Measuring Electrodeposit Thickness

A BETA BACK-SCATTER METHOD FOR THE PLATINUM METALS

In the electrodeposition of the platinum group metals it is particularly important that electrodeposit thicknesses are accurately known. Excessively thick electrodeposits are costly; insufficiently thick deposits may lead to breakdown in service. The average thickness of electrodeposits used industrially is 0.0002 inch, while in decorative applications the average is much lower. Small errors at these thicknesses often amount to a relatively large proportion of the total thickness, and there is thus a need for a process control technique capable of checking electrodeposit thicknesses rapidly and non-destructively.

The conventional methods of measuring electrodeposit thicknesses by microsectioning or by "strip and weigh" methods are inaccurate on thin deposits and are only accurate to about 20 per cent on thick deposits, while they are also time-consuming.

To find a more accurate measuring method that could be quickly used without damaging the electrodeposit a project was initiated in the Johnson Matthey Analytical Laboratories which led to the development of a prototype beta back-scatter thickness gauge (1). Further development work carried out in conjunction with Panax Equipment Limited has now resulted in a commercial apparatus, known as the Beta 750 Thickness Gauge and available to industry from Johnson Matthey. One of these units has been in use for over two years and has already paid for itself in the metal economies it has effected. This instrument is based on the principle that beta particles, when they pass through matter, collide with the electron shells of the atoms they encounter and after many collisions some emerge from the surface they entered. The beta particles that emerge are described as back-scattered. The intensity of the beta back-scatter from a material is proportional to its atomic number or aggregate atomic number. The amount of back-scattered radiation can be used as a means of determining the thickness of one electrodeposited layer on a different basis metal. A variation in coating thickness will give a change in overall atomic number, and in back-scatter intensity below a limiting value that represents the maximum thickness of deposit which can be measured with a particular isotope.

Fig. 1 For the accurate and non-destructive measurement of plating thicknesses of the platinum group metals the Beta 750 provides a simple and compact instrument, using the principle of "back-scatter" of beta particles

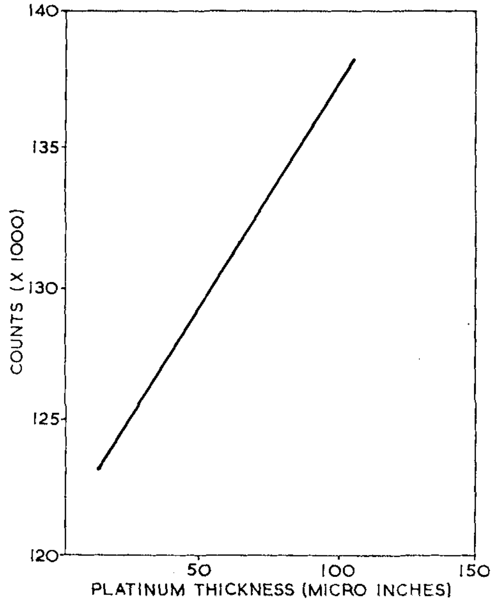

Fig. 2 A typical calibration graph for use with the Beta 750. This shows the thickness of platinum plating on titanium obtained from measurement of standard specimens. (Aperture 0.06 inch × 0.2 inch; counting time 60 seconds)

The Beta 750 comprises a krypton 85 source, which emits only beta particles, contained in a metal cylinder which is mounted above a radiation detector. The detector is connected to an electronic counter. In use the sample to be measured is positioned above the source. The beta particles back-scattered from the sample are collected by the detector and a count made over a certain period is referred to the calibration graph for the particular electrodeposit and the thickness read off. The points on the calibration graph are plotted by recording the count obtained on the instrument, using the same sample holder, from a number of standards. The standards have known thicknesses of the electrodeposit on the same basis metal.

There are several factors to take into account in using this method of measurement. The electrodeposit must differ by at least 3 in atomic number from the basis material. The energy of the beta radiation is sufficient to measure normal plating thicknesses. The beta source has a half life of 10.6 years, which allows stable calibration graphs to be drawn for a large number of metals and non-metals. The geometry of the source is such that it can be used to measure thicknesses over very small areas, down to 1 mm in diameter. This allows the instrument to be used for measuring thicknesses on curved surfaces with little loss of accuracy.

A typical calibration graph, shown in Fig. 2, gives the curve for platinum on titanium, while Fig. 3 shows the curve for palladium on copper.

