EFFECT OF ION FLUX PARAMETERS ON THE RATE OF AUSTENITIC STAINLESS STEEL NITRIDING IN ELECTRON BEAM GENERATED PLASMA

Nikolay V. Gavrilov[*], **Andrey I. Menshakov**

The Institute of Electrophysics, UB RAS,
Amundsen 106, 620016, Yekaterinburg, Russia.

ABSTRACT

Nitriding of metals in electron beam generated plasma provides change of ion current density j and ion energy E over a wide range, the constant temperature of a sample being sustained by change of electron beam parameters. Broad (100 cm^2) electron beam (5-20 A, 60-500 eV) was generated by electron source with a plasma cathode. 12X18H10T stainless steel was nitrided at 500 ^oC during 1 h. Dependences of nitriding layer thickness on j (1,6-6,2 mA/cm^2), E (100-300 eV), pressure of Ar-N_2 gas mixture (1-10 Pa) were obtained. Nitrided layer thickness was measured in cross-sections with the use of microhardness meter and SEM micropictures. The layer thickness decreases with the rate 4-5 microns per 100 eV with E growth and rises from 19 up to 33 microns with j increase. Character of nitriding rate dependence on j contradicts conclusions of the theory concerning restriction of the layer thickness by ion sputtering with j growth. The value of effective diffusion coefficient is an order of magnitude higher than the one known from literary data.

Key words: self-heated hollow cathode, plasma nitriding, electron beam, nitrogen diffusion, stainless steel, ion sputtering.

INTRODUCTION

The ability of electron beam to simultaneously provide plasma generation and heating of the articles permits independent change of current density and ion energy in the process of nitriding while sample temperatures are kept constant. Selection of stainless steel was determined by the difficulty of its nitriding caused by the influence of dynamic oxide layer, by wide data spread on the dependence of nitriding rate on ion flow density and by impossibility to explain it in terms of the theory of diffusion under the action of N concentration gradient [1]. The work was aimed at the study of influence of the parameters of ion flux from and gas pressure on the rate of stainless steel nitriding.

METHODS OF SAMPLE MANUFACTURING AND ANALYSIS

Electron source based on discharge with self-heating hollow cathode generated a broad (100 cm^2) electron beam (5-20 A, accelerating voltage U_a = 50-

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[*] gavrilov@iep.uran.ru, tel: (+38)3432678778

500 eV). Plasma was generated by ionizing of N_2-Ar gas mixture (Ar:N_2 = 1:1) at 0,1-10 Pa. The samples were heated by electron beam and ion flow accelerated by the bias voltage U_s = 100-300 V. Samples made of sheet stainless steel 12X18H10T were exposed to electropolishing, cleaned in ultrasound bath with acetone and by ion sputtering (Ar^+, 1 μA/cm^2, 300 eV, 20 min). The nitriding process took 1 hour at sample temperature T = 500 °C. The nitrided layer thickness h was determined by measurement of microhardness at cross-sections. Nitrogen distribution in the layer was measured by means of energy dispersive X-ray analysis. Plasma diagnostics was made with the use of movable collecting Langmuir probe shielded from the beam electrons.

RESULTS AND DISCUSSION

Increase of gas pressure results in the growth of inhomogeneity of beam generated plasma, decrease of ion current density to the samples and reduction of electron temperature of plasma to 1-2 eV.

The ion current density at T = 500°C and U_s = –100 V varies within j = 1,6 – 6,2 μA/cm^2. The layer thickness h rises with j from 17 to 33 μm at U_s = 100 V (*Fig. 1*). Increase of U_s by 100 V leads to decrease of h value by 5-6 μm at j = const. The thickness h increases with pressure growth till 7 Pa (*Fig. 2*). The width of nitrogen distribution profile with N concentration of 15-18 at.% corresponds to the thickness of hardened layer.

According to [2], the layer thickness h is determined as $h = 2D/v_s$, where D is nitrogen diffusion coefficient and v_s is the rate of ion sputtering. Estimation of v_s according to the differences in layer thickness got at the same ion current density j and different ion energy gives values of sputtering coefficients several times exceeding those calculated from the TRIM program.

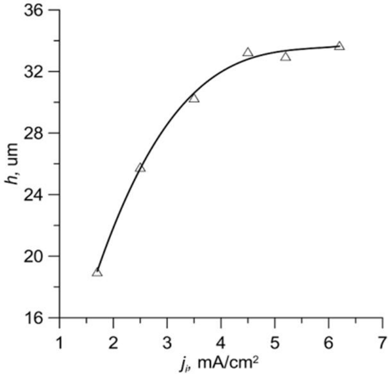

Fig. 1 – Nitrided layer thickness vs ion current density. U_s = -100 B

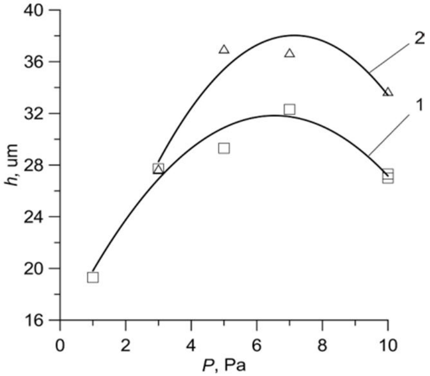

Fig. 2 - Nitrided layer thickness vs gas pressure. 1 – Ar+N_2 gas mixture, 2 – Ar+N_2+10 at.% C_2H_2

At D = const layer thickness h should be changed as 1/j, whereas experimental dependences h(j) have growing character. D value calculated basing on $h = (2Dt)^{1/2}$ [3] makes (5-15)*10^{-10} cm^2/s, which result substantially exceeds the known data. As diffusion saturation of steel is provided by atomic nitrogen [4] and falling-to-absorbed ions' fluences have 2:1 ratio, conclusion may be done that N^+ concentration in the beam is high, that is not confirmed by data of optic emission spectroscopy. Therefore growth of nitriding rate with pressure is determined by increase of N atoms content in plasma. With pressure increase the area of gas ionization by electron beam is localized nearby the electron source, and charge exchange of argon ions on N_2 molecules and dissociative recombination of N_2^+ ions predominate nearby the sample, that leads to growth of N concentration [5].

CONCLUSIONS

Independent variations of ion current density j and ion energy E at constant temperature of the sample were achieved by regulation of electron beam parameters. It was established that E growth leads to decrease of nitrided layer thickness, and j increase, at the contrary, rises the speed of layer growth. Enhanced rate of layer growth at gas mixture pressure rising can be explained by increase of plasma inhomogeneity and creating of conditions promoting growth of atomic nitrogen concentration nearby the samples.

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