ION CLEANING AND NITRIDING USING A HIGH APERTURE HALL CURRENT ACCELERATOR

B. B. STRAUMAL[1, 2], N. F. VERSHININ[1], S. A. POLYAKOV[2,3], P. V. ORLOVA[3], M. FRIESEL[4], and W. GUST[2]

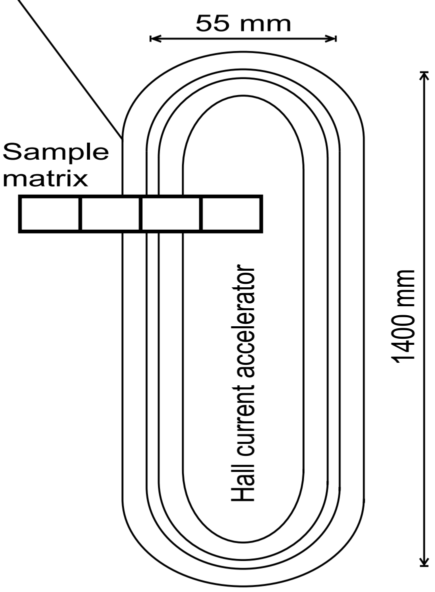
[1]I. V. T. Ltd. (Institute for Vacuum Technology) - P.O. Box 47 - 109180 Moscow - Russia
Tel.: 7.095.2382326 - Fax : 7.095.1117067 - e-mail : straumal@song.ru
[2]Institut für Metallkunde - Seestr. 92 - 70174 Stuttgart - Germany
[3]Institute of Solid State Physics – Chernogolovka - Moscow District -142432 Russia
[4]SIMS Laboratory - Chalmers University of Technology - 41296 Gothenburg - Sweden

ABSTRACT: A large aperture Hall current accelerator has been developed for ion cleaning of substrates before vacuum arc deposition of protective and decorative layers. The accelerator has a large aperture of 1400 mm and a power up to 10 kW. High ion currents permit one to use the source also for ion implantation. Various gases can be used for both purposes: argon, nitrogen, oxygen, etc. The current-voltage characteristics for nitrogen at various pressures are presented. The ion implantation of nitrogen was performed into austenitic stainless steel at a discharge voltage of 900 V and a discharge current of 3 A. The depth profiles were measured with the aid of secondary-ion mass spectroscopy. The load dependence of the microhardness after ion nitriding was measured.

Key words: Ion cleaning, Vacuum arc deposition, Hall current accelerator, Nitriding

1. INTRODUCTION

The nitriding of the substrates before deposition of nitride coatings allowes one to increase their adhesion and the overall corrosion resistance of coated product [1]. Recently, the method



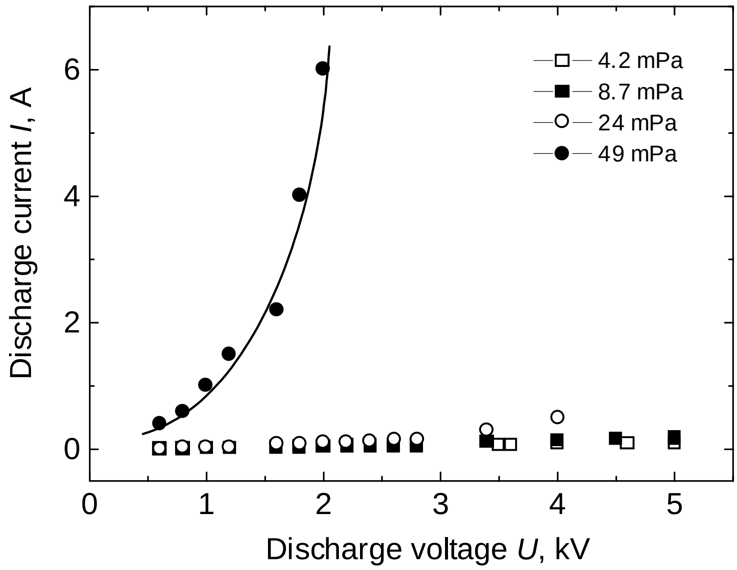


Fig. 1. Scheme of the Hall current accelerator and position of the sample matrix.

