COMPLEX ANALYSIS OF THE QUALITY OF THE NICKEL LAYER OF SCREWS

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Abstract. Chemical nickel plating is one of the functional coatings that extend the life of coated components. This type of coating has wide application, enables coated complex shape components, holes and edges. The procedure electro less nickel has several steps and consists of degreasing, rinsing, pickling (eg. Hydrochloric acid), rinsing, and insert into the nickel bath. Nickel coatings do not form pure nickel, but it is a phase of NiP, NiB, NiCoP, etc. The thickness and quality of the layer depends on the purity of the object, and the time of stamina the concentration in the bath. Follow by removal from the bath, rinsing and drying. The coating has a hardness of about 550 HV, given that it is an alloy coating nickel-phosphorus can be achieved by heat treatment hardness of 1000 HV. Chemical nickel coating is universal coating that their properties coated component simultaneously provides corrosion resistance, abrasion resistance and good sliding properties. The coatings exhibit a constant thickness across the plating surface, regardless of its geometry. In most common applications are used in coating thickness 25 μm, a thickness of 25 to 80 μm are used for the parts with a higher the risks to corrosion and the thickness of 80 μm for the repair of wear parts and the like. The aim of the paper was to analyse the quality of the nickel layer of screws of free-cutting steel, which are part of the fuel system of cars. Nickel coating of steel parts is carried out with the aim of extending the life of components. Any degradation of the layer results in deterioration of not only of screws, but also the whole. Layers will be evaluated using a microscopic methods and hardness measurements. The results will determine the causes of changes in the quality of the nickel layer and a determined of general and specific recommendations for the formation of the nickel layer.

Keywords: analysis, corrosion, screw, electroless nickel plating, nonconforming product.

Introduction

Corrosion is spontaneous and progressively extending action, leading to undesirable and harmful disruption solids chemical and electrochemical effects of the surrounding environment. Form by the interaction of the material and the environment. In technical practice it is undesirable corrosion factor. It is estimated that the economies of industrialized countries causes corrosion losses of 4-5% of gross domestic product [1-3].

Chemical nickel plating is one of the functional coating. Structure of the coating consists of spherical particles of Ni. Performed an alloy of Ni + P. Autocatalytically precipitated nickel allows to plate complex shape components, holes and edges, the precipitated nickel layer are on the entire surface a uniform, thus longer then requires no additional grinding. Chemical nickel plating partly replaced by hard chrome plating technology, since the heat treatment increases the hardness. An advantage of electroless nickel in comparison with galvanic processes are a large depth the effectiveness of the bath, which is important e.g. in plating various cavities and rugged surfaces. The disadvantage is a lower elimination rate compared with galvanic processes. Gradually it was developed for a variety of special applications, many different types of plating bath [4-6].

Nickel plating has several steps. To prepare component, this will be applied Ni layer. That is: degreasing, rinsing, pickling (eg. HCl), rinsing and placing in the prepared plating bath. The Ni bath is carried out in chosen time interval, segregation NiP alloy is to the surface of part. As the main reducing agents are used the bath like sodium hypophosphite, sodium borohydride or dimethylamine. Nickel coating is not excreted as pure nickel, but it is an alloy NiP, NiB, NiCoP, etc. The most widespread and most used is technically NiP alloy, which constitutes 98 % chemically excluded nickel deposits. The thickness and quality of the layer depends on the purity of course, the concentration and the time of holding in the bath. Follow by removal from the bath, rinsing and drying. Coating after exclusion has a hardness of about 550 HV, whereas it is a nickel-phosphor coating , can be applied heat treatment (350 °C, 2h), after is possible to obtain hardness of 1000 HV. Chemical nickel coating is universal coating with properties of coated component simultaneously provides corrosion resistance, abrasion resistance, good sliding properties and attractive appearance. Because of these advantages are becoming increasingly widespread in various parts, electrical engineering, mechanical engineering, petroleum, gas and chemical industry in the production of precision components for the automotive, aerospace, nuclear engineering etc. Electroless nickel plating is performed on parts made of materials such as steel, cast iron, copper and copper alloys, aluminium and aluminium alloys. [4, 5]

For a comprehensive system approach is important in assessing the internal and external factors that determine the final status of parts. Internal factors include, for example material - chemical composition, structure, inclusions, the method for manufacturing the blank - forming, casting, machining, ... External factors such as load, wear, corrosion stress, creep, ... In the analysis of non-conforming product or its degradation it is necessary to evaluate these factors and subsequently to initiate analysis using available methods. The most commonly used methods are light and electron microscopy and hardness tests [6-9].

