Other Methods of Thickness Testing

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This presentation will discuss methods, equipment and applications for thickness testing using the following systems: cross section, coulometric, magnetic induction, eddy current and beta backscatter.

For more information, contact: Joe Cummings Kocour Co. 4800 S. St. Louis Ave. Chicago, IL 60632 Phone 773-847-1111 FAX 773-847-3399 Coating thickness gauging has been an integral part of the metal finishing industry from the very beginning. Without accurate dependable gauging systems the plater cannot control costs, control product, or guarantee the customer that work specified is work received. Today's marketplace guaranteeing the work has become tantamount to keeping the work. With quality programs such as ISO and QS are being adopted by more and more manufacturers, incoming inspection criteria are becoming increasingly stringent. In order to stay in business, todays electroplaters today must be assured that the product leaving his facility will satisfy increasingly more stringent quality requirements. Rejections and rework are just too expensive.

Once the decision is made to put some type of gauging system in place, a whole new set of questions arise that must be addressed before the appropriate system can be purchased and put on line. As an owner or facility manager you must ask yourself the following questions: What type of measuring system best fits our needs? Are we limiting this system to do one very critical job or will it be used on all our products? How much money do we need to spend? How much money can we afford to spend? What kind of system is our customer using and will the system we buy be in substantial agreement with theirs? This last question is often overlooked and the consequences can be hugely frustrating.

The purpose of this discussion then is to give you a general overview broad general understanding of the methods and equipment available today that can be tailored to fit your existing coating thickness gauging requirements. The one system we will not discuss is X-ray diffraction. This method is generally recognized as one of the best available technologies existing today and is being presented in depth in another session. The only major drawback to the use of X-ray is the cost. Under circumstances where the cost can be justified due either to the number of parts requiring testing or the cost of individual parts and the reluctance to destroy them in the testing process, then X-ray is the way to go. However, if this justification is not readily evident, then there are other reasonable approaches.

The other testing methods available are primarily divided into two groups: destructive, where the tested part is sacrificed, and nondestructive where the specimen is left intact. Starting with the destructive group, there are two common systems. The first of these is called cross section analysis. In this process a slice (section) of the subject coating substrate is carefully prepared and mounted in a backing material. When the specimen has hardened, it is polished on a series of abrasive disks to a very high finish. This allows the various layers of coatings and substrate to stand out in high relief when placed under a microscope containing a calibrated lense. The thickness of one or more electroplated layers is then read directly against the calibrations on the lense. This method is widely used in metallurgical laboratories and for many years was popular in larger electroplating facilities where the cost of equipment and technical skill could be justified. It is listed as a referee method by NIST and is still used to settle disputes. The obvious drawback is the cost. A second important problem involves the measuring of very thin deposits. The polishing operation used to prepare the specimen will generally smear the layers slightly and on thin deposits this smearing or drag can increase the margin of error dramatically.

A second and very popular destructive method is the coulometric process. In this process, a miniature anodic deplating cell is fixtured on the surface to be tested. With the use of electrolytes, the coating is removed into solution and its' thickness displayed digitally. Since each electrolyte is specific to the metal to be tested, the system will automatically stop and display a reading whenever the next layer is reached. For instance, the nickel electrolyte will not deplate copper, zinc, steel or any other metal. The endpoint mechanism that controls this process is a function of the electrical resistance in the deplating circuit. Coulometric testers will test all of the common metal topcoat and substrate combinations in use today. The system can also be used to test multiple coatings such as chrome, nickel, copper over zinc die-cast and are very successful in accurately measuring coatings on plated wire. Finally this system will perform the so-called STEP test which measures the difference in electrochemical potential between the two

layers of a duplex nickel coating system. The magnitude of this difference is directly related to the corrosion resistance of these systems and is a valuable tool for any shop dealing in automotive hardware.

Coulometric measuring systems have several major advantages to recommend them. They will measure virtually any electroplated coating on a wide variety of substrates. The repeatability of the process is excellent, so the plater and the customer can be assured of producing similar test results on the same part. The accuracy of the system is quite high for several reasons. As mentioned earlier, each test electrolyte is specific to the metal being tested and the process will cease when that metal is consumed. Also the endpoint is not operator dependent. The system determines where the test will stop. Accuracies of $\pm 5\%$ are common and with proper calibration of the deplating current and the use of a strip chart recorder in parallel with the test equipment greater accuracies are attainable. Finally, coulometric systems are relatively low cost.

