

IMPROVEMENT OF STEEL SURFACES BY A COMBINATION OF MAGNETRON SPUTTERING AND PLASMA NITRIDING

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Abstract

The paper deals with a two-step process consisting of magnetron deposition of Ti-based films on steel substrates and subsequent plasma diffusion treatment of these pre-coated substrates. First, the Ti-based films with the thickness of 4.5 μm were prepared by DC magnetron sputter ion plating at the working pressure of 0.6 Pa, the substrate temperature $T_s = 200$ °C and various values of the negative substrate bias U_s . Then these pre-coated steel samples were nitrided in an abnormal glow discharge in a mixture of $\text{N}_2\text{-H}_2$ (1:1) at the total pressure of 1067 Pa, the constant nitriding temperature $T_n = 550$ °C and the nitriding time $t_n = 2$ h. It is shown that the plasma nitriding stimulates a strong interdiffusion between Ti and the substrate elements. It results in formation of a broad interfacial region (several micrometers) with a considerable intermixing of Ti with Fe and another elements from the steel substrates.

Introduction

At present, one of the key problems of surface engineering is achieving a good bond between the hard and protective coatings and the substrates.

It is well known that the good performance of a coating depends not only on its microstructure, physical and chemical properties, but also on its adhesion to the substrate. The adhesion is determined by the interface between the coating and the substrate. For coatings formed by various physical vapour deposition techniques the interface is usually very sharp, with a thickness of less than a nanometer, which does not result in good adhesion.

Recently [1, 2], we described a new duplex coating technique which increases substantially the interdiffusion of elements between the coating and the substrate. This two-step method, combining the magnetron sputtering and plasma nitriding, differs from the conventional duplex techniques [3 - 5] in the order of the individual steps.

The main aim of this paper is to report on the measurements of the elemental depth profiles in the interfacial region between the Ti-based coating and the steel substrate and on the stimulation of the Ti and steel elements intermixing by plasma nitriding of Ti-based coating/steel substrate couple.

Experimental

A plasma nitrided Ti-based coating/steel substrate couple was prepared in two steps.

First, the Ti-based film with the thickness of 4.5 μm was deposited by the magnetron sputter ion plating process onto a substrate (the diameter of 18 mm, the thickness of 6 mm) made of the 15 330 steel (chemical composition is given in Table 1) at the argon pressure of 0.6 Pa, the substrate temperature $T_s = 200^\circ\text{C}$ and various values of the negative substrate bias U_s . For sputtering a DC planar round magnetron with a Ti target of 60 mm diameter was used. The Ti-target of 99.5 % purity was kept by a stainless steel ring with 71.4 % of Fe, 18.1 % of Cr and 9.7 % of Ni. This resulted in a desirable small addition of stainless steel elements (about 10 %) into the Ti films [6].

After formation of the Ti-based coating, the Ti-based coating / 15 330 steel substrate couple was nitrided in an abnormal glow discharge in a mixture of $\text{N}_2\text{-H}_2$ (1:1) at the total pressure of 1067 Pa, the nitriding temperature $T_n = 550^\circ\text{C}$ and the nitriding time $t_n = 2\text{h}$.

More details on sputtering and nitriding devices are given in Ref. [7].

Table 1. Chemical composition (wt. %) of 15 330 steel.

	C	Mn	Si	Cr	Mo	V	P	S
Minimum	0.24	0.4	0.17	2.3	0.2	0.15		
Maximum	0.34	0.8	0.37	2.7	0.3	0.30	0.035	0.035

The elemental depth profiles across the nitrided Ti-based coating / 15 300 steel substrate couples were investigated by glow discharge optical emission spectroscopy (GDOS) using a LECO SPD - 750 spectrometer. The thickness of the sputtered Ti-based coatings was measured with an Alphastep 100 tester.

Results and discussion

In Fig. 1 the depth profiles of the selected elements in the nitrided Ti-based coating / 15 300 steel substrate couples are given to show what effect on the interdiffusion of elements between the coating and the substrate has the value of the negative substrate bias U_s which determines [6] the structural properties of the Ti-based

