On-Line Determination of Chromium Thickness During Plating

By A. Abbate, J. Frankel and M. Doxbeck

A new system that enables the plater to detect the thickness and the rate of application of chromium during vessel plating of steel tubes, has been designed and developed. This system provides a continuous computer display of thickness, rate of deposition and temperature at six different points of the tube during the plating process, with a thickness measurement resolution of 2.5 µm and an accuracy of about 7.5 µm. Prior to development of this system, this information could be obtained only after the process was finished and the specimens were cooled, cleaned and dried. The system can be used to control process parameters, **such as the rate of** flow of liquid or the temperature of the solution.

Electroplating is "blind" in the sense that no plating thickness information exists during actual electrodeposition. Until now, the plating parameters for the vessel plating process (current, flow rate and temperature) have been based on the plater's experience.[1] The proposed technology makes chromium thickness information available to the user during the operation, conveying the possibility of effecting appropriate and timely changes in the process parameters.

In the vessel plating technique for large hollow tubes, the bore contains the plating solution, which is pumped in at the bottom at about 85 "C (185 "F) and exits the top at a higher temperature. Currents on the order of 20,000 A are sent through the tube (the cathode), pass through the plating solution and return through the lead-plated anode (Fig. 1).

Ultrasonic pulse-echo techniques represent the only technology developed to date that can be used for thickness measurements when just one face of the substrate is accessible and there is no direct access to the plated surface, but special care must be taken to ensure stability of the echo return time measurement. A one-nsec(10-9 see) change in return time signifies approximately a 2.5-µm (10-4 in.) change in thickness. Our efforts were directed at limiting fluctuations of the measured return time to this range. Transducer holders and fixtures were designed and applied for a special liquid-buffer coupling technique. A specific coupling agent was used and a system devised for temperature compensation. A computer program was written to gather data (return times and temperatures), to calculate thicknesses and plating rates with temperature compensation, and to display all the information in numerical and graphical formats.

Taper or uneven distribution of chromium in the cylinders can easily occur as a result of various plating conditions. This is a serious problem, handled in the past by varying the plating parameters for the next tube. The new technique enables the operator to detect immediately any evidence of problems such as tapering, intermittent plating or curvature of the anode, so that a correction can be made promptly during the run, either by varying the plating parameters or by stopping the process.

Even though this system was designed for a vessel plating facility, its principle can be applied to any general plating set-up. The fixtures used to hold the transducer in place may differ from the one presented here, but the electronics and the software remain essentially the same.

Theory and Application

Thickness determination using ultrasonics is based on measurement of the time delay of a pulse traveling between two parallel surfaces. In the pulse echo method, if the velocity of the sound wave through the material is known, the thickness can be determined from the time measurement.'

A schematic of the fixture used in our measurements is shown in Fig. 2. An ultrasonic pulse from a transducer traverses the coupling medium or buffer and enters from the outside cylindrical surface of the tube. This pulse travels radially in the tube and is reflected back to the transducer from the inside diameter at surface B, before plating starts, or at surface C,

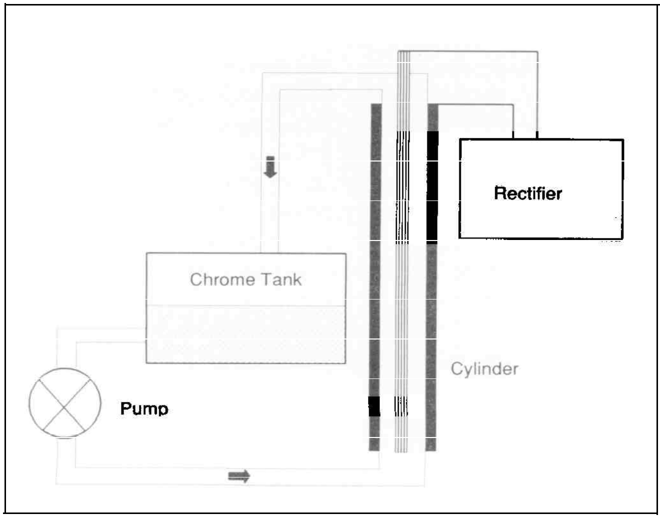


Fig. 1—Schematic representation of the flow-through process for large hollow cylinders.

