



## Microstructure analysis of boronized pure nickel using boronizing powders with SiC as diluent

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### ABSTRACT

In this study, boronizing of 99.9% pure nickel was performed by means of a powder-pack method using Commercial LSB-II powders (that contained SiC) at 850, 900 and 950 °C for 2, 4, 6 and 8 h, respectively. The coated samples were characterized by X-ray diffraction (XRD), scanning electron microscopy (SEM) equipped with energy dispersive spectroscopy (EDS) and hardness tests. The presence of boride (Ni<sub>2</sub>B) and silicide (Ni<sub>5</sub>Si<sub>2</sub>, Ni<sub>2</sub>Si) phases, formed on the surface of boronized pure nickel, were confirmed by X-ray diffraction analysis. The Ni<sub>3</sub>Si phase was found when pure nickel was boronized at 850 °C for 2 h. Depending on boronizing time and temperature, the thickness of coating layer ranged from 36 to 237 μm. The hardness values were 832 HV<sub>0.01</sub> for the silicide layer, 984 HV<sub>0.01</sub> for boride layer, and 139 HV<sub>0.01</sub> for the Ni substrate.

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### 1. Introduction

Boronizing is a thermochemical surface-hardening process by which the boron atoms diffuse into the metal substrate and form the metallic boride on the substrate surface at the temperature between 700 and 1000 °C for 1–10 h. The formation of the metal boride will provide the excellent surface properties in terms of high hardness, corrosion resistance, high temperature oxidation resistance up to 850 °C, and the length of service life up to 3–10 times [1–4]. Therefore, boronizing treatments have been applied to a wide range of materials including ferrous, non-ferrous and some super alloys [5–8].

Ni and Nickel alloys are extensively used in industrial plant and equipment for their high resistance to corrosion [9,10]. However, these alloys are not considered for applications where wear resistance, or erosion and corrosion in combination, is of primary concern [9]. It is necessary to improve their wear resistance through surface hardening treatments. In such surface treatments, carburizing and nitriding of nickel are difficult, because nickel has a very low solubility for carbon and nitrogen in

the solid state [11,12]. Boronizing, on the other hand, is appropriate for nickel because nickel can be easily boronized and the boride layer is thick.

Anthymidis et al. [13] used the Fluidized bed technology (FBT) to deposit boride coatings on nickel metal. It was found that only Ni<sub>3</sub>B was formed during the treatment and the coating thickness was up to 35 μm. However, fluidized beds require higher capital and operation costs than the conventional boronizing process [8]. The most frequently utilized method is pack boronizing which is a process similar to pack carburizing process. Especially, Power-pack boronizing has some important advantages in terms of easy handling, the flexibility with respect to the composition of the power, minimal equipment and low cost [14–16]. Nevertheless, due to the composition of the boriding powders which contain SiC and KBF<sub>4</sub> as a diluent and an activator, respectively, experiments and thermodynamic calculations of the reaction processes involved show that two competing processes occur during boronizing of nickel-based alloy, namely, boronizing and siliconizing. During this process, a dual-layer coating consisting of an outer layer of silicide, and an inner layer of boride is formed [17,18]. To avoid this, some special boronizing powders without SiC as diluent are developed for surface hardening of Ni and Nickel alloys [9,19,20]. At high temperatures, however, interfacial diffusion between the substrate and the coating accelerates, leading to a reduction in many of the coating's protective properties [21]. It is necessary to use a multi-layer coating to improve these performances.

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In the present study, 99.9% pure nickel was boronized by powder-pack method using Commercial LSB-II powders which contained SiC and  $\text{KBF}_4$ . The microstructure and properties of boronized pure nickel were analyzed by scanning electron microscopy (SEM), X-ray diffraction (XRD) analysis and micro-hardness tester. To determine the distribution of alloying elements of multilayer coating formed on the surface of pure nickel, energy dispersive X-ray spectroscopy (EDS) was used. The main goal of the study was to investigate the properties of multilayer coating of boronized pure nickel. This study enlightened the possibility of multilayer coating on the surfaces of substrate metals or alloys using the pack boronizing method.

## 2. Experimental details

### 2.1. Substrate materials

The substrate material used for this study was 99.9% pure nickel and had a dimension of  $10 \text{ mm} \times 10 \text{ mm} \times 10 \text{ mm}$ . Before boronizing, all the samples were ground with 600 grid emery papers to get the final surface finish and then ultrasonically cleaned in acetone.

### 2.2. Boronizing

In this study, pack boronizing method was used. Commercial LSB-II powders contained the boriding source (8%  $\text{B}_4\text{C}$ ), the activator (4%  $\text{KBF}_4$ ) and the diluent (88% SiC). All samples to be boronized were packed in the powders mix and sealed in a stainless steel container. Boronizing was performed in an electrical resistance furnace under atmospheric pressure at 850, 900 and  $950^\circ\text{C}$  for 2, 4, 6 and 8 h, respectively, followed by furnace cooling.

### 2.3. Coating characterization

The cross-section of boronized pure nickel was observed by scanning electron microscopy (SEM, HITCHIS-3400N) equipped with energy dispersive spectroscopy (EDS, HORIBA EMAX). The phase analyses were carried out using a Philips X-ray diffractometer with  $\text{Cu K}\alpha$  radiation. The hardness measurements of the layers formed surface were performed using the Vickers microhardness tester (SHIMADZU/HMV-2) with the loads of 10 g.

