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A CRITICAL EVALUATION OF KOCOUR ELECTRONIC THICKNESS TESTERS - --ETC(U)
JAN 78 B L MOURANT, L H ESMORE

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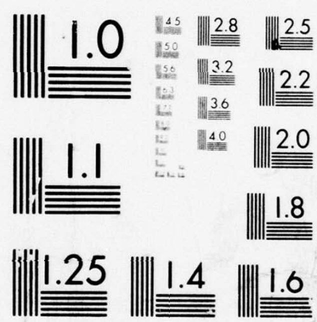

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A CRITICAL EVALUATION OF KOCOUR ELECTRONIC THICKNESS TESTERS - MODELS 955 AND S77

B.L. Mourant and L.H. Esmore

Approved for Public Release

JANUARY, 1978

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ABSTRACT

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A CRITICAL EVALUATION OF KOCOUR ELECTRONIC THICKNESS TESTERS - MODELS 955 AND S77

INTRODUCTION

The methods of measurement of the thickness of electroplated coatings may be classified into three groups :

1. Non-destructive
2. Destructive to both the coating and the component itself
3. Destructive to the coating only.

The methods in the first group are essential for many critical applications because every component in a batch may be examined without damage. These techniques include magnetic attraction, electromagnetic induction, eddy current, beta-ray back-scatter and X-ray fluorescence instruments. The accuracy of all of these may be affected by variations in coating purity or density and by underlayers of metals which differ from the coating being measured.

The second group includes sectioning of the components and, after suitable preparation, measurement of the coating thickness with a calibrated microscope. This method is widely adopted as the standard method in specifications but is limited to a minimum thickness of 2 μm and an absolute accuracy of ± 0.8 μm (1).

Methods in the third group employ dissolution of the coating either chemically or electrochemically without attacking the basis metal. The methods are commercially attractive because valuable items can be recovered by stripping and re-plating, the tests can be carried out in a works environment, and the accuracy is more than adequate for control purposes. Coulometric methods fall into this group and a number of suitable instruments have been designed and marketed. The Kocour* instruments were early in the

* Manufactured by the Kocour Co., 4800 S. St. Louis Ave., Chicago 32, Illinois, U.S.A.

field and have been widely employed throughout the industry. These instruments dissolve the coating electrochemically from a small area at constant current density, using electrolytes which do not attack the coating until current is applied. The area to be stripped is defined by a rubber gasket on a small Monel-metal cell. The d.c. current is controlled by a constant voltage source and precision series resistors. Time is recorded by a simple counting device driven by a synchronous clock and, by selecting the d.c. currents for particular coatings, the readings can be made directly in thickness units (2s = 0.00001 in). When the coating is perforated, the resultant rapid change in cell voltage triggers a relay system which terminates the test.

The Model 955 instrument is an early unit employing thermionic valves whereas the latest Model S77 (modified) uses solid state devices. The cells and gaskets for both models are the same and are used with accessory units as follows :

(a) Type "A" cells for normal operation.

(b) Accessory units "B" and "CB" with Type "B" gaskets and cells for smaller areas.

(c) Accessory units "MT" and "CM" with Type "A" cells and gaskets for coatings thinner than about 0.0001 in (2.5 μm).

The following examination of the two instruments was aimed at assessing the factors affecting the reproducibility and accuracy of the thickness measurements. These are :

1. The stability of the d.c. supplies and cell currents.
2. The consistency of dimensions of cells and gaskets.
3. The consistency of the areas stripped.
4. The reliability of the electroplated standard discs supplied by the manufacturer for calibration checks.

The Model 955 instrument is designed for 110V power supplies and was supplied with a 230V/110V stepdown transformer. The Model S77 instrument has optional transformer taps for either 110 or 220V operation, and was set to the latter.

The composition of the electrolytes supplied by the manufacturer are not disclosed but are similar to some described in specifications.

EXPERIMENTAL

Constant Current Supply

The voltage sources of both instruments are stabilised. These were supplied from a "Stabilac" Model SP2500 unit through a "Variac" which allowed variation of the supply within the range of 200-240 V. In each instance, the stabilised d.c. voltage from each instrument was measured with a high-quality digital voltmeter. The instruments were first balanced and calibrated in accordance with the manufacturer's instructions at 220 V mains setting and the supply then varied in steps of 10 V.

The instruments have provision for varying the calibration by ± 10% from the normal settings and the accuracy of this offset was similarly examined holding the supply constant at 220 V.

