin{aligned} \rho_{W_p D_p} &= (E(W_p - \mu_{W_p})(D_p - \mu_{D_p})) / \sigma_{W_p} \sigma_{D_p} \\ &= (E(W_p D_p) - \mu_{W_p} \mu_{D_p}) / \sigma_{W_p} \sigma_{D_p} \end{aligned}$$

So that:

$$\rho_{W_s D_p} = (E(W_m D_p) - \sigma_{W_p} \sigma_{D_p}) / \sigma_{W_s} \sigma_{D_p}$$

Now:

$$\rho_{W_m D_p} = (E(W_m D_p) - \mu_{W_m} \mu_{D_p}) / \sigma_{W_m} \sigma_{D_p} = 0$$

$$\mu_{W_m} \mu_{D_p} = 0$$

And:

$$E(W_m D_p) = 0$$

Then:

$$\rho_{W_s D_p} = (\sigma_{W_p} \sigma_{D_p}) / (\sigma_{W_s} \sigma_{D_p}) = \sigma_{W_p} / \sigma_{W_s}$$

Covariance of W_s and $D_p = E(W_s - \mu_{W_s})(D_p - \mu_{D_p})$, and

$$\begin{aligned} \text{Covariance of } W_s D_p &= (\sigma_{W_p} / \sigma_{W_s}) (\sigma_{W_s} \sigma_{D_p}) \\ &= (\sigma_{W_p} \sigma_{D_p}) \end{aligned}$$

Alternate Assumptions:

The variance of the true density of the discs is the most difficult estimate to make, based on the information available. Accordingly, ± 2 sigma estimates of the error in measuring thickness are presented with three estimates of the variance of the density.

<u>D_p</u>	<u>$2\sigma \Delta T$</u>
.000	.000000842
.003	.000001044
.030	.000008320

Improved estimates of the parameters needed for evaluating the expression for $\sigma^2 \Delta T$ will yield corresponding improved estimates of the measurement error.

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