

ANALYSIS OF CHEMICAL NICKEL PLATING PROCESS

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Nowadays the nickel electroplating process is commonly used for a variety of manufacturing applications. In general, electroplating improves a wide range of properties not inherently present in the base material. Nickel plating is a method which has many advantages i.e.: prevents corrosion, improves hardness and strength, increases resistance to wear as well as gives the aesthetic view of the products. Moreover, the nickel electroplating process in industrial applications is often used during joining or brazing. The applied coating increases the wettability of liquid brazing on the base material surface as well as improves the quality of the connection between different materials. The paper presents results concerning the analysis of setting for nickel electroplating process. Samples for investigation in the form of tubes with a variable diameter were prepared from austenitic stainless steel AMS 5510. The effect of nickel plating procedures and current parameters on the quality of the obtained coatings were analyzed. Uniformity and thickness of the applied layers were verified in randomly selected cross-section areas by microscopic observation. At least 100 measurements for each sample were performed. The optimized process parameters allowed saving 100 s in nickel-plating process, independent of the tube diameter. Obtained results have a significant importance in cost-savings and increased productivity of the nickel plating process.

Keywords: *nickel plating process, coatings, stainless steel, austenitic steel.*

It is well known that the most useful properties of many products depend on the microstructure and coatings applied on materials. The best results of preferred properties usually are achieved by proper selection of the materials and production technology. The manufacturing process of such products is usually multi-stage and depends on many factors. Moreover, materials during the exploitation are exposed to the different external influences i.e. mechanical, thermal or electrochemical [1]. Frequently among many factors affecting the destruction of materials one is a dominating. This process is independent of the material from which it is made. Usually the destruction mechanism propagates from the surface. One of the ways to prevent this kind of defects is applying a suitable surface layer – the protective coating [2]. Typically this process means permanent plating of the metal alloy or ceramic material which will isolate the material from the effects of e.g. aggressive environmental conditions. Therefore, nowadays the surface engineering is so important because it can significantly prevent such effects. This process can be applied for all kinds of materials [3–7]. Furthermore, through the application of various coatings the functionality can be increased, service life can be longer and operating costs reduced.

Currently there are many coating application techniques e.g. chemical, hot-dip galvanizing, thermal spraying, diffusion or electroplating. However, such methods, like physical vapour deposition, chemical vapour deposition or thermoreactive diffusion some have some disadvantages e.g. the high cost of apparatus and/or limit the relative shapes and/or sizes of metallic parts. Therefore, the electrochemical methods are more often chosen in industrial applications.

coating for such substrates, in most corrosive environments, is greater than the corrosion potential of the substrate. Therefore in such systems the nickel coating provides cathodic protection against corrosion of the substrate. Furthermore, the nickel coating is also applied before the brazing process. Nickel interlayer improves the brazing process, increases the wettability of liquid brazing on the base material surface and stops the creation of the reactive areas [15–17].

During electroplating the bath malfunction can be often met. This causes an objectionable decrease of the coating quality, reduction of the range of gloss, separation of significant amounts of gas or decrease of efficiency. There are many causes of bath malfunctioning or defective coating. Defective nickel layers are removed in the same way regardless of the nature of the coating. Removing the wrong applied coatings can be accomplished electrolytically by suspending objects as anodes in a sulfuric acid solution (VI) having a density of 1.74 g/cm^3 . During anodic process the current density usually is in the range $7 \dots 8 \text{ A/dm}^2$. Lead electrodes are used as cathodes. In order to protect steel against etching glycerine in an amount of $8 \dots 10 \text{ g/dm}^3$ is added to sulphuric acid (VI). Furthermore at high concentrations nickel can be very toxic [18–21]. According to the International Agency of Research on Cancer, exposure above $1 \text{ mg}\cdot\text{m}^3$ nickel soluble was evaluated as carcinogenic [22].

Materials and experimental procedure. The test was carried out on austenitic stainless steel AMS 5510. The chemical composition of this steel, according to the standard is the following (%): 0.08 C max; 0.25...1.00 Si; 2.0 Mn max; 0.04 P max; 0.03 S max; 17...19 Cr; 9...12 Ni; 0.7 Ti max; 0.1 N max; Fe bal. Material selected for analysis is commonly used in the aerospace applications, such as exhaust stacks, manifolds and ring collectors or constructing components for aircraft engine fuel. This type of stainless steel is characterized by high melting point and through titanium addition the resistance to intergranular corrosion.

Watt bath order of the electrochemical nickel plating process proceeds according to the scheme: I – degreasing the anode; III – degreasing the cathode; V – activation of the cathode; VII – nickel breakaway; IX – nickel sulphamic; II, IV, VI, VIII; X – municipal water. Individual blocks present the next stages of the nickel coating process. The standard full cycle consisted of ten treatments. In the first bath the process of degreasing the anode with an aqueous solution Slotoclean EL 210 was carried out. In every second bath the washing process in municipal water was performed. In the third bath degreasing of the cathode took place in the solution of Slotoclean EL 211. The next step was to activate the cathode by immersing the element in the solutions of Slotoclean Decasel 5, Slotonik M and water. The first compound is an inorganic salt mixture containing fluorides, while the second is supposed to apply the glossy nickel plating. Then, bath VII contains a mixture of nickel chloride (II) hexahydrate, chemically pure hydrochloric acid and deionized water. The last process is an electrochemical bath in sulphamate nickel obtained by mixing 60% nickel sulphamate, nickel chloride (II) hexahydrate, boric acid and deionized water.