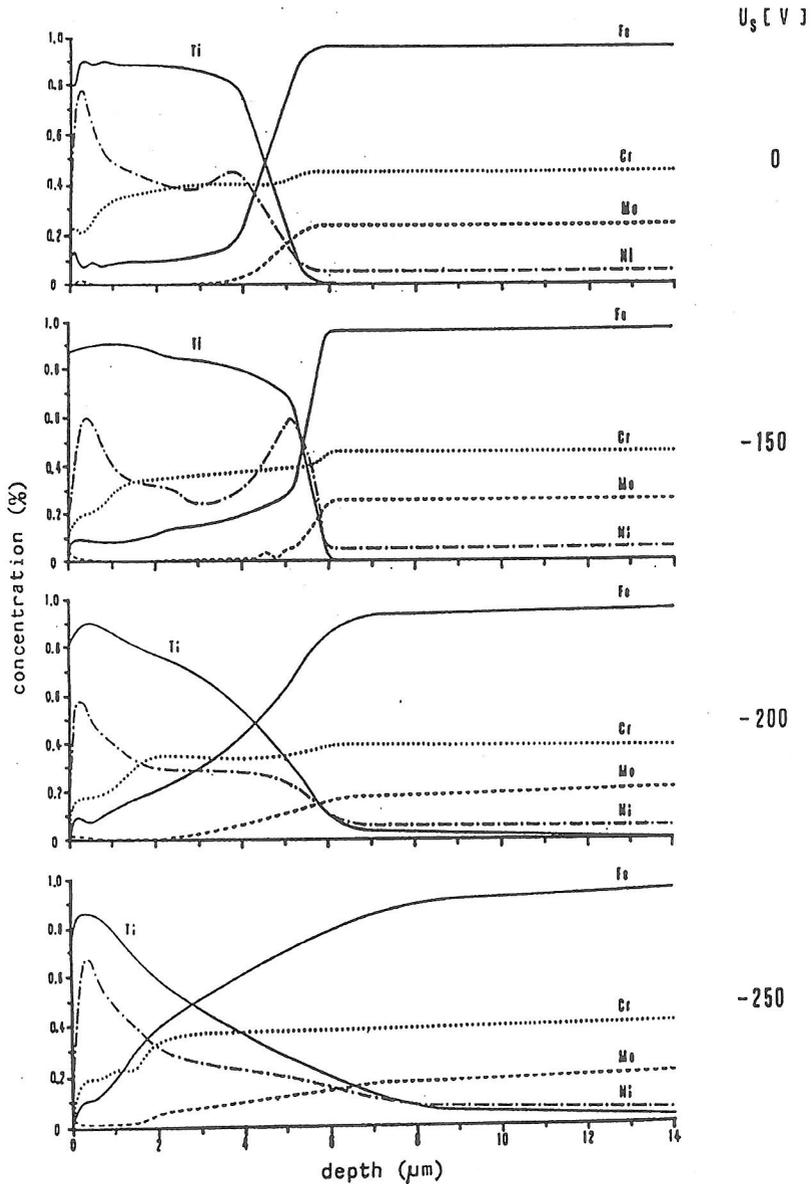


Fig 1. Depth profiles of the selected elements in the Ti-based coating / 15 330 steel substrate couples after their nitriding at $T_n = 550$ °C, $t_n = 2$ h. and $p_{tot} = 1067$ Pa. The Ti-based coatings were deposited at $T_s = 200$ °C, U_s in the range from 0 to -250 V and $p_{Ar} = 0.6$ Pa. Sensitivities for individual elements in GDOS analysis: 100 % (Fe,Ti); 5 % (Cr); 2 % (Ni) and 1 % (Mo).

films.

As can be seen, at the values of $|U_S| < 150$ V a sharp coating - substrate interface is registered. A decrease in the Ti concentration and the corresponding increase in the Fe concentration in the Ti-based coatings are caused predominantly by a crater effect inherent to the GDOS method.

At the higher values of the negative bias U_s considered the plasma nitriding stimulates a very efficient diffusion process, which results in a strong intermixing of the Ti with Fe and other elements present in the 15 330 steel (see the diffusion of the molybdenum). As a consequence, a broad interfacial region (several micrometers) is formed under these conditions, when the combined action of the ion bombardment and mixing of Ti with a low content of the stainless steel elements leads to production of amorphous or nanocrystalline films [8].

Typical depth profiles for N, decreasing from the surface of the coating towards the coating - substrate interface (as it is usual in plasma nitriding), are shown in our papers [1, 2], where the effect of a titanium nitride layer produced on the top of the Ti coatings is discussed.

References

- [1] J. Musil, J. Vlček, V. Ježek, M. Benda and Z. Weiss, Surf. Coat. Technol. (1995), in print.
- [2] J. Musil, J. Vlček, V. Ježek and M. Benda, Protection of aluminium by duplex coatings, Internat. Confer. on Metallurgic. Coat. and Thin Films, San Diego (1995), Paper BH. 03.
- [3] M. Zlatanovič, Surf. Coat. Technol. 48 (1991) 19.
- [4] M. Van Stappen, B. Malliet, L. M. Stals, L. De Schepper, J. R. Ross and J. P. Celis, Mater. Sci. Eng. A 40 (1991) 554.
- [5] J. D. Haen, C. Quaeys, L. M. Stals and M. Van Stappen, Surf. Coat. Technol. 61 (1993) 194.
- [6] J. Musil, J. Vlček, V. Ježek, M. Benda, M. Kolega and R. Boomsma, Production of Ti films with controlled texture, Internat. Confer. on Metallurgic. Coat. and Thin Films, San Diego (1995), Paper B3.02.
- [7] J. Musil, J. Vlček, V. Ježek, M. Kubásek, R. Čerstvý, T. Tolg, M. Benda, M. Kolega and J. Musil, jr., Mater. Sci. Technol. A 163 (1993) 181.
- [8] J. Musil, J. Vlček, V. Ježek, M. Benda and L. Soukup: Interdiffusion between Ti and steel elements in Ti coating / steel substrate couple, Proc. 13th Internat. Vacuum Congress and 9th Internat. Confer. on Solid Surfaces, Tokyo (1995).