The aim of the paper was to analyse the quality of the nickel layer of screws of free-cutting steel, which are part of the fuel system of cars. Nickel coating of steel parts is carried out with the aim of extending the life of components. Any degradation of the layer results in deterioration of not only of screws, but also the whole. Layers will be evaluated using a microscopic methods and hardness measurements. The results will determine the causes of changes in the quality of the nickel layer and a determined of general and specific recommendations for the formation of the nickel layer.

Materials and methods

As an experimental samples were used bolt corrosive attack the internal cavity of the screws (cavity for hex wrench), which was reflected in the final manufacturing operation and that it was nickel plated, in the absence of external load. Production technologies for the production of bolts were: drilling and subsequent compression screw cavity. Screws were part of the fuel system of machinery and hand tools designed for the construction and forestry industries. The components are made of drawn steel with a diameter 22h9 mm. Material of screws is steel grade 11 109 according to CSN 42 001 standard.

To improve the properties and increase protection against corrosion attack was formed on the components of the protective layer of chemical nickel plating technology. The thickness of the protective layer was set at manufacture to 20 microns. After the final manufacturing operation to components occurred corrosion attack in the cone cavity bolt, Fig. 1b, c.

The analysis of the surface protective layer by a scanning electron microscope TESCAN VEGA 3, see Fig. 1. Used markings are as follows.

- Sample A – the ample with Ni surface protection without corrosion attack.
- Sample B – the sample with Ni protective surface with the corrosive attack (cone).
- Sample C – the ample with Ni protective surface with the corrosive attack (cone), but without corrosion attack (wall).

Sample A *Sample B* *Sample C*

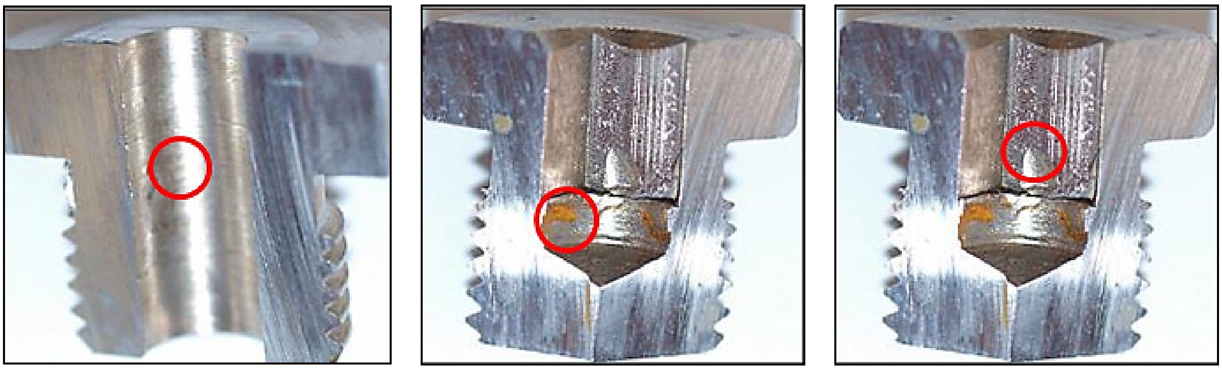


Fig. 1. **Analysed samples**

Sample A was a quality piece with Ni surface protection without corrosion attack, Fig. 2a. It was obvious that on component were showed no deformation on the surface, no corrosion and other visible damage. This component was selected to serve as a standard. The quality and character formed Ni layer was further observed by electron microscopy. Ni surface layer has characterized morphology, which corresponds to knowledge from the literature.