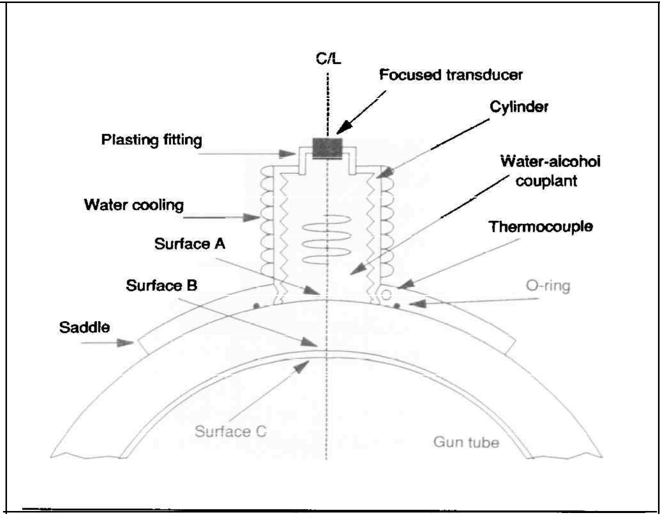


Fig 2—Sketch of the fixture using liquid-buffer coupling of transducer.

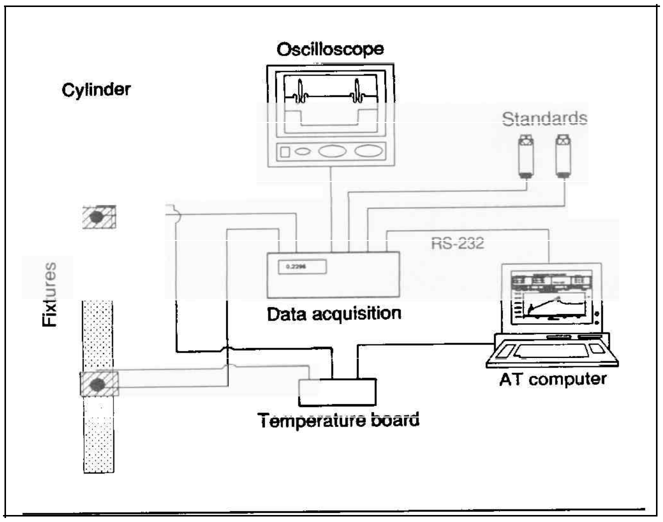


Fig. 3-Schematic representation of the ultrasonic measurement system.

Fig. 4—Photograph of the actual fixture used for the ultrasonic thickness measurement.

when some chromium has accumulated. There is also a partial reflection of the sound energy from surface A, for which a pulse that has only traversed the coupling medium is also detected. The difference in return times between the reflection from C and the reflection from A is processed by the system. At constant temperature, this difference is a measure of the thickness of the steel-chromium combination, the steel thickness being constant and the chromium increasing during plating to an ultimate thickness between 125 and 250 µm (0.005 and 0.010 in.). The total wall thickness of the tube ranges from approximately 2.5 cm. (1 in.) to 7.5 cm. (3 in.). It is important to note that the proposed measurement system is valid as long as the inside and outside surfaces are parallel.

Several concerns were addressed during the design of the measurement system and of the fixture holding the transduce

1. The mechanical construction and coupling of the system had to be sufficiently stable for minute changes in the return time, one nsec, to be associated with changes in chromium thickness.
2. During plating, the steel tube temperature can be as high as 95 °C. The high temperature of the tube could not be allowed to damage commercial transducers.
3. In order to measure the distance between parallel surfaces, the ultrasonic beam had to be directed along the radius of the tube.
4. The transducer had to be electrically insulated from the plating circuits.