The samples depending of the tube diameter were subjected to nickel plating process at different current settings, as presented in Table 1. The established new current parameters had to be as close as possible to the approved production standards accepted by the recipients. For the largest diameter tube, the effect of omitting three stages of washing on the quality and thickness of the resulting nickel coating was also analyzed (sample № 7). After the nickel plating process, samples were subjected to microscopic observation. The samples were cut out perpendicular to the direction of applying a layer (tube axis). A polishing process was finishing by using diamond suspension with $3 \mu\text{m}$ particle size. The sample was etched with the Kallings reagent: CuCl_2 , hydrochloric acid and ethyl alcohol in the ratio of 5 g – 100 ml – 100 ml, respectively.

Table 1. The current settings of nickel plating process depending of the tube diameter

| Symbol of sample | Tube diameter, mm | Current setting, A | |
|------------------|-------------------|--------------------|-----|
| | | used as standard | new |
| 1 | 9.5 | 0.6 | – |
| 2 | | – | 0.8 |
| 3 | 19.0 | 1.2 | – |
| 4 | | – | 1.6 |
| 5 | 25.4 | 1.5 | – |
| 6 | | – | 2.2 |
| 7* | | 1.5 | – |

* – lack of washing process, the IV, VI and VIII bath in the nickel coating process presented as shown above.

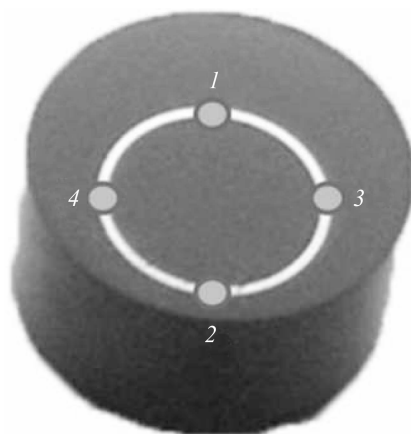


Fig. 2. Sample № 5 showing four randomly selected areas shifted by 90° for nickel thickness measurements.

Microscope Nikon Eclipse LV150 was used to perform a microscopic observation. For each sample the measurements were taken at four randomly selected areas. Their mutual orientation relative to the tube axis was shifted by 90° (Fig. 2). For each sample about 100 measurements were made (at least 25 measurements in each of the four areas). The maximum, minimum and average thicknesses with standard deviation of nickel coating were evaluated.

Results. Fig. 3 presents a representative microstructure of sample № 1, which shows the visible applied nickel coating. It was observed that the nickel layer was spread evenly on the stainless steel surface around the circumference of the tube. Good coherence of the coating with stainless steel was obtained for all samples tested regardless of the tube diameter and

applied current parameters. On the basis of microstructure analysis, it was also found that the elimination of three stages of the surface rinsing in the coating process (sample № 7) did not result in any negative impact on the quality of adhesion of the resulting nickel coating. Moreover, such a procedure saved about 100 s in the process of its production, which is important because it allows increasing the production efficiency. All analyzed coatings had the thickness required by the AMS 2403 standard.

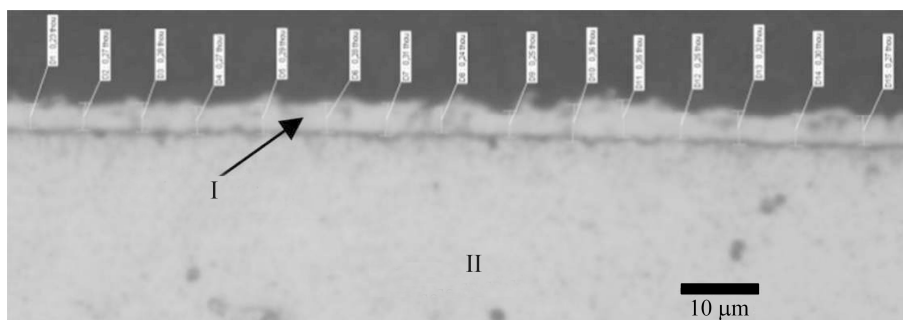


Fig. 3. Representative microstructure of sample № 1 with thickness measurements of the nickel layer: I – nickel layer; II – base material.

The maximum and minimum thicknesses of their nickel coatings in four randomly selected areas on the circumference of the tubes (Fig. 2), depending on their diameter and applied current parameters, are summarized and presented in Table 2. The measurements allowed confirming the uniformity of thickness of superimposed nickel layers. The smallest coating thickness was measured for sample № 5, while the thickest nickel layer was recorded for sample № 7.