The cell currents for each setting of the coating selector switch including those on the accessory units Type "B", "CB" and "CM" were also measured again with a precision digital meter. (An "MT" unit was not available for the Model 955 instrument).

Uniformity of Cells and Gaskets

The important cell dimension is the external diameter over which the gasket is fitted. If this is too small, electrolyte leakage could occur and, if too large, the gaskets could be unduly stretched. These dimensions on two type "A" and type "B" cells were measured at two positions at right angles.

Two dimensions on a number of new gaskets, shown in Fig. 1 as "P" and "Q", were measured by means of a travelling microscope on two diameters at right angles for both type "A" and type "B" gaskets.

In use, the gaskets are slightly stretched when fitted to the cell and they are compressed under normal cell loading. The area stripped is affected by these factors and, in addition, the gaskets may be distorted by relative movements of the sample and the cell during set-up of the tests.

The combined effects of these factors in actual measurements of coating thickness were examined by stripping the coating from tin plate. The coating on this was thin enough to allow numerous tests to be conducted rapidly and the contrast between the tin and the stripped steel aided in the precise measurement of the spots with a travelling microscope. The reproducibility of spot sizes, using a single "A" gasket on a particular cell, was determined to check the effect of re-positioning the cell ten times. For each spot, two diameters were measured at right angles to check possible ovality of the spots which may arise due to the off-centre cell mounting. These measurements were repeated with a "B" gasket and cell.

The consistency of eleven gaskets used with two cells was similarly determined for a single spot with each combination to check the variation in cells and gaskets as supplied. These measurements were also repeated with "B" gaskets.

Kocour Thickness Standards

The manufacturer supplies circular discs of various basis metals which have been electroplated with the coatings of copper, nickel, zinc, etc. It should be noted that these standards have been specifically prepared for coulometric measurement and should not be used for magnetic and other instruments because undercoats are sometimes plated under the standard coating.

These discs are plated in cells designed to achieve uniformity of thickness and with accurately controlled current and time to obtain specified thicknesses. The coatings are specified to be within ± 5% of a marked thickness, and the reliability of this claim is of paramount importance if they are used to calibrate the instruments, as recommended by the manufacturer. It is also of some importance to establish whether the calibration of the instrument should be adjusted so that it reads the value marked on the standard or whether the instrument should be set up with zero correction and checked against the standard to ensure that the readings are within 5% of the labelled figure.

The thicknesses of the coatings on five standards were determined by cutting a sector from each disc, overplating this with copper, mounting, polishing and etching to permit measurement with a calibrated metallurgical microscope.

RESULTS AND DISCUSSION

Constant Current Supply

The effects of varying the a.c. mains supply ± 20 V from the nominal 220 V are negligible, the stabilised open-circuit voltage across the cell being 296 ± 1 V for the Model 955 instrument and 85.9 ± 0.3 V for the Model S77. When the calibration controls were set 10% high or low, the d.c. voltage agreed with the calculated figures for the Model 955 instrument, and were better than ± 1% for the S77.

The actual cell currents at each setting of the coating selector switches for each instrument and accessory unit are recorded in Table 1.

Cells and Gaskets

In the unstressed condition, the dimension "P" for twelve "A" gaskets averaged 8.26 ± 0.07 mm compared a mean cell neck dimension of 8.35 mm. For eleven "B" gaskets, dimension "P" averaged 6.31 ± 0.04 mm compared with the cell dimension of 6.45 mm. No electrolyte leakage occurred when the largest gaskets were tested with any cell.

The effects of cell positioning and pressures on a single "A" gasket on one cell applied ten times to the tin-plate surface gave spot diameters shown in Table 2. Calculations of the areas of the spots as circles (using mean diameters) or as ellipses show that this effect is not significant. The spot diameters for eleven gaskets each fitted to two different cells with two spots produced with each combination are shown in Table 3. Similar measurements were made on a set of "B" gaskets. For ten applications of a single gasket on one cell, the mean spot diameter was 2.49 mm (s.d. 0.07 mm) and for eleven gaskets on two cells with each combination tested twice, the means and standard deviations were 2.50 mm (s.d. 0.10 mm) and 2.51 mm (s.d. 0.09 mm).

A calculation of the theoretical area of a spot, based on a zinc coating, shows that the diameter of the spot with an "A" cell should be 3.45 mm and for a "B" cell, 2.44 mm. The calculations are in the Appendix. In the two tables, the largest spot was 3.66 mm, which corresponds to an area 11.3% greater than the calculated figure, and the smallest spot was 3.33 mm, which is 6.7% below the theoretical value. The mean of all 44 readings in Table 3 is 3.45 mm, which coincides with the calculated value.

