

# ELECTROLYTIC NICKEL-PLATING FOR SURFACE PROTECTION AGAINST HIGH-TEMPERATURE OXIDATION AND DECARBURIZATION

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### Abstract

In steel research, as well as in the manufacture of semi-finished metallurgical products and finished formed products, such as forged parts, the material is often heated to high temperatures which leads to surface oxidation and decarburization. Material-technological modelling involves the use of small-size specimens which are treated according to schedules identical to those in real-world processing. From these treated specimens, test bars are made for various mechanical tests. If severe decarburization occurs, the affected surface layer may give rise to substantially different results of mechanical testing than in a thermomechanically treated material with an intact surface. Therefore, protective barriers were sought by which formation of scale on the surface of test specimens, and decarburization in subsurface layers could be prevented. One of the available options involves depositing a coating of 10-15 micrometre thickness by electrolytic nickel-plating. By this simple and readily-available technique, very good protection was obtained for specimens exposed to high temperatures.

**Keywords:** Surface protection, high-temperature oxidation, decarburization, Ni coating, material - technological modelling

#### INTRODUCTION

Material-technological modelling is a powerful method of investigation of real-world thermomechanical treatment processes [2, 4, 7, 10]. The quality of its results, depends on accurately reproducing the conditions encountered by the material in the real-world process. These conditions predominantly involve strain, stress and temperature. It should be noted, however, that the surface of the specimen and its interaction with the environment play an important role as well. Quite often, the simulation specimen represents a very small portion of the entire real-world product or structure. This portion can sometimes be located on the product surface but often it is found within the interior, with no means of interacting with the environment [2, 6]. For this reason, one must make sure that the surface layer of the specimen is not affected by the environment, e.g. by oxidation and decarburization [1] which alter material properties but cannot take place within the interior of a real-world product [2]. Another example is the press-hardening operation on sheet parts [4, 5]. In such case, the physical simulation substitutes the sheet-die contact-cooling mechanism by a jet of mixed water and air [3]. By this means, the temperature conditions are simulated faithfully but the operator needs to ensure that the surface of the specimen is not affected by the jet.

This was the motivation for the trials of electrolytic nickel-plating as a means of surface protection of specimens. Nickel is effective in preventing scale formation but can form solid solution with carbon [11, 12]. To experimentally verify this solution, specimens with and without the protective coating should be exposed to high temperature in air for various time periods. After quenching, the sub-surface layer of the test bars and the effectiveness of the protection provided by the nickel coating were examined.



#### 1. EXPERIMENTAL

The trials of the protective nickel coating were carried out on specimens of low-alloy steel with 0.28 % C. Their

thickness was 1.46 mm (**Figure 1**). One side of the specimens was coated with a nickel layer of 10-15  $\mu$ m thickness. These specimens were subjected to temperatures of 940 °C and 1200 °C for various time intervals. Afterwards, they were quenched to ambient temperature in a process that corresponded to press-hardening (**Figure 2**). The resulting microstructures were examined.

The thermal exposure took place in a thermomechanical simulator. This is a machine which can impose thermal and mechanical loads on specimens. [3]. The specimens are heated by electrical resistance using high-frequency current. The process is controlled by a feedback system with a four-stage regulator. The specimen temperature is regulated at 5 kHz frequency. Gradients of up to 200 °C/s can be achieved by the system, depending on the type of material and specimen dimensions. The specimen temperature is detected by means of an attached thermocouple. Cooling is provided by an in-house-designed module. The coolant is a variable-ratio air-water mixture. The cooling intensity is regulated by a feedback loop, i.e. in response to the instantaneous temperature of the specimen. In the current configuration, the maximum cooling rate is 130 °C/s, depending on the specimen size and instantaneous temperature. The cooling intensity is a non-linear function of the temperature of the object being cooled. Both systems communicate and interact with each other in order to provide good agreement between the actual and the desired specimen temperature.