There is an optical arrangement allowing the exact point being measured to be seen even though the board is face down to the beta source. The thickness of rhodium electrodeposits on nickel, often used as in electronic

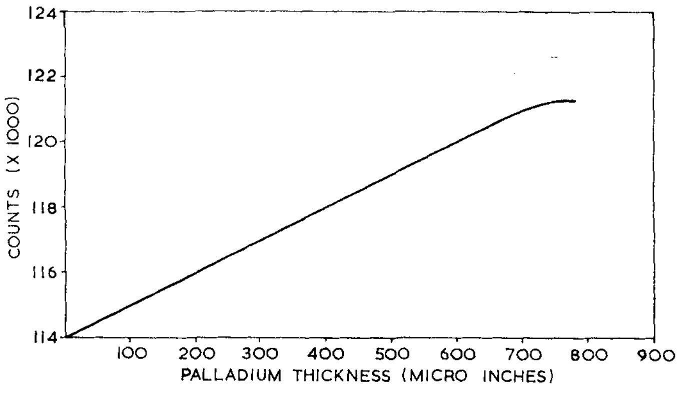

Fig. 3 A calibration graph for palladium plating on copper (Aperture 0.06 inch diameter; counting time 60 seconds)

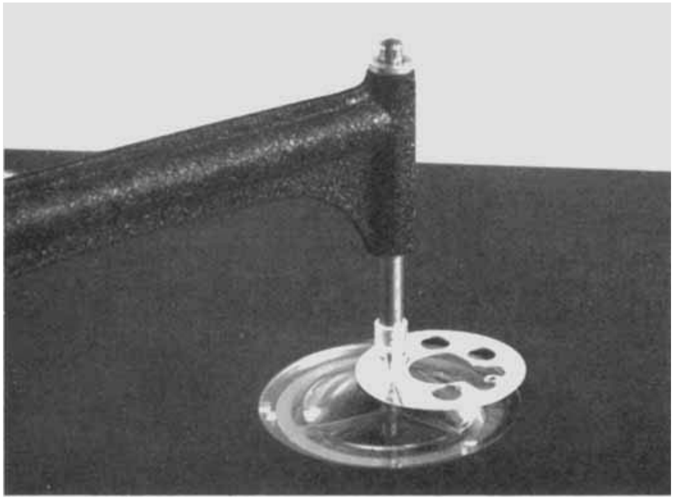

Fig. 4 A close-up view of the measuring head being used to determine the thickness of a rhodium electrodeposit on nickel

contact applications, can be measured, but because the atomic numbers of rhodium and silver only differ by one the unit cannot distinguish between them.

A fuller account of the development of this equipment and of its applications not only to the platinum group metals but also to other metals will shortly be published elsewhere (2).

References

1 B. S. Cooper and W. Westwood, *B.P. Appln.* 5942/65

2 S. P. G. Melrose and B. S. Cooper, *Trans. Inst. Metal Finishing*, in the press

Osmium-coated Tungsten Cathodes

Thermionic valves, and a number of related devices such as klystrons and magnetrons, depend for their operation on the control of a stream of electrons generated by a thermionic emitter. A common form of emitter consists of a porous tungsten substrate in association with a reservoir of barium calcium aluminate which, by mutual reaction, provides a supply of barium to the emissive surface.

The total work function of the assembly depends on the work functions of both the substrate and the adsorbate and on the degree of surface adsorption and, as it is considered that no better adsorbate than barium exists, attention has been directed in recent years towards alternative substrates.

Recent theoretical work led to the prediction that, paradoxically, a higher work function of the substrate could lead to a reduction in the total work function of the whole system and, in a recent paper by P. Zalm and A. J. A. van Stratum, of the Philips Research Laboratories (*Philips Tech. Rev.*, 1966, **27**, (3/4), 69–75), results are given for systems in which rhenium, ruthenium, iridium and osmium were investigated, the metals being applied as thin coatings to the tungsten substrates. It was confirmed that the work function of the emitter decreased as the work function of the substrate coating increased and more detailed investigation of the most promising assembly containing osmium revealed that the reduction in work function was so marked that, for example, at 800°C a current density ten times that of a normal cathode was possible.

It is considered that the use of osmium cathodes with their higher performance will be of considerable practical advantage in magnetrons, reflex klystrons and disc-seal triodes and further, as a consequence of the lower operating temperature for a given performance, these cathodes may find application where the expense of initial purchase and replacement is an important consideration.

During this investigation the work functions of iridium and osmium have been determined as 5.50 ± 0.005 V and 5.93 ± 0.05 V respectively, and although the work function of ruthenium was not determined it is known to be in excess of that of tungsten at 4.54 V.

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