Study of the variables that influence the coulometric determination of tin oxides in tinplate

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One of the stages of the tinplate manufacturing involves a heat treatment at temperatures above 230 °C (brightening) right after the electrolytic coating in order to convert the matte appearance of the coating into a smooth bright continuous coating. During this process tin oxides are quickly formed due to high temperature oxidation of the tin coating. The tin oxide films have been associated with adhesion failures of organic coatings, surface discoloration and soldering problems, so tin oxide film control is required for quality purposes during manufacturing.

Due to the low thickness of the oxide film, it is difficult to measure the oxide film formed. One of the easiest methods used is the coulometric method.

The coulometric method consists of reducing tin oxides by passing a small amount of cathodic current in a solution free from oxygen and inert to tin oxides. The progress of reduction of the oxide is followed by potential measurement and the charge passed for the complete reduction serves as a measure of the tin oxide on the surface. This method is based on the Faradays law that relates the reduced mass with the passing charge by means of the following equation:

\[ m = \frac{1}{F} \cdot t \cdot i \cdot E, \]

being \( F \) the Faraday constant, \( i \) the electric current, \( t \) the time during which the current passes, and \( E \) the equivalent weight.

There are several factors regarding the coulometric test that can influence the results obtained:

Film thickness: the higher the film thickness, the better the accuracy of the measurement. Current density: a too high current density can modify the reduction yield or hinder the test end point detection. Test surface area: the higher the surface area measured, the better the accuracy of the measurement. Dissolved oxygen: if existing in the solution, it can be cathodically reduced and interfere in the oxide reduction measurement. For this reason, a minimum deaireation time should be used before the start of the test.

A study of these variables was performed in order to optimize the test configuration for accurately measure tin oxides in tinplate samples. Some variables, such as the test surface area, do not significantly influence the test results, whereas others, such as dissolved oxygen in the test solution, have major interferences, and were optimized for better results.