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Measuring the qualitative factors on copper wire surface

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ABSTRACT

This paper is focused on improvement of quality of copper wire manufacturing process. Through the Design of Experiment (DOE) method, it presents the way to keep the thickness of oxide layer during rolling copper wire at the level requested by customer. Variation coefficient of measurement replications performed in each test within the planned experiment was used as the response in the DoE method. Settings for the selected process parameters of copper wire rolling that have a significant effect on the thickness of the surface oxide layer, and were obtained on the basis of the planned experiment, were verified in the production conditions.

1. Introduction

Ever increasing demands of customers require manufacturers to continually improve quality of their products. Succeeding in the current competition means paying due attention to improvement of manufacturing process, upgrading the existing machinery, optimizing the process and product parameters, and all by means of different improvement tools [3,13].

All real processes are stochastic in nature. Random variation is a part of all processes, i.e. including production processes. The variation of individual parameters generally represents an undesirable phenomenon particularly in the process of mass production. The message of W. Edwards Deming, the quality guru says: "If I had to reduce my message to management to just a few words, I'd say it all had to do with reducing variation." [5].

By using the example of quality improvement of 8 mm copper wire casting process, we are going to demonstrate that even with the use of Design of Experiment method, it is suitable to pay due attention to variability of parameters in addition to the attention paid to the characteristics of their position.

The DoE method is aimed to examine the relation of various independent variables – factors to the dependent variable – response [4,10]. Planed experiment follows in advance elaborated strategy. Plan of the experiment states the number of attempts, consisting the experiment, conditions under which attest happen and order of the attempts. The attempt is being understood as a tool detecting the value of the quality indicator at certain (preplanned) conditions. Experiment is being understood as the system of all attempts [11].

In the practical implementation of this method, most experimenters measuring the response focus on the parameters of the position, i.e. the average, respectively, other mean values, in which case the calculation of variation is generally focused mainly on the evidence of suitability, respectively, the relevance of the mentioned position parameter. We are going to demonstrate that sometimes it is suitable to use the actual variation parameter as the response.

2. A brief description of copper wire production

Production of 8 mm copper wire runs continuously. It consists of copper cathodes melting in a shaft furnace, molten copper casting and its controlled solidification in the crystallizer, hot rolling of the copper cast to an 8 mm-diameter wire, reduction, cooling, cleaning the surface with isopropyl alcohol, and the wire surface passivation with wax to protect it from oxidation. The wire made in this way is then coiled into 3.5, 4, 5-ton coils, stored on wooden pallets, and foil-wrapped.

The melting charge consists of copper cathodes, and a small part of recycled material (max. 5% of the melting charge). Copper cathodes are made of an electrolytically pure copper (99.95–99.99% Cu). A returnable waste copper material consists of cast copper wire coils unsuitable for shipment, and cut off sections from casting wheel. The quantity of returnable copper is at a level of about 3 kg per 1 ton. The melting charge is melted in the furnace with a

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moderately reducing atmosphere (0.1–3.0% CO). The molten liquid copper flows down the pouring gutter into a fixation furnace that serves as a reservoir for a perfect homogenization of the liquid metal, for slag separation and extraction, and for fine-tuning the temperature of the molten metal. The homogenized metal flows from the fixation furnace through the casting gutter into the casting machine funnel. The temperature of copper in the casting machine tundish ranges between 1115 and 1135 °C and the oxygen content ranging between 0.0001 and 0.0003 (100–300 ppm). By means of regulation system (nozzle), copper is continuously cast from the funnel into the crystallizer, from which a copper profile solidified at about 920 °C comes out. Guide rollers further drive the profile into the milling and cleaning machine that performs a surface treatment of the profile. This modified profile with the temperature ranging from 780 to 830 °C enters the rolling mill where it is thermoformed by gradual passing through the rolling heads. The first rolling mill forms the profile in the vertical direction and the second one in the horizontal. Other mill rotational systems roll alternately on a triangle – ring system, while an 8 mm wire comes out from the last rolling head [9].

The rolled wire with the temperature of about 500 °C is fed by guide rollers into the reduction tunnel where it is freed from the surface oxide layer in an intense reduction reaction. It is further cooled to about 40 °C in the cooling tunnel. After leaving the cooling tunnel, the wire is dried by compressed air and sprayed with protective wax. Such a waxed wire is wound in a steel safety cage on the prepared wooden pallets to form coils weighing 4–5 tons.

2.1. Surface oxides as a subject of improvement

In the literature, there are numerous data on the adhesion, growth rate of surface layers, properties of both forms of copper oxide, namely Copper (I) oxide (Cu_2O) and Copper (II) oxide (CuO) [1].