Fig. 2. Current-voltage characteristic for nitrogen at various pressures.

of low-energy high current ion implantation (**LEHCII**) was developed [2]. In this method the energy of ions is below 1000 eV (ballistic penetration depth about 5 nm [3]) but the ion flux is very high (up to 1 mA/cm^2). Particularly, the Kaufman broad beam ion sources are used for this purpose [4]. LEHCII permits to alloy the surface layers of steels with nitrogen over a thickness of a few μm even without additional heating [2]. The high power large aperture Hall current accelerator [5] has several important advantages in comparison with the Kaufman sources [4, 6]. Particularly, the Hall current accelerator has a larger aperture permitting to treat big substrates (1400 mm scalable up to 3000 mm in our case), high power (up to 10 kW), is more robust and simple in exploitation. It is easy to combine the Hall current accelerator with existing technologies for the deposition of coatings. It can be used not only for ion cleaning [5] but also for ion implantation. Various gases can be used for both purposes: argon, nitrogen, oxygen, etc.

2. EXPERIMENTAL

The Hall current accelerator described elsewhere [5] has the form of an elongated loop with vertical aperture of 1400 mm (Fig. 1). The nitrogen implantation into austenitic stainless steel 12X18H9T (Russian standard GOST 5632) was studied at a discharge voltage *U* of 900

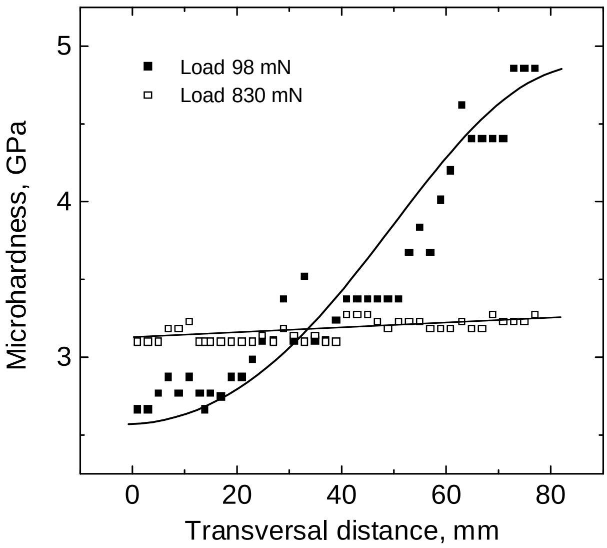


Fig. 3. Distribution of microhardness along the axis of sample matrix (cf. Fig. 1)