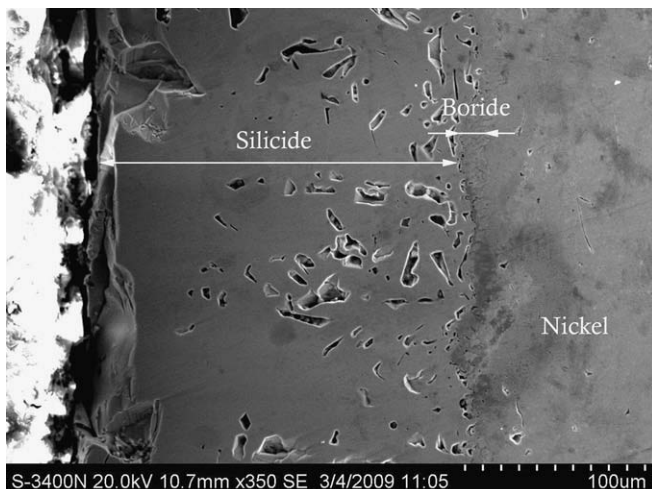


Fig. 1. Cross-section view of the pure nickel boronized at  $950^\circ\text{C}$  for 8 h.

## 3. Results and discussion

### 3.1. Microstructure

Fig. 1 shows the SEM image of the cross-section of the pure nickel boronized at  $950^\circ\text{C}$  after 8 h. Silicide formed on the surface

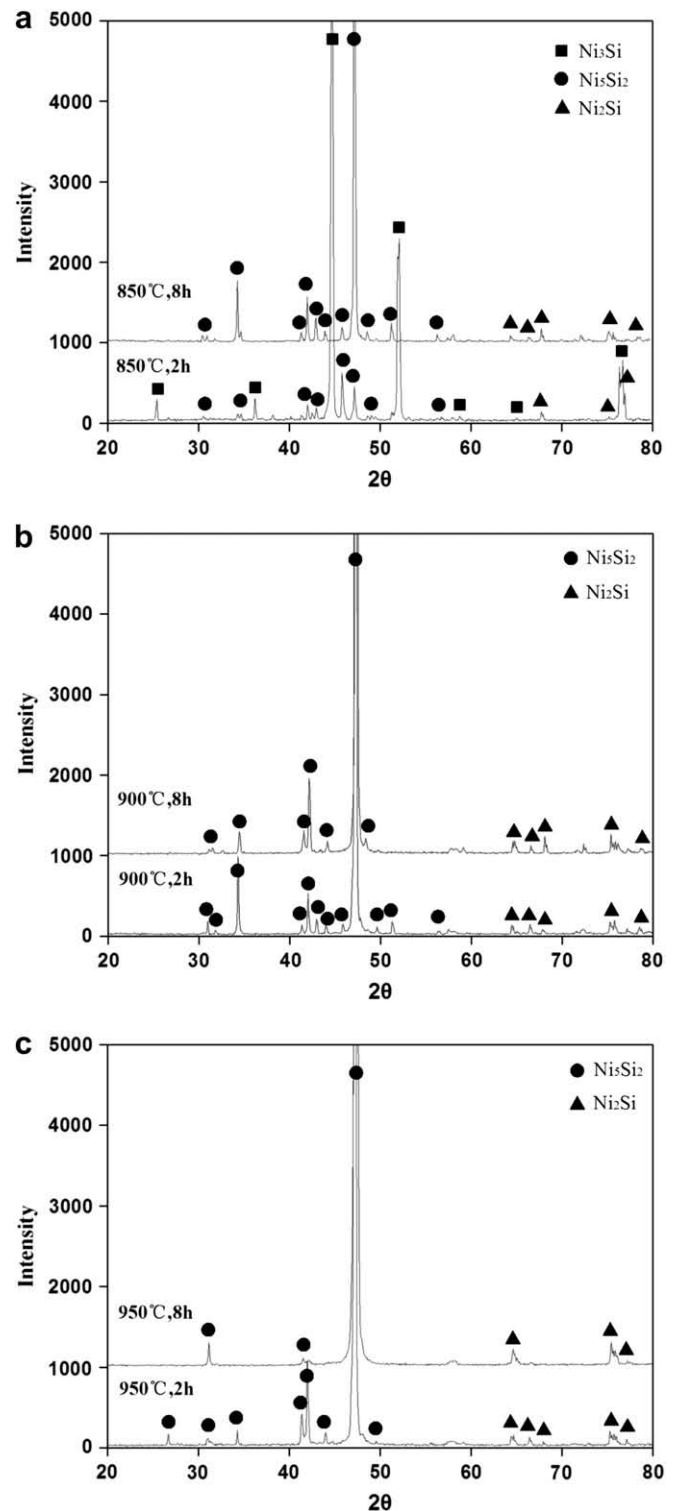


Fig. 2. XRD pattern of pure nickel boronized for 2 and 8 h at various temperatures: (a)  $850^\circ\text{C}$ ; (b)  $900^\circ\text{C}$ ; (c)  $950^\circ\text{C}$ .

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