Macroscopic analysis, mag. 50 x *REM*

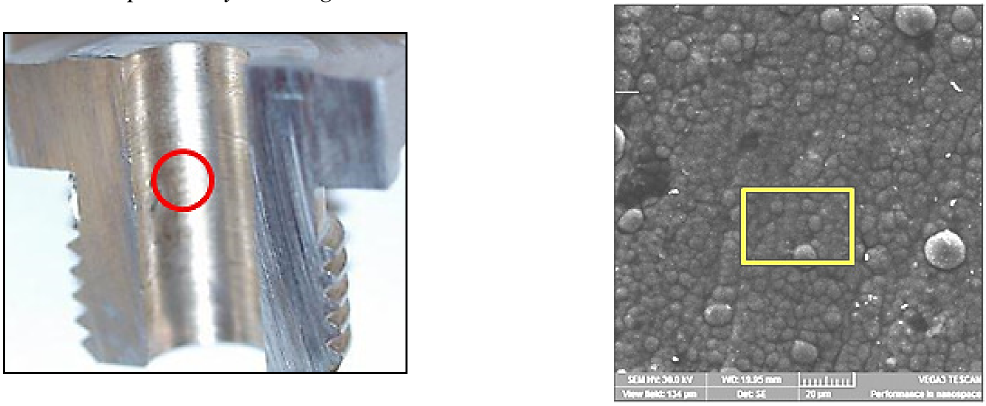


Fig. 2. **Sample A**

The Ni layer is solid, uninterrupted surface; Ni particles are uniform in size and distribution, Fig. 2b. From macroscopic and microscopic observation it can be seen that the surface protective layer of the same Ni component exhibits no mechanical damage, deformation and corrosion attack. The surface morphology of the protective Ni layer was completely homogeneous. Microscopic analysis confirmed the result of a macroscopic analysis, and that is that of the surface of the same part is free of surface deformations and corrosion attack. On this sample B was already by the visual analysis showed substantial corrosion attack and this in the lower part of the cavity of the screw (Fig. 3a).

Macroscopic analysis, mag. 50 x *REM*

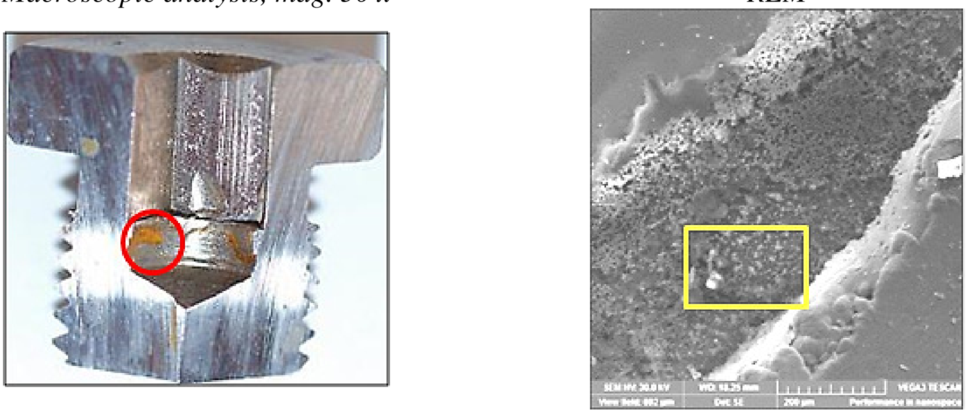


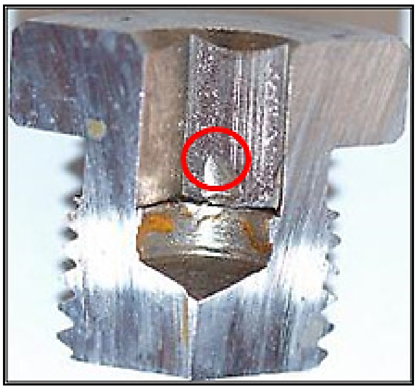
Fig. 3. **Sample B**

Microscopic analysis in detail revealed a heterogeneous morphology of the Ni layer in the transition region between the wall and the cone of screw. Based on Fig. 3b it can be concluded that the corrosion attack formed on the edge of two different production technologies of the cavity, and hence the drilling and subsequent pressing. Predominantly pressing technology increases the reinforcement on the walls and on the edge of the cone bolt in the cavity. Reinforcement of material can cause a change in surface roughness component before the nickeling. On the basis of literary observations, we note that the surface roughness is one of parameters influencing the process of creating a protective layer. Changing the surface roughness, namely, increase of surface roughness, is a positive factor for creating quality nickel protective layer, which was formed by chemical nickel plating. Despite this finding it is in the surveyed area marked change of surface morphology and the cumulation of unwanted oxide particles.