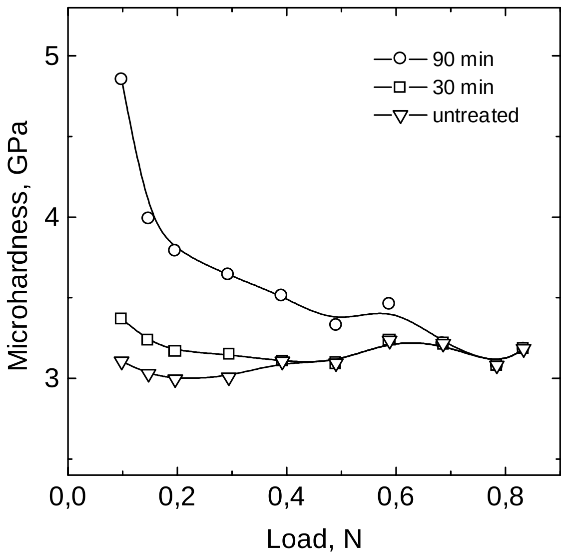


Fig. 4. Load dependence of microhardness at transversal distance 75 mm

V and a discharge current *I* of 3 A during 30 and 90 min. No additional heating of the samples was used. The composition of the steel was controlled by spark spectral analysis. The steel contains (in wt. %) 0.11 C, 17.0 Cr, 8.8 Ni, 0.35 Ti, 0.28 Mo, 0.55 Si, and 0.35 Mn. The samples having the dimension 20×15 mm were cut from a rolled strip of 2 mm thickness, ground and polished. The sample matrix (four samples with an overall length 4×20 = 80 mm) was mounted into a holder positioned in the front of the Hall current accelerator at a distance of 10 cm (Fig. 1). The distribution of C, N, Fe, Cr and Ni in the samples after nitrogen ion implantation was determined using the secondary-ion mass spectrometry (SIMS). An *IMS-3F* apparatus (Cameca, France) has been used for in-depth analyses of the films and substrates. The O_2^+ ions accelerated with a voltage of 12.5 kV were used as primary ions. The primary ion current, I_p, ranged from 250 to 1800 nA. The primary ion beam was rastered over a square area of 250×250 μm. The secondary ions, accelerated by 4.5 kV, were collected from a square area of 100×100 μm in the middle of the rastered area. The energy band pass filter for the secondary ions was 50 eV, centered at the maximum energy of the secondary ions. The distributions of C, N, Ti, Fe, Cr and Ni were studied by profiling the ions $^{12}C^-$, $^{14}N^-$, $^{24}C^-$, $^{28}CO^-$, $^{26}CN^-$, $^{56}Fe^+$, $^{52}Cr^+$ and $^{60}Ni^+$, respectively. The $^{26}CN^-/^{24}C^-$ ratio was used for the estimation of the nitrogen concentration by depth profiling due to the very low intensity of the $^{14}N^-$ line. The depth of the sputtered craters were measured with a *Talysurf 10* instrument

(Rank Taylor Hobson, UK). Each crater was measured several times in the central region of the crater. The deviation in the average depth ranged from 2 to 11%. The microhardness of the nitrided samples was measured at various loads (from 0.1 to 0.85 N) with the aid of an instrument for conventional microhardness measurements*PMT* (LOMO, Russia).

3. RESULTS AND DISCUSSION

The voltage-current characteristics for nitrogen discharge at various nitrogen pressures p are shown in Fig. 2. It can be seen that at p below 30 mPa a slow increase of the discharge current I proceeds with increasing dicharge voltage U. Generally speaking, the voltage-current characteristic of apparata like Hall-current accelerator contain low-voltage and high-voltage branches with sharp transition in between, where the process is rather unstable. Such transition area becomes visible at p = 49 mPa. The dependence of the microhardness on the transversal coordinate is shown in Fig. 3 for two loads (98 and 830 mN) for a sample matrix implanted by nitrogen at U = 900 V and I = 3 A during 90 min. The microhardness measured at the high load corresponds to the bulk value hardness of stainless steel and remains almost unchanged. The microhardness measurements at the low load are sensitive to the properties of the surface layer. The microhardness increases after the nitrogen implantation. It is maximum in the zone close to the source (Fig. 1). The full current density (sum of the electron and ion current) measured in this zone was about 1 mA/cm^2. The microhardness measured along the sample matrix decreases outwards of source axis. At a distance of about 80 mm from the source axis the microhardness of the surface layer is lower than that of the bulk one. It can be explained by the combined influence of (already low) nitriding and (still rather high) heating by ion beam. The dependence of microhardness on the load is shown in Fig. 4 for the untreated stainless steel substrate and after nitriding during 30 and 90 min. The indentation depth changes from 14 μm at 0.1 N to 50 μm at 0.8 N (90 min). Therefore, at high loads the thickness of the nitrided layer is negligible in comparison with the indentation depth, and the hardness of the bulk material is measured (about 3.2 GPa in all three curves). The hardness of the untreated material is nearly independent on the load. The implantation of nitrogen increases the surface hardness of the material. After 30 min the hardness at loads below 0.3 N is higher than that of the untreated sample. With increasing duration of the ion nitriding the surface hardness increases as well. After 90 min the hardness at 0.1 N is almost two times higher than that of the untreated material. The thickness of the nitrided layer increased as well, namely the hardness drops down to the bulk value only at a load of 0.6 N. The depth of the nitrided layer could be rougly estimated from the curves shown in Fig. 4. It can be supposed that the measured hardness reaches the bulk value if the thickness of the hardened layer is less than 0.1 of the indentation depth. This estimation gives about 3 μm for the thickness of the hardened layer. The SIMS depth profiles are shown in the Fig. 5 for 30 and 90 min treatments. The depth of the nitrided layer is about 0.3 μm after 90 min and about 0.1 μm after 30 min. Therefore, the estimation given above delivers overestimated values for the