The most feasible solution to these problems was found in using focused transducers, coupled to the tube through a constant liquid path, with constant alignment such that the beam would travel radially to the tube (Fig. 2). Commercially available, one-in. diameter, focused transducers, with 5 MHz center frequency, were placed 120 **degrees apart** in two rings at different positions along the tube. A watercooling line around the cylinder containing the liquid coupling agent was also used to reduce the temperature at the transducer (Fig. 2). By using a configuration of six transducers located on the two rings, we tried to optimize the information obtained for an overall description of the plating (Fig. 3).

A photograph of the actual fixture (one ring) used is shown in Fig. 4. The set-uptime of each fixture is roughly 30 min for one operator. Much less time is required for breakdown. Usually, two operators are involved insetting up the measuring system, to speed the process.

By comparing measurements between transducers on the same ring, the effect of the anode curvature and the wall variation of the chromium around the tube could be evaluated. Further, by comparing results between the two rings, the evenness of the plating along the length of the tube and, in particular, any possible tapering could be estimated.

The velocity of a sound wave in steel is a strong function of temperature. Therefore, the return time in the steel substrate must be temperature compensated to achieve high resolution in thickness measurements. Consequently, the temperature at the surface of the tube near the transducer was monitored by a thermocouple and recorded by the computer. Ideally, the temperature distribution in the wall where the ultrasonic transducer was placed should have been obtained. Usually, the temperature varies according to the current density distribution in the wall, the temperature of the incoming plating solution, the heat diffusion in the steel, the heat transferred to the bubble-filled plating solution, the radiation to the atmosphere, and the starting temperature. As a first order approximation, it was assumed that the temperature was constant throughout the material.

Satisfactory real-time chromium thickness measurements were made, because the temperature of the outside tube diameter and of the incoming and outgoing plating solution were roughly constant. This means that the temperature during the plating process remained unaltered and, consequently, that the monitored change in return time could be correlated with the change in thickness of the plated chromium.

The measurement was not only performed continuously on the six transducer locations, but similarly on two parallel steel disks of known but different thicknesses (the standards). The results obtained from the two steel standards were used not only to convert the voltage readings to thickness, but to estimate and correct any drift in the electronic circuitry as well.[3]

The equipment used for the measurement generates a voltage V, proportional to the time between reflections from surfaces A and C (Fig. 3) for the specimen or standard. To calculate x, the temperature compensated change in thickness, the following relationship was used:

$$x = \frac{s}{V_{s2} - V_{s1}} \cdot \left[V(T) - V_o(T_o) - \frac{(V_{s2} - V_{o2}) - (V_{s1} - V_{o1})}{2} - \beta V_o \cdot (T - T_o) \right] \quad (1)$$

V_{S1} and $_{S2}$ are the voltage readings for the two standards, V is the voltage reading from one of the transducers on the tube, T is the surface temperature of the tube near the transducer and ß is the coefficient of change of sound velocity with temperature. All quantities with the subscript zero represent initial readings. The term $V(T) - V_0(T_0)$ represents the difference in voltage readings at the same transducer at two different times and possibly different temperatures. This quantity represents the change in thickness as a result of plating. In the ideal case, T= TO and all the other terms are zero. The second term on the right hand side of Eq. (1) accounts for any electronic drift of the equipment, and the last term represents the temperature correction, *viz.,* it takes into account the change in velocity resulting from a temperature T different from TO.

The Measurement System

The measurement system consists of a multiplexed ultrasonic gage,[a] an oscilloscope, and an IBM-compatible AT/PC with an A/D temperature board.[b] The multiplexer continually samples the eight transducers, with a four-see interval between transducers. By utilizing the pulse-echo method, the system determines the return time of the ultrasonic pulses. The voltage proportional to this return time is transmitted to the computer in ASCII format via an RS-232 port. The least count, or resolution of the system, is 2.5 µm. Two of the transducers connected to this system are used to measure the thickness of the standards. The standards consist of parallel steel disks, and their difference in thickness is 0.509 cm. The oscilloscope, which is connected to the multiplex system, allows viewing of the echo train of the transducer in use. The computer records, stores, computes and displays the data. An analog/digital temperature board inside the computer is used to measure temperatures. A complete cycle of measurements comprises the eight transducers and eight thermocouples, with a duration of 40 sec.