If both of these phases are present in the copper rods in a thick layer, they can be identified by their colour being either more reddish, and then it is Copper (I) oxide, or black. This is caused by Copper (II) oxide staining. However, it is much more difficult to characterize such thin continuous oxide layers on copper wires as they are often not visible to the naked eye. Although they are almost invisible, in the wire production, they may be as destructive as the thicker oxide layers.

2.2. Analysis of oxide layers

Determining oxide layer thickness on metals by means of electrolytic reduction dates back to the 1930s [12].

This method was initially used to study the layers of silver iodide, silver chloride, cuprous oxide, cuprous sulphide, and iron oxide with thickness ranging from 10 to 1000 Angströms (one Å is equal to 10^{-10} m, it means 10^{-1} nm). Angström is a unit that is not internationally recognized, thus it is not included in the SI system. However, for copper wire manufacturers, this measurement unit represents a part of a sector, an informal standard that is required by customer. For these reasons, we will prefer to use the Angström units rather than the traditional metric units defined in the SI system.

Method of measuring the surface oxide layer thickness on copper wire by means of electrolytic reduction consists in reducing carbon hydrogen ions in the electrolytic cell. A copper sample forms a cathode related to the inert anode and a constant DC current passes through the system. Surface potential of the cathode is measured by means of reference electrode during the layer reduction. During the reduction, the cathode potential remains relatively constant for each type of surface layer. In case of a mixture of oxides, there's primarily a reduction of the less stable Cu_2O. After the reduction of Cu_2O, voltage rises to reach the second phase, which corresponds to the reduction of CuO to copper. The end of the reaction is indicated by hydrogen gas evolution at the cathode. The length of each of the intervals between the inflection points of the tension diagram – the time is directly proportional to the amount of carbon present. Reaction at the cathode is:

$$Cu_2O + 2H + +2e^- \Longleftrightarrow 2Cu + H_2O \quad (1)$$

$$CuO + 2H^+ + 2e^- \Longleftrightarrow Cu + H_2O \quad (2)$$

This reduction process is shown schematically in Fig. 1 wherein is shown the hydrogen ions diffusion through the mixed copper oxides and the reduction of cupric oxide to copper at the interface of oxide and metal. After a complete reduction of Copper (I) oxide, it begins to reduce to Copper (II) oxide.

2.3. The apparatus

The experimental apparatus for the electrolytic reduction is shown in Fig. 2. After removal of grease and other non-metallic substances from the surface of copper, the sample is placed in a closed cell with a 0.1 M sodium carbonate Na_2CO_3 electrolyte, which is kept at a room temperature and continuously bubbled with argon to prevent reaction of the dissolved oxygen with the hydrogen gas.

A constant current passes through the platinum electrode, electrolyte and the sample. The surface potential of the sample as measured against the saturated calomel reference electrode is connected to a voltmeter with high internal resistance so that no observable current can pass through the measuring system. The surface potential of the sample is then recorded by a belt recorder.

2.4. Oxide layer thickness calculation

During the phased reduction of cuprous oxide and cupric oxide, the potential passes through two inflection points. Another inflection point at a higher voltage corresponds to copper sulphide. It is possible to measure the layers of any thickness, but first you need independent methods to identify the corrosion product. The

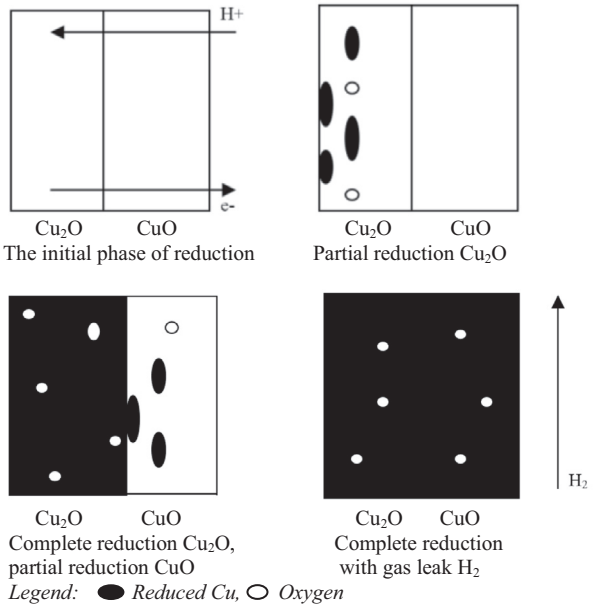

Fig. 1. Diffusion of hydrogen ions through the mixed oxides of copper [12].