

Formation of borohydride-reduced nickel–boron coatings on various steel substrates



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ARTICLE INFO

Article history:

Received 8 September 2015

Received in revised form 23 October 2015

Accepted 27 October 2015

Available online 1 November 2015

Keywords:

Electroless nickel–boron

Initiation

Roughness

Plating rate

Substrate

Steel

ABSTRACT

Electroless nickel–boron coatings are widely used in industrial on various substrates: ferrous and non-ferrous alloys mainly but also in some cases non-metallic materials. However, their growth process is still not fully understood and the influence of the nature of the substrate on this process is completely unknown.

The formation of electroless nickel–boron was observed on five ferrous alloys: a mild steel, a high carbon unalloyed steel, a cryogenic steel (that contains 9 wt.% nickel), an austenitic stainless steel and an austeno-ferritic (duplex) stainless steel.

Nickel–boron films were prepared by electroless deposition, using sodium borohydride as a reducing agent. Samples were immersed in a plating bath for times ranging from 5 s to 60 min. The influence of the nature of the substrate on the initial deposition of the coatings was investigated in detail: the initiation mechanism was identified for all substrates and it was found to be related to catalytic oxidation of the reducing agent rather than to a displacement process.

The delay before initiation was influenced by the nickel content of the coating and by a high number of grain boundaries.

In all cases, the plating rate varied with plating time, with a slower period during the first 10 min that corresponds to morphological modification of the coating.

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1. Introduction

Electroless plating is a surface engineering process that allows the deposition of metal–metalloid alloys on various kinds of substrates, including, but not limited to, metals [1–3]. The first successful application of the method was carried out in 1946 by Brenner and Riddel who plated the interior surface of tubes with a nickel–phosphorous alloys [4]. Some year later, Schlesinger discovered the borohydride ion, leading to the development of electroless nickel–boron coatings, obtained with that chemical as the reducing agent [5,6].

The principle of electroless plating is relatively simple: metallic ions placed in aqueous solution are reduced by a chemical agent, without the need for an external current source. The bath is engineered to ensure that the reduced metal deposits on the substrate and not on other surfaces by the addition of stabilizing agents that act as catalytic regulators. Metalloids from the

reducing agent are co-deposited with the main metal (in the present case nickel) as are, if they are used, heavy metals salts used as stabilizer [7,8].

The spontaneous and catalytic nature of the plating process has great influence on the formation of electroless nickel coatings, including nickel–boron, and several authors have investigated the initiation and/or growth of the coating in the last few years [9–18]. However, knowledge of the influence of the substrate state and its nature is still very limited, because most authors work on a single substrate with a proprietary plating process that makes comparison difficult. Nevertheless, it has been shown that the surface state of the substrate influences the plating process, but also the coating's properties [19].

In previous work, we investigated the formation of electroless nickel–boron coatings on mild steel and the influence of the substrate roughness on the beginning of the process [9,10]. The aim of this study is to investigate the influence of the substrate nature (chemistry and structure) on the formation of the electroless nickel–boron coating. This was carried out only on ferrous substrates, in order to keep sufficiently comparable materials.

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Table 1
Chemistry of the substrates used for the study.

Composition (wt.%)	Mild steel St 37	Low alloy C steel AISI 1070	Cryogenic steel ASTM A353	Austenitic stainless steel AISI 304	Austeno-ferritic stainless steel ASTM A182 (grade F55)
C	0.17	0.65–0.75	<0.13	<0.08	<0.03
Ni			8.5–9.5	8–10.5	6–8
Cr				18–20	24–26
Mo	0.009				3–4
Mn	0.2–0.5	0.6–0.9	<0.9	<2	<1
Cu					0.5–1
W					0.5–1
N					0.2–0.3
Si	0.3		0.15–0.4	<1	<1
P	0.05	<0.04	<0.035	<0.045	<0.03
S	0.05	<0.05	<0.035	<0.03	<0.01
Fe	Balance	Balance	Balance	Balance	Balance

2. Experimental details

2.1. Substrate selection

It is not easy to study the influence of substrate nature: if the substrates used in a comparative study are too dissimilar, it is impossible to identify which features of the substrate are the most influent in the deposition process. For example, roughness influences the initiation of the coating and plating begins earlier on rougher substrates [9]. Moreover, some substrates, which are less reactive than others, require specific pre-treatments before electroless plating [20–23]. It is thus nearly impossible to compare them with other group of substrates that do not require those pre-treatments.