The program used for data acquisition, analysis, and display was written in Quick Basic 4.5. The program provides a variety of graphs of the variable vs. time, while the plating system is operating:

a. Voltage-the raw data from the multiplex system;
b. Temperature;
c. Thickness [without temperature correction; Eq. (1) with ß = 0];
d. Temperature-corrected thickness.

All of these graphs can be called to the screen. They maybe either absolute values or relative to a “reset” value defined by the user. This is very useful because actual plating does not start until approximately 30 min after the system has been initiated. Accordingly, the thickness of the chromium plated at time (t), can be obtained from the difference between the values of the thickness evaluated at that time and at the start of plating.

The user also defines the start and stop points, with two markers, for determination of the plating rate. As shown in Fig. 5, the markers have been positioned at times 60 and 250 rein, so that the straight line joining them fits the actual thickness data. Here, the plating rate was 54.7 µm/hr (0.002153 mil/hr) and the thickness difference between the two markers was 177.8 µm (0.007 mil). The data shown in Fig. 5 were obtained during an actual plating run. The accuracy of the thickness measurement was calculated to be 7.5 pm. During the run, eight transducers and eight thermocouples were used, though for simplicity, the numerical display in Fig. 5 shows only three transducers. STAND 1 and STAND 2 measure the standard voltages and TRANS 3 measures tube thickness. The three

[a] *Model 5215, Panametrics, Inc., Waltham, MA*
[b] *Industrial Computer Source, San Diego, CA*

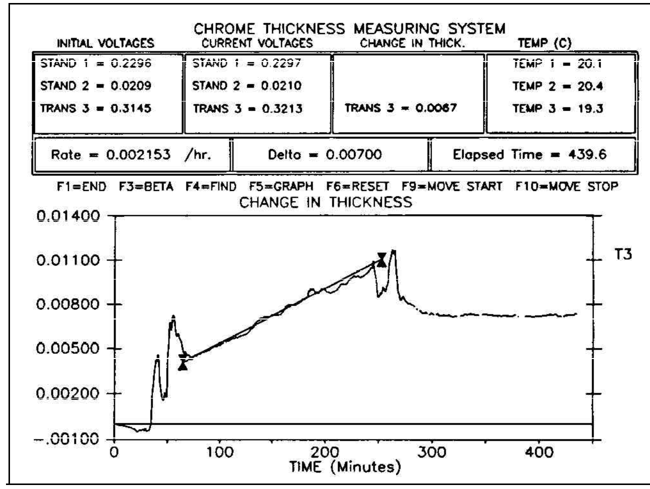


Fig. 5-Computer display during the plating run. The straight line is a fit used to obtain the plating rate. The beginning and endpoints of the line are set visually by the operator, and are used for determining the change in thickness (Delta).

thermocouple readings measure the temperature of the steel near the transducers at the elapsed time shown on the screen. The computer cycles in a loop of data acquisition/analysis as long as desired. The data are saved in an ASCII file, and can be imported into any program, such as plotting utilities or spreadsheets for further analysis.

Results

The system described has been implemented for many months in our vessel plating facility. The following are some of the situations encountered during testing of the system.

First, a normal run was made. A plot of some of the data obtained is shown in Figs. 6 and 7. In Fig. 6, the temperature of the fixture, and therefore the approximate temperature of the cylinder at that level, is plotted as a solid line, while the temperature of the transducer is plotted as the dashed line. The two arrows labeled START and STOP represent the start and stop times of plating, respectively. It can be seen that the temperature of the tube increased rapidly as plating started, then remained practically constant until plating was stopped. Figure 7 shows the change in thickness measured by one of the transducers. The change increased linearly during plating at a constant rate of 58.4 µm/hr. The final thickness measured was 182.8± 7.5 µm. Mechanical measurements showed a thickness of 190.5 ± 12 µm.

