Measuring Plating Thickness Using XRF

Instrumentation and proper procedures for using thickness testing systems...

By FRANCIS REILLY
Manager
Veeco/UPA Technology
Plainview, New York

The basic design of x-ray fluorescence (XRF) plating thickness and composition measurement instruments incorporates four features: 1. A source of high-intensity x-rays; 2. A mechanism to define the x-ray beam size; 3. An x-ray detection and pulse processing system; and 4. Sample positioning optics and staging.

The XRF systems used for plating measurement use x-ray tubes as the primary x-ray source. The output of an x-ray tube is known as bremsstrahlung[1] and consists of a continuum of x-ray energies.

The x-ray beam size and geometry are defined by a "collimator," which is essentially a pin-hole aperture. The size of the pin-hole, the collimator-to-work distance and the focal-spot size of the x-ray tube determine the beam size at the work surface.

This beam of x-rays induces characteristic x-ray emissions in the sample, which is positioned on a stage below the x-ray tube and collimator. It is these fluoresced x-rays that are collected to build an x-ray spectrum of the sample, the most important part of any XRF measurement. Fig. 1.

During x-ray detection, a gas-filled proportional counter produces an output charge pulse proportional to the energy of the x-ray entering through a beryllium window. The processing of these pulses requires amplifying and shaping the pulse, determining the pulse height and finally building a PHA (pulse height analysis) or spectrum[2] from the collected pulses. Fig. 2.

Rather than the tungsten (W) "target" found in most x-ray tubes, a tube incorporating a molybdenum (Mo) target is used. This provides for greater precision. In addition to

bremsstrahlung, the output of the Mo-target x-ray tube includes the intense characteristic Kalpha and Kbeta Mo emission lines. Simply put, the energy of the Mo K emissions is above the absorption edge[3] of gold. The intense Mo x-rays combine with the bremsstrahlung to produce extremely intense gold fluorescence (i.e., high countrate). As a further refinement, the glass envelope comprising the x-ray tube incorporates a beryllium (Be) "window" that allows more of the x-rays, particularly the low-energy x-rays, to reach the sample. Therefore, countrates from copper and nickel are dramatically improved as well.

Calibration. To measure thickness and/or composition for a given application, it is necessary that the XRF system be calibrated using known references or standards. In calibrating for a specific application, such as gold over nickel, an appropriate calibration mode is first selected[4]. A "region of interest" (ROI) is defined that encompasses the channels in the x-ray spectrum in which the characteristic x-rays from only that coating (i.e., gold) occur. The countrate within this ROI will increase as the thickness of the layer increases, up to the point of saturation of infinite thickness.[5] By taking countrate data on reliable thickness standards and fitting the data to a calibration curve, the constants defining the thickness curve are defined. These constants are stored in the computer's memory for repeated use.

The specific thickness standards used for the calibration are not arbitrary. These values are selected to fall on the curve at preselected regions that will define the curve most accurately for the thickness ranges most commonly measured. For example, 30 to 50 microinches is the value for gold over nickel. Additionally, infinite samples of the coating and substrate are used during calibration to set x-ray tube intensity and to define relative counts or "normalized counts" in which the ROI countrate from a sample will be scaled to fall between zero (the relative countrate from the base) and one.

Thickness standards are selected whose values will fall at certain predefined normalized counts, typically 0.20, 0.40, 0.60, and 0.80 for single layer gold. Foil thickness standards are better than plated thickness standards, since they deliver improved accuracy and versatility.

Selecting beam size. The user must select the beam size to use for the calibration. This decision is primarily based upon the size and geometry of the part. For flat surfaces, use a beam size that is at least two mils smaller than the width of the area. For example, if measuring a contact that is 15 mils wide, use a 12 mil beam size.

The size of the collimator pinhole does not directly indicate the beam size. The beam size at the work surface is determined by three factors: 1. X-ray tube focal-spot diameter; 2. Distance between the collimator and the sample; and 3. Size of the collimator. Some manufacturers have adopted the use of beam size in place

of collimator size to make calibration easier for operators.

When measuring cylindrical parts or other curved samples, the rule of thumb is to select a beam size that is no larger than one-third the diameter of the part.

Selecting calibration measurement time. Measuring time is a critical factor during calibration, since data for each standard form the basis for the calibration as well as for measurement on unknown finishes. As a rule of thumb, the user should always use a measuring time for calibration that is greater than the measuring time he will use for samples. Thirty sec are recommended. This is almost always more than satisfactory for applications, since increasing the time further will not result in significantly improved calibrations.

Standard positioning is also important. Well-prepared XRF standards have their centers marked with a circle. The standard should be positioned manually, or with a motorized stage driven by a joystick or point-and-shoot mouse, so that the reticle on the TV camera viewing window is centered within the circle. This ensures that the surrounding support material does not cause any "shadowing" and that the intended measuring area of the standard is used.

The collimator-to-work distance on all XRF systems is adjusted for each sample by visually focusing or using an auto-focusing laser on the measuring area.

After calibrating, a reading should be taken on one or more of the standards to ensure proper tracking. A measuring time sufficient to obtain better than plus or minus five pct uncertainty should be used also. The readings should come within five pct of the labeled values. If not, the calibration should be repeated.

Reference measurement. During calibration, all modern XRF systems adjust their pulse processing electronics to ensure that certain channels in the spectrum correspond to the correct x-ray energies. Readings on the reference standard are used to make necessary adjustments[6]. In addition, the countrate on the reference standard is stored along with the calibration. Later, this data will be compared with new readings on the same reference standard and any differences in the countrates will be corrected, adjusting for any drift.