Table 2. The maximum and minimum nickel thickness measured in four randomly selected areas on the circumference of the tube schematically shown in Fig. 2

| | Area 1 | Area 2 | Area 3 | Area 4 |
|-----------------------------------|--------|--------|--------|--------|
| | μm | | | |
| Sample № 1 | | | | |
| Maximum thickness of nickel layer | 7.6 | 5.4 | 6.1 | 6.6 |
| Minimum thickness of nickel layer | 4.2 | 4.0 | 3.7 | 4.2 |
| Sample № 2 | | | | |
| Maximum thickness of nickel layer | 7.4 | 5.8 | 6.3 | 6.8 |
| Minimum thickness of nickel layer | 4.3 | 4.1 | 3.8 | 4.6 |
| Sample № 3 | | | | |
| Maximum thickness of nickel layer | 7.5 | 7.1 | 8.1 | 6.1 |
| Minimum thickness of nickel layer | 3.5 | 3.3 | 4.3 | 4.3 |
| Sample № 4 | | | | |
| Maximum thickness of nickel layer | 7.3 | 6.8 | 7.2 | 8.5 |
| Minimum thickness of nickel layer | 4.4 | 4.1 | 3.9 | 4.5 |
| Sample № 5 | | | | |
| Maximum thickness of nickel layer | 4.3 | 5.8 | 6.9 | 7.4 |
| Minimum thickness of nickel layer | 2.7 | 3.3 | 2.8 | 4.6 |
| Sample № 6 | | | | |
| Maximum thickness of nickel layer | 5.5 | 6.2 | 7.1 | 6.8 |
| Minimum thickness of nickel layer | 3.4 | 3.9 | 4.4 | 4.3 |
| Sample № 7 | | | | |
| Maximum thickness of nickel layer | 7.6 | 7.1 | 8.9 | 6.7 |
| Minimum thickness of nickel layer | 3.8 | 4.2 | 4.3 | 4.3 |

The mean values of more than 100 measurements for each of the samples, allowing a global comparison of the thickness of the nickel layers, depending on the tube diameter and the applied procedures are presented in Fig. 4.

Based on the results obtained it was found that the proposed new (higher) current parameters allowed the slightly thicker nickel coatings relative to the value which was measured for the samples produced at the lower current parameters. The effect was independent of the applied tube diameter. Furthermore, the thickest nickel layer characterized by the procedure of sample preparation in addition to the higher current set points included omission of the three washing steps.

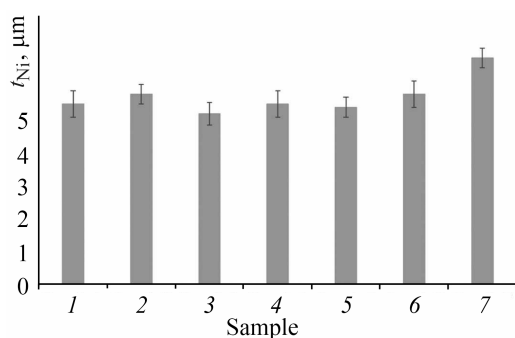


Fig. 4. Mean thickness of nickel coating depending on the tube diameter and applied technological parameters.

CONCLUSIONS

The results demonstrated that both the current parameters and the number of applied stages of the process have a significant influence on the thickness of applied nickel coatings. All of the coatings produced were characterized by a good consistency with the base and met the requirements of the standard. It was shown that the increase in current settings made it possible to produce the thicker nickel layers. In addition the possibility of such choice of the nickel-to-stainless steel processing

technology was demonstrated and it was possible to save up to 100 s of production time without worsening the quality of the applied coating. This solution allowed significant cost savings and increased productivity.

РЕЗЮМЕ. Нікелювання часто використовують у різних виробничих процесах для поліпшення властивостей основного металу. Цей метод має багато переваг, зокрема, попереджає корозію, підвищує твердість та міцність, збільшує зносотривкість та поліпшує естетичний вигляд виробів. Крім того, його часто застосовують під час зварювання та паяння. Нанесений покрив краще змочується, а також з'єднання різних матеріалів якісніші. Зразки виготовлено у формі трубок різного діаметра із аустенітної сталі ASM 5510. Проаналізовано вплив нікелювання та його параметрів на якість покривів. Мікроскопічними дослідженнями підтверджено рівномірність нанесення покривів у випадково вибраних ділянках поперечного перерізу. Виконано близько 100 вимірювань. Отримані результати мають важливе значення для рентабельності та підвищення продуктивності нікелювання.

РЕЗЮМЕ. Никелирование часто используют в различных производственных процессах для улучшения свойств основного металла. Этот метод имеет много преимуществ, в частности, предупреждает коррозию, повышает твердость и прочность, увеличивает износостойкость и улучшает эстетичный вид изделий. Кроме того, его часто применяют при сварке и пайке. Нанесенные покрытия лучше смачиваются, а также соединения различных материалов более качественные. Образцы изготовлены в форме трубок различного диаметра из аустенитной стали ASM 5510. Проанализировано влияние никелирования и его параметров на качество покрытий. Микроскопическими исследованиями подтверждена равномерность нанесения покрытий в случайно выбранных участках поперечного сечения. Выполнено около 100 измерений. Полученные результаты имеют большое значение для рентабельности и повышения производительности никелирования.

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Received 21.11.2017