There are some drawbacks associated with coulometric systems. Primarily, it is They are a destructive test. One of a kind or expensive parts should not be measured in this way. Secondly, there are some size limitations as the test cell must be affixed to the part such that the electrolyte will not leak. However, at least one manufacturer has a test cell with a deplating test spot diameter of 0.03", although it must be remembered that smaller test areas negatively impact repeatability and accuracy. Further, this is a wet chemistry process and requires good housekeeping to function properly. Finally the test is relatively time-consuming. The average set up to breakdown time for a test the is test requires about three minutes. Obviously, if large numbers of parts need to be tested, adequate operator time is required.

There is a third destructive method that I will only mention in passing because the equipment is no longer being manufactured. This method is called the drop test and relies on the fact that certain acid solutions will dissolve metallic coatings at a fairly constant consistent rate. If the specimen is fixed in such a way that the acid solution can make contact in a steady stream of droplets, then a stopwatch or a counter can be used to measure the time it takes for the acid solution to remove the coating and expose the substrate. Approximately 1/100,000 of an inch of coating will be dissolved for each second of elapsed time. A simple calculation at the end of the test will result in a reasonable estimate of the coating thickness. This system was very popular with the fastener industry for years due to the requirement for testing coating thickness on threaded parts.

Moving on to the nondestructive testing methods, there are four major types, permanent magnet instruments, magnetic induction methods, eddy current, and beta backscatter. The oldest of these methods is the permanent magnet instrument. They range from the simple pocket pen style gauge to the most sophisticated of this type, that employs a counterbalance, a high quality spring and a gear mechanism to delicately exert pull on the permanent magnet to the point at which it releases. These instruments will measure with some degree of accuracy, any non-magnetic coating on iron or steel. They all rely on the fact that their magnetic fields weaken in proportion to the distance they are removed from the ferrous base metal. In other words, the strength of the magnetic field is inversely proportional to the thickness of the coating. With all of these instruments the permanent magnet is brought into contact with the surface of the test piece and then drawn away by means of a spring-loaded lever or dial until the magnet breaks contact. At this point a thickness measurement is either read directly from the instrument or is extrapolated from a graph to which the instrument has been calibrated. These instruments generally will not measure nickel coatings because nickel has magnetic properties of its own; however, with the use of a counterpoise which allows the unit to measure the magnetic field of the nickel coating itself, that field being directly proportional to the thickness of the nickel layer, some success is achievable. Repeatability of these instruments is for the most part good but due to a number of factors, especially operator error, accuracy can never be better than $\pm 10\%$. These gauges have great value, however, because they are inexpensive, very portable, and can be used as front line detectives to uncover problems early in the process. These problems can then be verified with more sophisticated equipment and corrective action can be taken earlier than would otherwise have been possible.

More sophisticated than the permanent magnet gauges but relying on the same basic principle are the magnetic induction instruments. These units use a coil to generate a magnetic field in the iron or steel substrate. Again the strength of that field as measured by the probe is inversely proportional to the thickness of the interference layer (i.e., the coating). Magnetic induction units can be portable or bench top and will measure many types of coatings, including paint, powder coats, lacquer and certain electroplated topcoats. Typically, this method is not amenable to nickel coatings due to the magnetic permeability of nickel. Because they are electronic, a large array of statistical software can be built in to these systems. This allows for the gathering of a great deal of data, such as a large number of individual measurements around the surface of a single part or measurement on the same spot on a vast number of parts, and the generation of valuable statistical information, such as high, low, number of measurements, average reading, mean, and six sigma projections. This last being a valuable predictor of the failure rate associated with a particular batch of parts.