The third sample was specimen C. The analysis was performed on the cavity wall, where a considerable deformation of the wall surface (Fig. 4).

The analysed area outside is the occurrence of deformation of the surface, Fig. 4b, the surface morphology similar to the morphology of the surface of the sample A. In the area of significant plastic deformation, which showed formation of deformation, is changed the morphology, Fig. 4b. This has been a change in connection protective layer, which also indicates a change in thickness. Deformation on the wall cavities allow most likely caused by the movement of the tool (press). During pressing occurred in part of the wall of the cavity to reinforce the material. Reinforce the material influences the diffusion processes during the nickeling surface of the screw. With regard to the heterogeneity of the surface, it offers the possibility to give a new area of corrosion products.

Macroscopic analysis, mag. 50x



REM

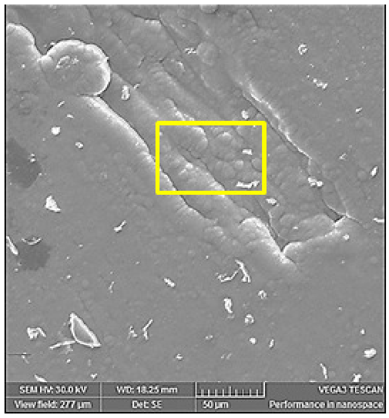


Fig. 4. **Sample C**

Quantitative evaluation of the surface layers

A part of the scanning electron microscope TESCAN VEGA 3 is an EDX analyzer BRUKER, Esprit 1.6, which was used for determining the chemical composition of selected areas of the surface layer. The samples A, B, C (Fig. 2b, 3b, 4b) by a surface EDX analysis were used. The results of EDX analysis are shown in Table 1. In Table 1 are the values of the most watched chemical elements, namely nickel, phosphorus, carbon, oxygen and iron. For nonconforming parts we could see decline element concentrations of nickel and phosphorus and growth elements like carbon, oxygen and iron. Results of the analysis showed different morphology and significant differences in the chemical composition of Ni protective layer.

Table 1

The results of EDX analysis of samples

Sample A		Sample B		Sample C	
element	**wt %**	**element**	**wt %**	**element**	**wt %**
Ni	79.32	C	11.65	C	5.94
P	7.97	O	26.66	O	0.24
C	8.62	P	0.79	P	9.65
O	0.70	Fe	30.8	Fe	1.31
B	1.26	Ni	27.76	Ni	79.49
N	0.58	N	1.06	N	1.43

Hardness measurements of Ni layers

The hardness of the surface protective layer is one of the parameters determining the service life of screws. Vickers hardness test (CSN EN ISO 6507-1) was performed at 400x magnification samples with the time load 12 s. Hardness was evaluated in all three samples was performed five measurements, Table 2.

Table 2

The hardness of the surface to be analyzed according to CSN ISO 6507-1

Sample	HV1	
	ϕ	σ
A	387	7.45
B	331	8.91
C	350	8.47

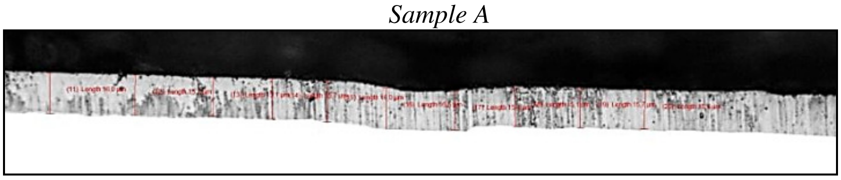
Note: ϕ - average, σ - standard deviation

Relatively high dispersion of the measured values is due to the condition of the analyzed surface, when there are differences in the range (30-50) HV. Changes in properties are caused by changing the chemical composition and morphology of the protective layers. Samples B and C show lower resistance to mechanical stress, that can be caused by degradation of the surface protective layer to corrosive attack, undesirable diffusion processes of manufacturing hexagonal hollow of the screw or unstable nickel bath. On the basis of theoretical knowledge hardness of the layer is set to a value of 500 HV. The values determined hardness of the surface layer did not reach the prescribed value. This low hardness protective surface component is not guarantee resistance to abrasion and mechanical stress.

