Vacuum 86 (2012) 2035–2039

Contents lists available at SciVerse ScienceDirect

Vacuum

journal homepage: www.elsevier.com/locate/vacuum

Rapid communication

Effect of coating morphology on the electron stimulated desorption from Ti–Zr–Hf–V nonevaporable-getter-coated stainless steel

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ARTICLE INFO

Article history:
Received 19 March 2012
Received in revised form
23 April 2012
Accepted 24 April 2012

Keywords:
Electron stimulated desorption
Non-evaporable getter (NEG)
Coating
Activation
Pumping

ABSTRACT

The electron stimulated desorption (ESD) was studied for quaternary Ti–Zr–Hf–V alloy coated stainless steel samples with different surface structures: dense on one sample and columnar on another. The ESD yields were measured as a function of electron accumulated dose up to ~10^{23} e^-/m^2 or greater and three different NEG coating activation temperatures: 150, 180 and 250 °C. After each ESD experiment the samples were saturated with a mixture of H_2, CO and CO_2. Both samples depicted lower ESD yields for all desorbed species compared to a ternary Ti–Zr–V alloy. It was also shown that although the columnar NEG coating demonstrated better pumping properties and, for NEG activated at 150 °C, lower initial ESD yields, the higher activation temperature may result in a significant H_2 yield increase with dose for the columnar NEG coating. This effect was demonstrated for the first time and should be considered for application in particle accelerators.

The non-evaporable getter (NEG) coating was primarily designed for use in particle accelerator vacuum chambers bringing two nice properties into a conductance limited environment: distributed pumping speed as well as low thermally and particle induced outgassing rates [1–3]. The gas density along the accelerator vacuum chamber is proportional to desorption yield and is decreasing with sticking probability [4]. These two properties of the NEG coating allow the reduction of the gas density along the accelerator vacuum chamber (or its sections) by orders of magnitude [5] or achieving the specified gas density at much lower cost [6]. It was known from previous studies that the columnar structure of the NEG film provides greater sticking probability and capacity than the dense one [7,8]. However, there was no data to compare vacuum performance of dense and columnar coatings under bombardment by energetic particles, such as photons, electrons and ions. Therefore, the aim of the present work was to study these different coatings bombarded with electrons under the same experimental conditions in order to help to predict their behaviour for use in particle accelerators.

Two identical 0.5-m long and 38-mm diameter 316LN stainless tubes were used in this experiment. The internal surface roughness is determined to be in the region of 5 microns measured with an atomic force microscope (AFM) on a small piece cut out from the centre of the sample tube after ESD measurements. The internal surface was coated with 1 micron thick quaternary NEG of equal atomic percent Ti–Zr–V–Hf, using a 3 mm alloy rod as target. The depositions were performed on the solenoid magnetron deposition facility described in details in Ref. [8]. The depositions were carried out at a partial pressure of 10^{-2} mbar of research grade Kr gas and a deposition temperature of 110 °C, with DC and pulsed DC mode to produce columnar and dense thin film structures, respectively. Both tubes were baked prior to deposition and the base pressure was 10^{-10} mbar. Two test pieces of Si and stainless steel were deposited at the same time; these were used to determine film morphology and structure.

X-ray diffraction of both sets of deposition, deposited on Si test samples, showed only a very broad peak at $2\theta = 37°$, representing grain sizes of 2–4 nm calculated by Scherrea equation assuming no stress broadening of the peak. Planar and cross section SEM micrographs showed that the film deposited in pulsed DC mode has very dense structure, the film deposited in DC mode having resulted in a columnar structure with pores ranging from 20 to 40 nm. These are shown in Figs. 1 and 2 (a and b), respectively.

The existing dedicated experimental facility was designed and built to study pumping properties and ESD from NEG coated samples and is described in details in Ref. [8–10]. The tubular sample geometry and the coating conditions are similar to those in an accelerator vacuum chamber, so these experiments replicate the conditions at which the NEG coating is designed to be used. Besides, the closed tubular sample geometry provides the best

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0042-207X/$ – see front matter
doi:10.1016/j.vacuum.2012.04.033

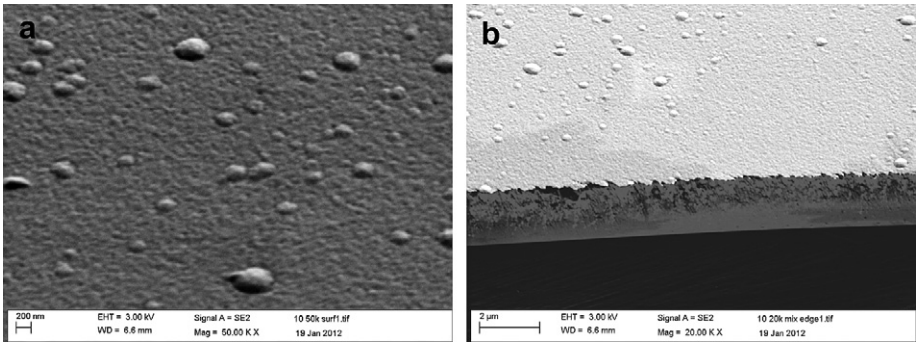

Fig. 1. Planar and cross section SEM micrographs of dense film.