Figure 1 Specimen dimensions







#### 2. RESULTS AND DISCUSSION

The first step in mapping the impact of the exposure on decarburization processes was microstructure examination after nickel coating deposition (**Figure 3**, **Figure 4**). The nickel layer was found to adhere to the prior ground surface of the specimen very well. The grinding caused visible but mild microstructural effects in a 5  $\mu$ m-deep layer. Deeper beneath the surface, the microstructure consisted of a mixture of ferrite and pearlite in which the mean size of ferrite grains was 5  $\mu$ m (**Figure 5**).

The microstructure within the interior of the specimen was virtually the same as the one just described, except for slightly larger grains. Specimens in this initial condition were brought to the desired temperature and quenched immediately after the exposure. The purpose was to obtain hardening microstructure and seek the differences between sub-surface layers beneath the nickel coating and the interior which had not been affected by the diffusion process. In case of a drop in carbon level, one could expect ferrite grains to be found in martensite. This was not the case upon the exposure at 940 °C (**Figure 6**). Neither the 100-second exposure (**Figure 6**), nor the longer 700-second one (**Figure 7**) had any effect on the microstructure in the sub-surface layer. The nickel coating thus perfectly fulfilled the requirements for the protection against surface decarburization.

Its effectiveness at 940 °C is also evidenced by the comparison with the reference surface of the specimen. It had no protective coating and was exposed under identical conditions. Metallographic observation revealed that the microstructure of its sub-surface layer had changed markedly. Ferrite grains within the martensitic matrix could be found down to the depth of at least 25  $\mu$ m (**Figure 8**). In fact, no martensite at all had formed in the 5- $\mu$ m sub-surface layer (**Figure 9**). Microstructural changes were visible down to 50  $\mu$ m depth, most notably between martensite particles, i.e. in the locations of prior austenite grain boundaries.

Upon thermal exposure at 1200 °C, the coating fails to perform its function, as opposed to the exposure at 940 °C. Diffusion processes lead to high-temperature oxidation even underneath the coating (**Figure 10**). The oxides cause delamination and fragmentation of the coating, which accelerates the degradation of the steel surface. These oxides comprise wustite, magnetite, and hematite. Their formation is also accompanied by decarburization down to 200- $\mu$ m depth. This means that although the coating slows down surface degradation, it is not able to avert it.



Figure 3 Initial ferritic-pearlitic microstructure beneath the nickel coating



Figure 4 Detail of the nickel coating-steel interface where the grinding-induced deformation of steel is visible





Figure 5 Initial microstructure in the centre of the sheet feedstock



Figure 7 Fully martensitic microstructure at the Ni coating-steel interface after the extended 700second exposure at 940 °C



Figure 9 Detail of ferrite grains in the decarburized plain surface without the Ni barrier upon exposure at 940 °C



Figure 6 Perfect performance of the Ni barrier during 100-second exposure at 940 °C



Figure 8 Decarburization of the plain surface without the Ni barrier upon exposure at 940 °C



Figure 10 Degradation of the Ni coating, decarburization and scale resulting from the exposure at 1200 °C



### 3. CONCLUSION

This investigation has shown that electrolytic nickel coating of  $10-15 \,\mu\text{m}$  thickness provides an adequate protective barrier against sub-surface decarburization and surface oxidation in steel specimens. This barrier is effective at lower hot-forming temperatures. Upon exposure at 940 °C, no microstructure degradation in surface or sub-surface layers was noted.

It has thus been documented that electrolytic nickel-plating can be employed for obtaining a barrier usable in hot working of steel sheet during material-technological modelling. The coating is effective across the entire range of ordinary process parameters which correspond to press hardening. In addition, such a coating provides constant conditions during heating and cooling of specimens in a thermomechanical simulator. This aspect is very important to achieving exact temperature profiles which must be in the best possible agreement with temperature profiles in real-world processes.

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