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POLYTECH.MONS

Improvement of hardness and mechanical properties of electroless nickel-boron deposits by thermochemical treatment.

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Introduction

Autocatalytic nickel-boron deposits, obtained from the reduction of nickel salts by sodium borohydride, present very good hardness and corrosion resistance.
Heat treatment at 400°C induces an increase of their hardness from 650-750 hv_{100} up to 1200-1300 hv_{100}.
The object of this work is to improve the mechanical properties of nickel-boron deposits by the way of alternative treatments such as thermochemical treatments. We discuss here the influence of vacuum heat treatment under pure nitrogen atmosphere on the structure and properties of nickel-boron coatings deposited on mild steel.

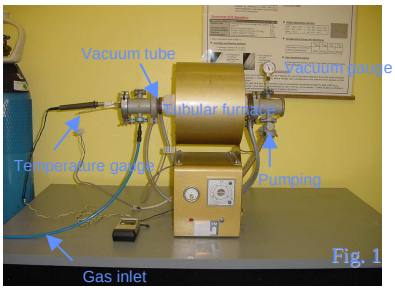

Fig. 1

Sample preparation

Nickel-boron deposits were synthesized using an alcaline borhydride reduced deposition bath. The complexing agent is ethylene diamine and the stabiliser is lead tungstate. Deposition was carried out at 95°C, with mechanical stiring.
The thermal treatment, under argon and nitrogen atmosphere, were realized in a vacuum tubular furnace that can be seen on fig. 1.

Hardness measurements

Vickers indentations were carried out on the surface of the coating, while knoop microninentations and Berkovitch instrumented nanoindentations were performed on polished cross-sections.
To compare the results obtained from various techniques, the 'zero-load' hardness values were calculated by linear regression.
During vacuum nitrogen heat treatment, the hardness of the coating first increases up to the maximal hardness value for heat treated nickel-boron coating, which is obtained for 400°C (see fig.2). For comparison purpose; the results obtained for 400°C heat treatment under argon atmosphere are displayed on the graph.
For temperatures above 400°C, the hardness continues to increase, and reaches a maximal value of 1600hv_{100} for a treatment at 600°C.

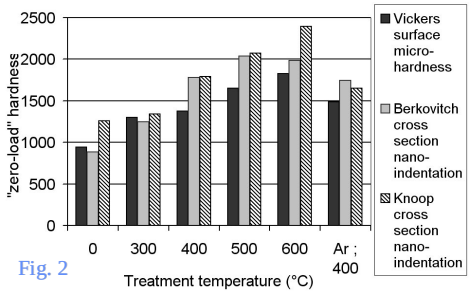

Fig. 2

Nanoindentation profile

Nanoindentation profiles were obtained using rows of indents across the coating on polished cross-sections as shown on fig. 3. We observed an higher hardness near the surface of the sample than in the bulk of the coating (fig. 4). This is the consequence of the interaction of nitrogen with the surface of the coating.

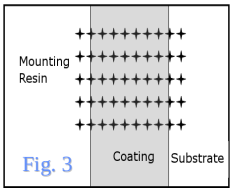

Fig. 3

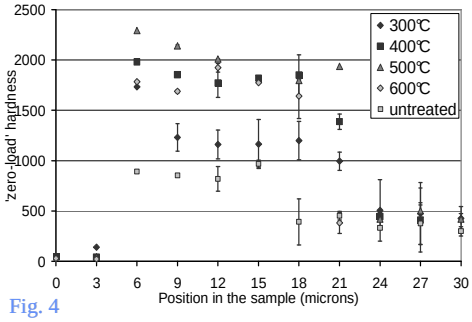

Fig. 4

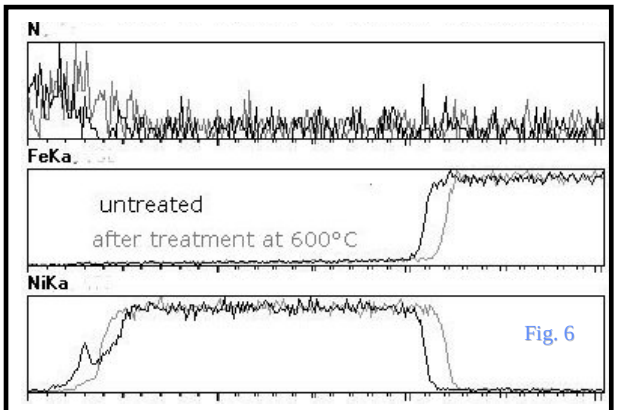

Fig. 6

SEM observation and analysis

Scanning electron microscopy imaging showed that after heat treatment at 600°C, the coating no longer present the columnar structure typical of electroless grown deposits (Fig5.)
Linescan analysis across the coating didn't show any diffusion between the substrate and the coating (fig. 6).

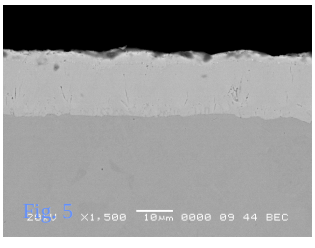
Fig. 5

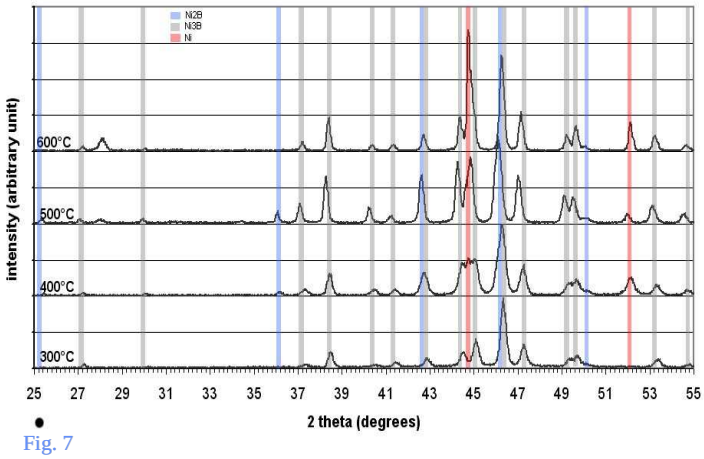

Fig. 7

XRD analysis

The XRD spectra of the nitrogen treated samples present a number of peeks that can be attributed to Ni, Ni_2B and Ni_3B (fig. 7) However, for high temperature treatment, an unidentified peek is forming at a 2-theta angle of 28°. This unidentified phase is probably the result of interactions between the treatment atmosphere and the nickel surface.

Conclusions

Nitrogen atmosphere heat treatment were carried out on nickel-boron deposits and induced a hardness increase that is not explained by the crystallisation of nickel and nickel boride phases. A further hardness increase was also observed near the free surface of the coating.
XRD and SEM analysis were performed but the mechanism responsible for this hardness increase has not been identified yet.