For those reasons, this study was limited to ferrous substrates that can be prepared with the same activation treatment. The roughness was also kept similar for all samples.

The alloys that were selected are (i) St 37 low carbon steel (ferritic steel), which is a very simple alloy, nearly monophased and containing only a small amount of carbon; (ii) ASTM A353 steel, which is a perlito-ferritic low alloy steel, close to the eutectoid chemistry. That substrate was chosen to observe the influence of carbon content (and structural modification); (iii) AISI 1070 cryogenic steel, which contains 9 wt.% Ni and has a martensitic microstructure; (iv) AISI 304 stainless steel (austenitic stainless

steel); (v) ASTM A182 (grade F55) (austeno-ferritic duplex stainless steel). The chemistry of all alloys is shown in Table 1.

Those alloys were selected because they present a variety of structures (fully ferritic, fully martensitic, fully austenitic, austeno-ferritic and austeno-perlitic). High alloy steels (stainless steels and cryogenic steel) were chosen in a way that various structures could be obtained with the smallest possible variation in nickel content (all three alloys contain between 6 and 10.5 wt.% nickel).

2.2. Sample preparation

The size and shape of samples varied with the nature of the alloy, depending on their availability and machinability: blocks, rods and plates were used. To ensure a similar roughness for all substrates, samples were prepared by grinding up to 4000 grade SiC paper. The roughness was then checked and only samples with a roughness (Ra) of $0.15 \pm 0.03 \mu\text{m}$ were used for the study.

After grinding, the samples were degreased with acetone and submitted to acid etching (activation) in 30 vol.% HCl for 1 to 5 min, depending of the alloy, followed by a rinse in flowing deionized water and direct immersion in the nickel–boron plating bath. The variations in sample size were taken into account during the plating process and a bath load of $25 \text{ cm}^2 \text{ l}^{-1}$, similar to the one used in other studies of our group [7,8], was used for all the experiments

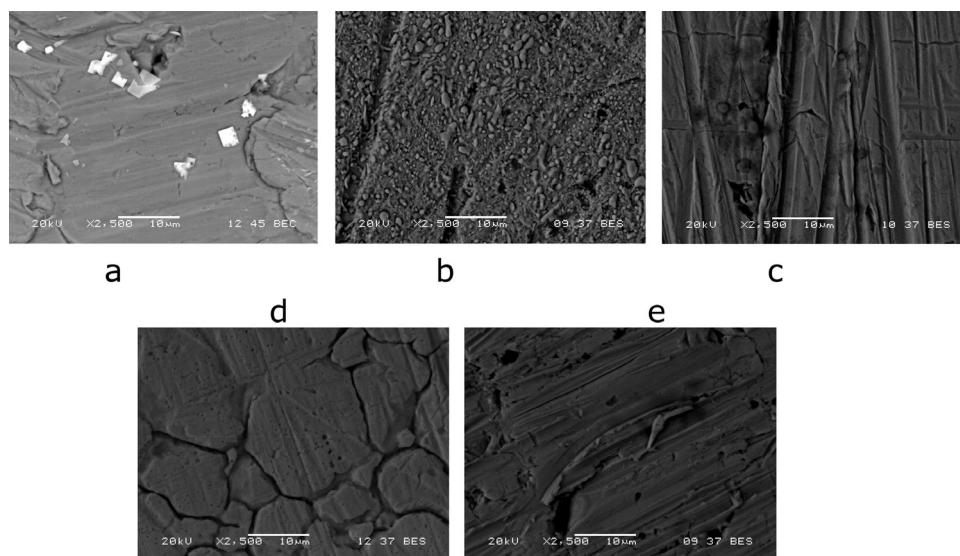


Fig. 1. SEM image of samples immersed for 4 min in a bath exempt of reducing agent. (a) St 37 steel (mild steel), (b) AISI 1070 steel (low alloy carbon steel), (c) ASTM A353 (cryogenic steel), (d) AISI 304 (austenitic stainless steel), (e) ASTM A182 (grade F55) (duplex austeno-ferritic stainless steel).

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