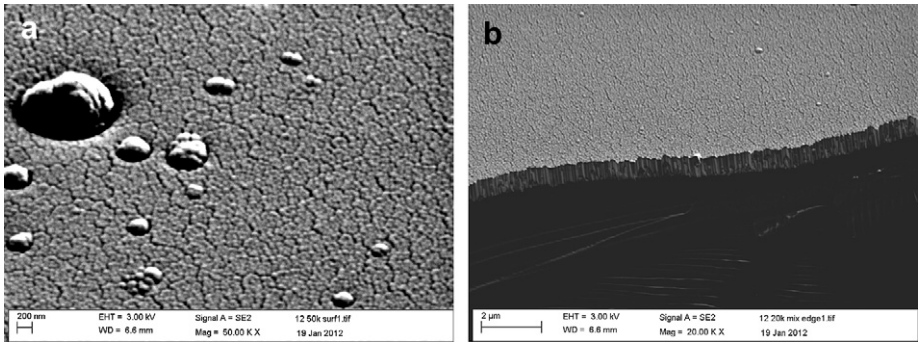

Fig. 2. Planar and cross section SEM micrographs of columnar film.

conditions for NEG coating activation without poisoning it by gas molecules desorbed from the non-coated parts of the vacuum chamber [9]. An existing installation for NEG coating pumping and desorption properties evaluation is described in Ref. [10]. This installation allows the obtaining of two main characteristics for a NEG coated vacuum chamber:

- the NEG sticking probability and pumping capacity by injecting gases that can be pumped by NEG (H_2, CO, CO_2, etc.);
- the electron stimulated desorption (ESD) yields by bombarding the sample tube with electrons emitted from a thoriated tungsten filament placed along the sample. The energy of electrons can be varied between 10 eV and 6.5 keV.

Each sample was studied following the same procedure. After sample installation the facility was pumped and baked to 250 °C while the NEG coated sample was kept at 80 °C. The NEG coating was then activated to 150 °C for 24 h while the rest of the facility was kept at room temperature [9,10]. The sticking probability was measured before ESD measurement by a short injection of H_2, CO and CO_2 mixture, then it was repeated after ESD measurements until full NEG saturation. After that, the NEG activation, sticking probability measurements, ESD measurements and NEG saturation were repeated for higher activation temperatures: 180 °C and 250 °C consequently.

The measured H_2, CO and CO_2 sticking probabilities were practically the same before and after the ESD measurements, they are shown in Table 1 together with and CO sorption capacity. As expected, the columnar film demonstrates a higher sticking probability and pumping capacity than the dense one.

The results for the ESD yields as a function of specific electron dose are shown in Fig. 3. The ESD results for the quaternary alloy are presented for the first time. Comparison between this and our previous results for a ternary Ti–Zr–V alloy [10] depicts that for all desorbed species the ESD yields are significantly lower for both samples even after full saturation with CO and CO_2.

The behaviour of the ESD yields obtained from the dense film activated at 150 °C is quite similar to many other curves of this type [8–10]: a progressive reduction of ESD yields with an accumulated electron dose. The ESD yields for the sample with a columnar NEG coating are approximately one order of magnitude lower for the sample with a dense NEG coating.

Activation at 180 °C significantly reduces the ESD yields from the dense film: factors ~10 for H_2 and CH_4, 100 for CO and ~30 for CO_2 at a dose of 10^{21} e^-/m^2. The ESD yields from the columnar film reduce much less: factors 1.2 for H_2, 4 for CH_4, 5 for CO and ~1 for CO_2 at the same dose. Although the ESD yields from the columnar film were less than from the dense film, the difference between the two samples is much less than when it was activated at 150 °C: 30% for H_2 and a factor of 7 and 5 for CH_4 and CO for doses below 10^{22} e^-/m^2. The observed behaviour of the H_2 yield was very unusual for the columnar film for doses above 10^{22} e^-/m^2, above this dose, the slope of the curve begun to change. The ESD yield was not reducing

Table 1
Sticking probabilities for different gases and CO sorption capacity for the columnar and dense samples activated at different temperatures T_a.

T_a	Dense				Columnar			
	Sticking probability			CO sorption capacity	Sticking probability			CO sorption capacity [ML]
	H_2	CO	CO_2		H_2	CO	CO_2	
150 °C	0.002	0.04	0.075	0.004	0.004	0.2	0.13	3.5
180 °C	0.0013	0.025	0.012	0.13	0.014	0.2	0.13	3.5
250 °C	0.004	0.085	0.02	0.12	0.02	0.2	0.13	–