Proper calibration is extremely important when using a magnetic induction instrument. To be accurate, the instrument must be calibrated introduced to a true base line for the product to be tested. More often than not, the use of a hard standard will not achieve net this result. Hard standards are produced using high quality, homogenous cold rolled steel. Generally, the products to be tested will not be fabricated with metals of this quality. Further, most products are not flat or perfectly smooth. Since these instruments must be calibrated to a zero point, (bare steel), and an infinity point, (usually a coating of about 25 mils), the use of mylar foils and a raw part from the batch to be tested will yield the most dependable calibration results. This method of calibration helps reduce margins of error from shape effect, edge effect and metallurgical variations from batch to batch.

Properly calibrated and applied, magnetic induction instruments will provide a high degree of accuracy in the measurement of non-magnetic coatings over ferrous-based parts. Results with an error margin of 5% or less are common.

Another method of testing that has had wide acceptance over the years is the eddy current method. Instruments relying on this principle are generally used in the measurement of non-conductive coatings over non ferrous bases. Applications such as anodizing over aluminum and paints and laquers over copper and brass can be measured very effectively in this way. The principle of operation relies on the generation of an eddy current field in the base metal. As with magnetic induction, the strength of that field is inversely proportional to the thickness of any interference layer. Theoretically, eddy current systems can be used to measure any topcoating substrate combination where the conductivities of coating and base differ in a ratio of 2:1 or greater. In practice, the system works best when the coating is non conductive. Advances in eddy current technology over the past few years have helped to make these systems suitable useable for a wider variety of topcoating substrate combinations. Matching the probe frequency (which largely determines the depth of penetration of the eddy field) to the nominal thickness of the coating substrate greatly increases repeatability and accuracy. The use of combination probes, magnetic/eddy allows for the simultaneous reading of multilayer coatings such as paint over zinc over steel. Lift-off compensation of the probe system allows for success in some non-contact applications where the probe can be fixed at up to 50 mils above the test piece and produce product repeatable measurements.

It must be remembered when considering an eddy current system that the results are based on conductivity of the layers being measured. This means that the method is temperature sensitive and density sensitive. The latter mandates that hard standards, where they are to be used for calibration should be plated from the same type of bath as the test piece. As with any other type of measuring instrument, the best predictor of the success for any application is the field test, the results of which can then be compared to results from a referee method and the repeatability and accuracy of the system firmly established.

Calibration is extremely important to success with these instruments. Reducing error factors such as those mentioned in the magnetic induction section, edge effect and shape effect, can only be accomplished if calibration is done on a representative part. More importantly, with eddy current, differences in conductivity from one metal to another make it less desirable to rely on specific hard standards for calibration. Because these instruments are electronic, statistical software can be incorporated into the systems, greatly improving the repeatability, accuracy and volume of information retrieved.

The last and most sophisticated test method to be discussed here is called beta backscatter. This method is best adapted to measure very small surface areas, very thin coatings, and precious metal coatings. It can however, be used to measure any coating substrate combination where the real or equivalent atomic numbers of the two layers have a difference of at least five. The system is non-destructive and non-contact, and when properly calibrated, is highly repeatable and accurate.

In simplest terms, an energy source such as strontium 90 or promethium 147 is used to bombard the test piece with beta particles. These particles then backscatter at known rates, depending on the atomic weights of the materials being tested. The test area is delineated and sealed using carefully constructed platens and the backscatter is collected with a Geiger Mueller tube and counted. The resulting counts produce measurements of mass per unit area and are directly proportional to thickness.

Calibration with these units will not be accurate unless the standards used have the same mass per unit area as the test part. The strength of the energy source is also important and information is readily available to allow you to match the most appropriate source to the measuring problem at hand. Platen geometry is critical. The test area must be accurately defined and sealed against beta particle escape. Both the calibration standard and the activity of the source degrade over time. It is recommended that the radiation source be replaced before its half-life is reached. Regular calibration upgrade is also important as the energy sources continuously degrade and the instrument must be compensated for this.

These instruments have proven very popular with the electronics industry for the reasons listed above, utilization for thin, coatings expensive coatings and the need to preserve the test specimen.

In conclusion, when choosing a measuring system, remember what was said at the outset of this paper. Know your requirements. Pick the most appropriate system for those requirements. And finally, don't get bogged down in the quest for accuracy. The repeatability of a measuring process is often a more important criteria for choosing that process than is the manufacturer's accuracy statement.