Thickness Standard Discs

The microscope measurements of the thicknesses of the coatings on five discs are recorded in Table 4, together with mean values and a calculation of errors. Accepting the accuracy of the microscope to be ± 0.8 μm for a single reading, it is important to note that some readings show greater errors than the claimed figure of ± 5%, even when the mean is within the limit. It is therefore, not recommended that the instrument be re-calibrated if a test on a disc shows an error in respect to the marked value. If the reading is within 5% of the value, the instrument should be taken to be correctly calibrated. If the reading is not within the 5% value, the gasket, which may be damaged, should be replaced and a further test conducted.

A final observation is that the standard coatings may be damaged by cleaning with a pencil eraser as recommended by the manufacturer. It is preferable to remove passive films with a small swab of cotton wool mounted on a match stick and charged with freshly prepared paste of magnesia. The swab should be applied lightly through the gasket after the cell has been positioned. It is thus only applied to the test area thereby avoiding damage to adjacent areas of the standard coating.

CONCLUSIONS

Both Kocour instruments have been shown to be reliable for the coulometric determination of thickness of electroplated coatings. An accuracy of better than ± 10% should be achieved provided that the rubber cell gaskets and electrolytes are in good condition. Some recommendations are made in respect to the standard-thickness discs and their use.

REFERENCE

1. Australian Standard, ASK 173-1971, para. 2.2.1.1.

TABLE 1

TYPICAL CELL CURRENTS

Coating Setting	Model S77, S/N 5085			Model 955, S/N 1626	
	Normal mA	Accessory "CB", mA	Accessory "CM", mA	Normal mA	Accessory "B", mA
Cr ÷ 2 = 10^{-5}	46.8	23.1	-	45.4	22.95
Ni	35.5	17.65	3.49	35.0	17.60
Cu	32.7	16.25	3.23	32.0	16.17
Brass	30.5	15.15	3.01	29.7	14.95
Zn	25.32	12.74	2.52	24.9	12.44
Cd	17.77	8.89	1.77	17.45	8.74
Sn	14.18	7.08	1.407	13.87	6.92
Ag	11.35	5.67	1.127	11.12	5.55
Cr x 10^{-6}	9.29	4.63	0.927	9.07	4.55
Au	2.23	1.14	-	-	-

TABLE 2

REPRODUCIBILITY OF STRIPPED AREA - TYPE "A" CELL

(One gasket/One cell)

Spot No.	Diameters of Spots, mm		Areas of Spots, mm^2	
	a	b	$\frac{\pi}{4}\left(\frac{a+b}{2}\right)^2$	$\frac{\pi}{4}$ (a x b)
1	3.62	3.57	10.15	10.15
2	3.64	3.60	10.29	10.29
3	3.56	3.60	10.07	10.07
4	3.62	3.60	10.24	10.24
5	3.54	3.64	10.12	10.12
6	3.62	3.53	10.04	10.04
7	3.66	3.56	10.24	10.23
8	3.66	3.57	10.26	10.26
9	3.49	3.55	9.73	9.73
10	3.51	3.62	9.98	9.98
Mean, 20 readings	3.588		10.11	10.11
Standard Deviation	0.049		(Circle)	(Ellipse)

TABLE 3

REPRODUCIBILITY OF STRIPPED AREA - TYPE "A" CELLS

(11 gaskets/Two cells)

Gasket No.	Diameters of Spots, mm			
	Cell No. 1		Cell No. 2	
1	3.58	3.52	3.46	3.38
2	3.48	3.41	3.44	3.40
3	3.47	3.50	3.32	3.33
4	3.54	3.53	3.43	3.44
5	3.55	3.51	3.41	3.47
6	3.47	3.49	3.48	3.48
7	3.37	3.33	3.34	3.35
8	3.45	3.53	3.52	3.49
9	3.52	3.46	3.71	3.48
10	3.46	3.46	3.36	3.39
11	3.55	3.43	3.37	3.38
Mean, 22 spots = 3.48 mm Standard Deviation = 0.061 mm			Mean, 22 spots = 3.43 mm Standard Deviation = 0.085 mm	