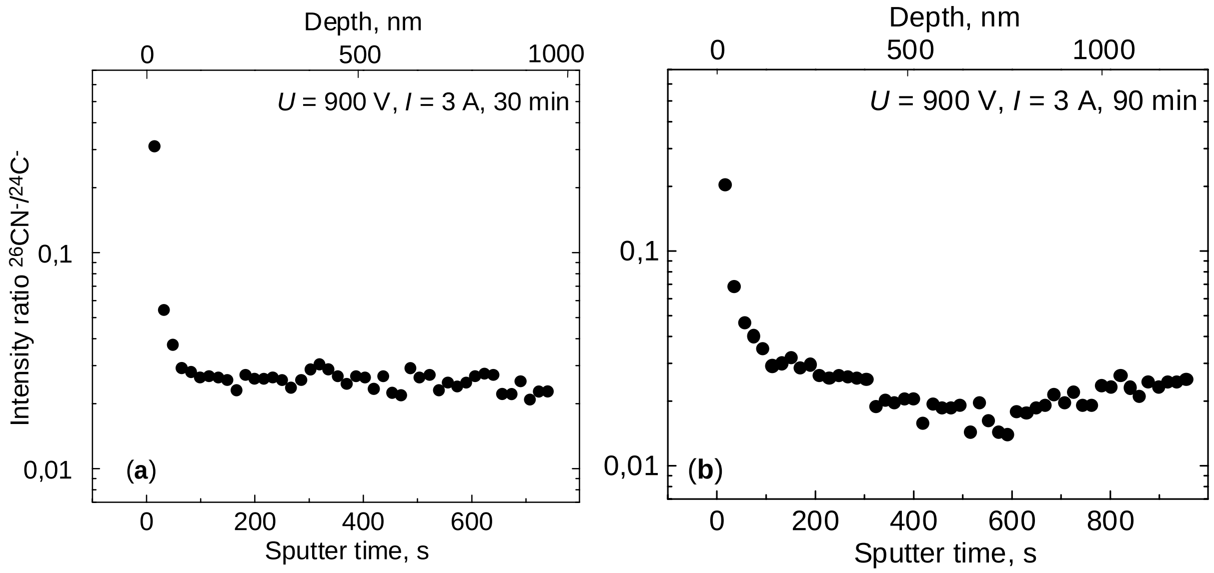


Fig. 5. SIMS depth profiles for the nitrogen implanted into stainless steel at $U = 900$ V and $I = 3$A. Implantation time $t = 30$ min (a) and 90 min (b).

thickness of the hardened layer. Therefore, the actual hardness values of the nitrided layer is higher than the measured ones even by load of 0.1 N because the depth of the crater is much ddeper than the thickness of nitrided layer. Therefore, the measurement of nanohardness with lower loads are needed for a correct estimation of the surface hardness. The thickness of the nitrided layer obtained in our experiments without additional heating of the samples is only slightly smaller than that of a layer obtained under comparable conditions (700 keV, 2 mA/cm^2, 60 min) by heating up to 280 °C [2]. The thickness of the penetration layer is two orders of magnitude higher than the ballistic penetration depth for 900 V and also higher than the length of conventional bulk diffusion of nitrogen. The large aperture high power Hall current accelerator permits one to perform the ion nitriding of large area substrates improving the surface hardness of the stainless steel. This treatment can be used for the preparation of substrates for subsequent coating with the aid of plasma spraying, vacuum arc deposition, magnetron sputtering etc. On the other hand, the recently developed Hall current accelerator will allow to investigate carefully the paradoxally deep penetration of nitrogen by changing the ion energy, current, substrate temperature and duration of the treatment.

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