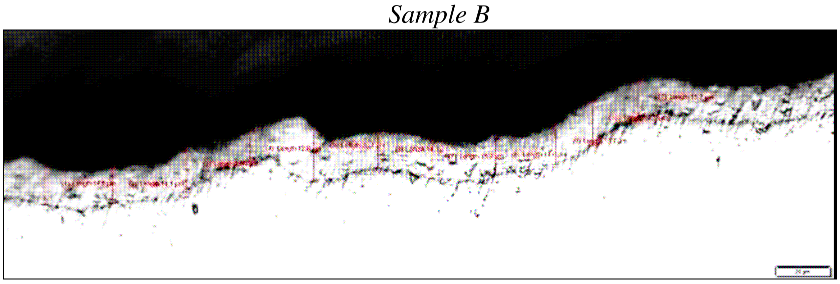
Evaluation of layer thickness

Microscopic evaluations of the thickness of the nickel protective layer of the screw were made on microscope OLYMPUS BX51M. On the basis of theoretical knowledge, the thickness of the protective layer should have a minimum value of 20 microns.

Sample A



Sample B



Sample C

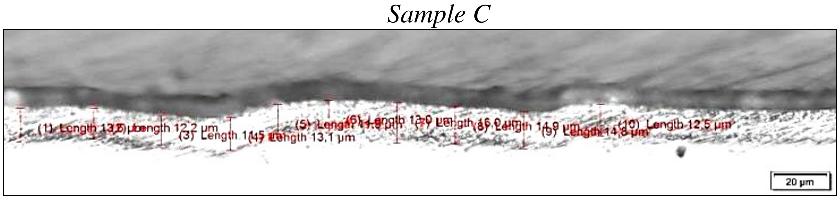


Fig. 5. **Evaluation of layer thickness**

In Fig. 5 it is possible to observe the different condition of the protective layer. Compared to Sample A, for samples B and C is seen the uneven thickness and considerable complexity surface layer. The ruggedness may be due to the state of the component surface, which is influenced by the way of the screw manufacturing.

The basic characteristics of chemical nickeling are the uniformity of the thickness of the protective layer. To remove the unevenness of the protective layer is crucial to respect the principles by preparing of Ni protective bath In order to assess course of the protective layer was carried out on each sample eleven measurements. The results are shown in Tab. 3.

Table 3

The Evaluation of layer thickness

Sample	**Thickness, µm**	
	ϕ	σ
A	15.67	0.45
B	15.90	2.40
C	13.36	1.47

The mean value of all samples analyzed exceeds the value of 16 microns compared to the specified value of 20 microns. Failure to follow the layer thickness can be reduced abrasion resistance and mechanical stress components. How thickness of a protective layer determines the resistance to mechanical stress, abrasion, corrosion attack and other potential degradation of the material, it can be assumed that for the experimental samples will shorten component life.

Conclusions

On the results of the analysis and measurements can be stated the following conclusions:

- Based on analysis by electron microscopy has been demonstrated the typical morphology of the protective layer of quality piece in the form of spherical particles of nickel. The results of EDX analysis showed and quantify concentrations of chemical elements contained in the protective layers.
- For a sample of the nonconforming piece was analyzing by electron microscopy showed the morphology of the protective layer in the form of pads, bead-like formations and flakes. The results of EDX analysis showed and quantified different concentrations of chemical elements contained in the protective layer of Ni, which are different from the results of EDX analysis of conforming piece. Vickers hardness test has shown that the highest hardness of the layer was detected at the conforming piece, compared to a significantly lower hardness nonconforming parts with corrosive attack. Microscopic analysis was performed measuring the thickness of the protective layer, which showed topography and uneven of Ni protective layer of non-conforming parts.
- Based on the results we can say that the cause of corrosion damage connecting parts (screws), was the failure to observe technological process of preparing the protective of Ni bath. The basic parameters in the preparation of the bath are the prescribed concentration and chemical composition, temperature and time interval of the action of the coating bath. Compliance the process of preparing the bath allows the formation of the comfortable protective of Ni layer, which acts as a protection against abrasion, mechanical stress, formation of corrosion and creates shiny appearance parts.

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