TABLE 4

STANDARD THICKNESS DISCS

Coating	Zinc	Zinc	Cadmium	Nickel	Copper
Basis Metal	Steel	Steel	Brass	Steel	Steel
Marked Thickness, μm	11.2	11.7	12.2	12.5	12.7
Microscope Thickness, μm	11.8	12.1	10.7	12.2	12.8
	11.8	12.2	11.0	13.6	13.3
	11.8	13.0	10.7	13.0	12.8
	11.5	12.5	11.9	13.4	11.9
	10.5	12.1	11.3	12.2	11.9
	11.6	12.4	10.7	12.4	13.9
	10.4	12.7	12.2	13.0	12.8
	10.4	11.9	12.2	13.1	12.5
	10.7	12.7	12.4	12.2	12.5
	10.8	12.1	12.8	13.0	12.5
Mean Thickness, μm	11.1	12.4	11.6	12.8	12.7
Standard Deviation, μm	0.6	0.35	0.8	0.5	0.6
% Error, Mean v Nominal	-0.9	+6.0	+4.9	+3.2	0
% Error, Maximum v Nominal	+5.4	+11.1	+4.9	+8.8	+9.4
% Error, Minimum v Nominal	-7.1	-1.7	-12.2	-2.4	-6.3

APPENDIX

CALCULATION OF SPOT DIAMETER FOR "A" CELL

If we take the following constants,

Atomic Weight of Zinc	=	65.37
One Faraday	=	96,500 coulombs
Density of Zinc	=	7.14 grams/cm^3
Anodic Current Effic.	=	100%

and apply these to the following instrument and coating characteristics,

Zinc Coating Thickness	=	0.00100 inch = 25.40 micrometre
Deplating Current	=	25 mA (Model S77 25.32 mA, Model 955 24.9 mA)
Stripping Time	=	200 s

the following calculations may be made

200 s x 25 mA = 5000 millicoulombs

$$\text{Mass of Zinc} = \frac{\text{millicoulombs}}{F} \times \frac{\text{A.W.Zn}}{z}$$

$$= \frac{5000}{96{,}500} \times \frac{65.37}{2} \text{ milligram Zn}$$

$$= 1.694 \text{ mg Zn}$$

$$\text{Volume of Zinc} = \frac{\text{Mass}}{\text{Density}} = \frac{1.694 \times 10^{-3}}{7.14}$$

$$= 0.2373 \times 10^{-3} \text{ cm}^3$$

$$\text{Equivalent Area for 25.4 } \mu\text{m thickness} = \frac{0.2373 \times 10^{-3}}{25.4 \times 10^{-6}} = 9.3425 \text{ mm}^2$$

$$\text{Diameter of Spot} = \sqrt{\frac{9.3425}{3.1416} \times 4} = 3.449 \text{ mm}$$

This figure should be compared with the diameters measured and shown in Tables 2 and 3.

For the Type "B" gasket, the area of the spot should be half that for the Type "A".

Area = 4.6712 mm^2 Diameter of Spot = 2.439 mm

This figure should be compared with the diameters quoted on Page 4.

Taking the average "A" gasket spot size to be 3.45 mm, the current required for each metal coating can similarly be calculated. A comparison between these values and the measured values for each instrument is as follows :

Coating	Valency Change	Density (g/cm^3)	Calculated Current, mA	Measured Currents, mA		Difference, %	
				955	S77	955	S77
Cr	3	7.14	47.1	45.4	46.8	-3.6	-0.6
Ag	1	10.50	11.15	11.12	11.35	-0.3	+1.8
Sn	2	7.29	14.07	13.87	14.18	-0.1	+0.8
Cd	2	8.64	17.60	17.45	17.77	-0.9	+1.0
Zn	2	7.14	25.00	24.9	25.32	-0.4	+1.3
Cu	2	8.93	32.17	32.0	32.7	-0.2	+1.7
Ni	2	8.90	34.72	35.0	35.5	+0.8	+2.2

It should be realised that the density figures for electrodeposits may vary from the values for wrought metals used in the above calculations.

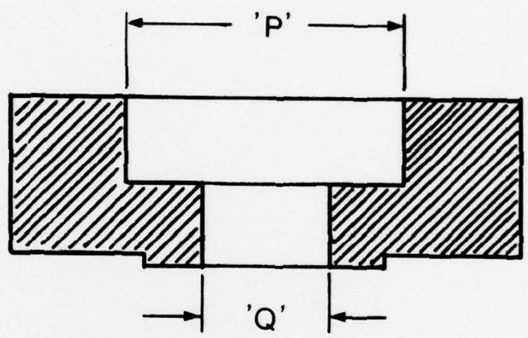

Fig 1 TYPE 'A' GASKET

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