There were instances in which the system was able to inform the plater of problems. For example, in a very early run, it was

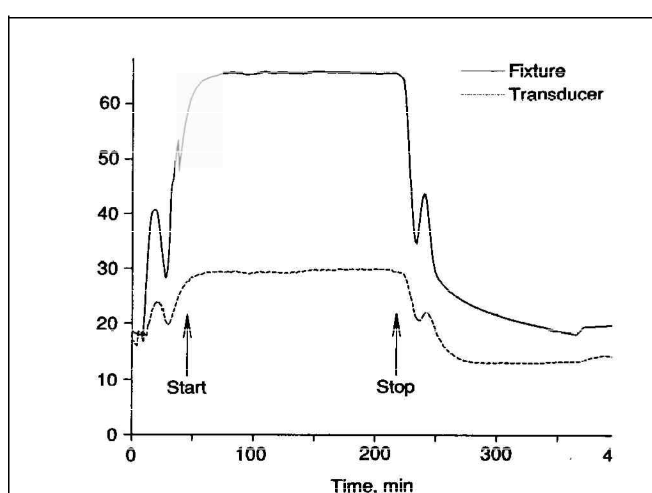
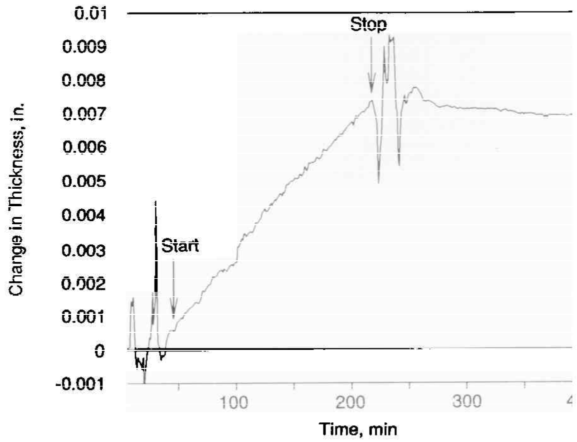


Fig. 6-Plot of the temperature of the fixture and of the transducer during plating.



ig. 7—Change in thickness measured ultrasonically during plating. Quantities are expressed in inches.

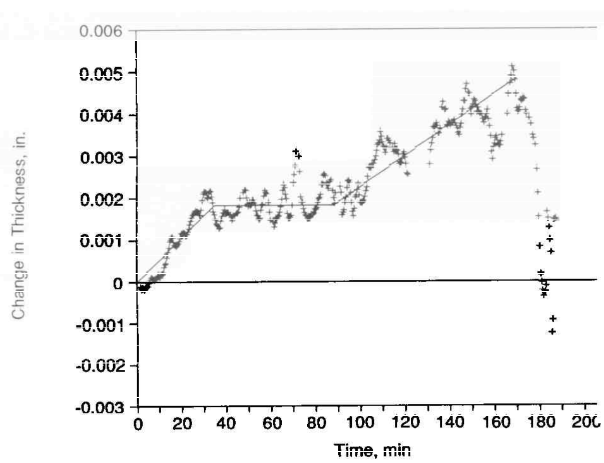
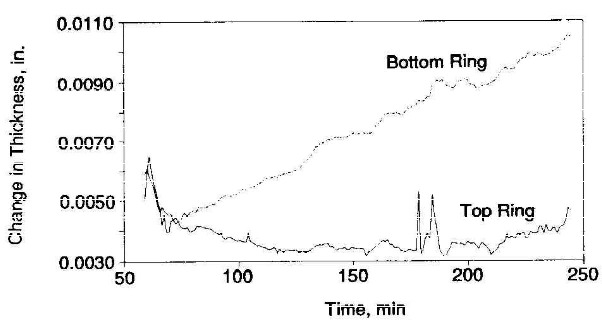


Fig. 8—Change in thickness measured during plating. The three lines were plotted to indicate the regions with different plating rates, as described in the text.



ig. 9—Run showing drastically different rates on the two rings.