Sample positioning. Positioning samples properly in the x-ray chamber is fundamental to measurement accuracy. The first thing that must be ensured is proper collimator/optics alignment. If the optics are out of alignment, the actual measurement area may be quite different from what is indicated by the reticle.

With the collimator/optics in alignment, care must be taken to position samples properly. First, the proper beam size should be used. Under no circumstances should the beam be larger than the width of the part. If a cylindrical part is to be measured, the beam size should be no larger than one-third the diameter of the part.

When measuring concave surfaces

or parts with raised edges, it is critical that the part be positioned accurately to avoid detector shadowing. This occurs when the part blocks its own x-ray emissions. Samples should be rotated so that there is an unobstructed path to the detector or, in the case of some samples, destroyed to provide proper access to the measurement surface. Also, systems can be equipped for measuring thickness in recessed cavities.

The most important thing to remember is to orient long and narrow parts perpendicular to the long axis of the detector, in other words, with their opposite ends facing the detector and chamber door. Parts must also be positioned so that the area under the x-ray beam is flat or "normal" to the beam. If a complex-shaped part does not lay flat on the stage, it should be propped up using a stage fixture, or even clay, so that the measuring surface becomes perpendicular to the x-ray beam. Positioning errors or detector shadowing can result in measurement errors.

Measurement accuracy, precision and reproducibility. The accuracy of an XRF measurement refers to how close the measurement value conforms to the actual value. Measurement accuracy is mainly a function of the calibration accuracy, which is a function of the accuracy of the algorithm used in the instrument's software, as well as equipment condition, the measuring time used in calibrating, and the accuracy and condition of the standards.

The accuracy of a single measurement, however, is highly dependent upon the precision or repeatability of the measurements. Precision describes how well a series of measurements taken on the same area of a sample (without moving the sample) gives the same or nearly the same reading. Perfect precision is impossible; we can get close.

Measurement precision is indicated by the "predicted" percentage uncertainty of the readings and upon countrate. Percentage uncertainty represents $2s$[7], expressed as a percentage of the measurement value. For example, if a 50 microinch reading has 3 pct uncertainty, this means that the uncertainty was plus or minus 3 pct of 50 or plus or minus 1.5 microinches. Therefore, we can say with 95 pct confidence that the actual value is between 48.5 and 51.5 microinches. Obviously, the lower the percentage uncertainty, the more precise or repeatable the measurements. If we take only a single reading on each part, it is easy to see why it is important to keep precision as high as possible, or the pct uncertainty low.

Precision is related to the square-root of the counts obtained in the ROI during the measurement cycle. For example, if there were 100 counts, the countrate standard deviation would be the sq root of 100/100 = 10/100 = 10 pct. However, if more counts were collected, say 10,000, the resulting standard deviation would be sq root of 10,000/10,000 = 100/10,000 = 1 pct, a significant improvement. For thickness and composition mea-

surements, the use of the more efficient Mo-target Be-window tube provides much higher countrates and better precision.

Precision can be maximized by using the largest beam size appropriate for the sample to be measured and using the longest possible time. Remember, by increasing the measuring time by a factor of n, the pct uncertainty decreases by 1/sq root of n. By quadrupling the measurement time, say from four to 16 sec, the pct uncertainty of the measurements will be cut in half!

The XRF instrument user should be knowledgeable in the basic components of an XRF measurement instrument so that the advantages and limitations of the various systems available can be understood and equipment selected appropriately.

An understanding of applications, including proper sample positioning, collimator selection, and measurement accuracy, precision and reproducibility can help the user solve application problems and eliminate many sources of error.

Instruments should be selected on the basis of performance, minimization of operator errors, and availability of accurate measurement algorithms and good thickness standards. By following proper operating procedures and precautions, XRF measurement instruments can be used as an effective tool for the quality control and/or process control of plated materials. **PF**

More Information?

To obtain more information on products or processes mentioned in this article, circle corresponding numbers on the Reader Service Card.

X-ray fluorescence thickness measuring instruments..... Circle 275

To request an additional copy of this article, write on company letterhead to "Reprints," c/o PRODUCTS FINISHING, 6600 Clough Pike, Cincinnati, OH 45244.

1. SPECTRAL analysis screen
2. PRINCIPLES of x-ray fluorescence measurements
3. XRF fluorescence thickness and composition measuring system

[1] literally, "braking radiation" resulting from the deceleration of the electrons bombarding the target in the x-ray tube.

[2] This is accomplished by converting the analog voltage representing the pulse height to a digital value, for current XRF systems, on a scale of 0 to 1023. This value becomes an address to a channel whose content or "count" is incremented. Each channel represents a different x-ray energy and the count in each channel represents the x-ray intensity at that energy. The result is an x-ray spectrum.

[3] the energy at which a photon will cause characteristic emissions in a given material at highest efficiency.

[4] Each calibration mode represents a specific technique or mathematical algorithm for effectively analyzing sample spectra and obtaining thickness and/or composition of the layer or layers involved.

[5] Infinitely-thick refers to that thickness at which any further increase in thickness produces no increase in counts.

[6] The gain and offset of the spectroscopy amplifier are controlled by software and adjusted until nickel and tin appear in the correct channels.

[7] One sigma (σ) represents the area under the normal distribution within which approximately 68 pct of the readings occur and, therefore, gives a 68 pct "confidence level". Two sigma (2σ) represents 95 pct of the readings and, therefore, gives a 95 pct confidence level which is more meaningful.