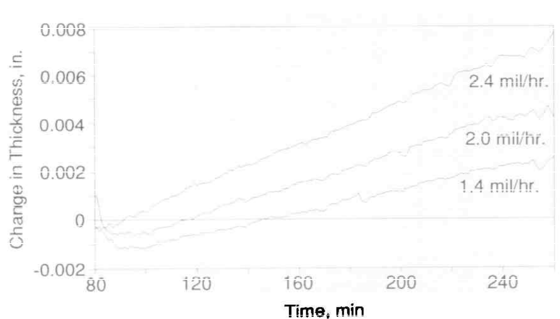


Fig. 10-Data obtained during one test run by three transducers on the same ring. Different rates of chromium deposition were observed.

possible to make a quantitative determination of an interruption in deposition after a small interruption in current (Fig. 8). The change in thickness data showed an initial plating rate of 76.2 µm/hr, then a level region and a third region with plating rate of 50.8 µm/hr. The flat portion began just after a small interruption in current because of a failed rectifier, which was quickly switched out. Even though the interruption was very short—less than one rein-we monitored an interruption in plating of approximately 50 min. Because the plating solution is a strong oxidizer, the cathode surface could have become passivated by formation of chromium oxide. If so, increased hydrogen formation resulted without deposition of chromium. Hydrogen, however, is a reducing agent and, over the 50-min interval, acted to clear the cathode surface of the oxide, allowing deposition to resume. Again, our results were validated, because they matched quite well the value obtained mechanically. The data shown in Fig. 8 are somewhat noisier than the other data shown here, because they were taken from very early measurements, using different equipment.

During one of the testing runs, a tapering effect in the plating was observed (Fig. 9). The cylinder under test was longer than usual and the anode had to be extended via a screwed-on section. On the top section, the lead was electroplated on and, at the bottom, it was “burned on. Six transducers were mounted and data gathering was successful from four—two at the top and two at the bottom. The top transducers, mounted above the new anode, recorded a slight loss in thickness during plating, while the lower ones recorded an increase in thickness (Fig. 9). We were able to detect this anomaly in deposition along the tube during plating, and results were later confirmed by mechanical measurements. The reason for the negative growth rate measured at the top ring in Fig. 9 is not clear. As already mentioned, the lead coat of the anode corresponding to the top ring was applied differently from the rest, and it is believed that its surface was not properly activated. Another detail of interest is that a chromium film approximately 76 µm (0.003 in.) thick was already present on the material as a result of previous plating. Accordingly, in the run shown in Fig. 9, the thickness of the chromium film was reduced, but not the steel substrate.

A serious problem encountered in plating long tubes was the straightness of the anode. Any curvature of the anode causes a different spacing between the anode itself and the inside diameter of the tube, resulting in a different amount of chromium deposited across the section. The system was able to detect this problem by comparing the measurements of the three transducers on the same ring. Figure 10 is an example of such readings. Three different rates of increase of thickness were measured on the same ring with variations up to 30 percent of the average rate.

All the data discussed were obtained from test runs during development of the ultrasonic measuring system. Now, with the system fully developed and in use, plating is stopped or remedial action is taken as soon as any of the problems mentioned are detected.

Conclusions

We have added ultrasonics and computer technology as the means for “seeing” during plating. Now, evenness of the plate, its thickness and the plating rate at six specific locations, both around and along the tube, can be evaluated while the tube is being plated. Measurement resolution is much better than that of mechanical measurements performed after the process is finished. The resolution is 2.5 µm, and the accuracy is better than + 7.5 µm.

With this system, it is also possible to accomplish plating R&D. It would be useful to explain the effects measured quantitatively, that were previously unpredictable or irregular (e.g., interrupted plating, inadequate treatment of anode or cathode in the shop, and uneven plating around the tube). It is also possible to observe different rates of plating at different levels in the tube. The most important result is that the plater can intervene during plating, by changing the flow rate or other plating parameters, for remedial action. All this could not be done before, either in the laboratory or in the shop. Now, quantitative observations are available in the shop.

Further studies would be fruitful for process control, temperature compensation, current density distribution in the tube and complete system description of the process.

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