Journal of Nuclear Materials 466 (2015) 621–626

Contents lists available at ScienceDirect

Journal of Nuclear Materials

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

Retention of nanocrystalline WN_x layers exposed to high-fluence deuterium plasmas

E. Vassallo [a, *], R. Caniello [a], G. Angella [b], D. Dellasega [a, c], G. Granucci [a], V. Mellera [a], D. Minelli [a], M. Pedroni [a], D. Ricci [a], V. Rigato [d], M. Passoni [a, c]

[a] CNR, Istituto di Fisica del Plasma "P. Caldirola", 20125 Milano, Italy
[b] CNR, Istituto per l'Energetica e le Interfasi, 20125 Milano, Italy
[c] Politecnico di Milano, Dipartimento di Energia, 20156 Milano, Italy
[d] INFN – Laboratori Nazionali di Legnaro, Legnaro, 35020 Padova, Italy

HIGHLIGHTS

- Nitrogen in the process of tungsten redeposition in tokamak plays a very important role for fuel retention.
- Incorporation of nitrogen in tungsten decreases the D retention.
- WN_x compounds showed a level of D retention lower than the acceptable limit for tritium (T) operation in ITER.

ARTICLE INFO

Article history:
Received 30 April 2015
Received in revised form
2 September 2015
Accepted 3 September 2015
Available online 7 September 2015

Keywords:
Hydrogen retention
Tungsten nitride
Tokamak

ABSTRACT

For high-power plasma operation regimes in tokamak fusion devices the power load onto W divertor plates must be kept below acceptable limits for materials. N_2 gas is likely to be used to reduce the power load. However, because of erosion phenomena, WN_x compounds will be produced in the divertor and tritium retention is issue of concern. We report recent experiments using the GYM linear plasma device that examined D retention in WN_x compounds exposed to D plasma at divertor relevant fluence ($\sim 10^{24}$ m^{-2}). It is shown that WN_x compounds with different nitrogen concentration have very similar D retention, lower than the case of the tungsten without nitrogen and in any case lower than the acceptable limit for operation in ITER.

1. Introduction

In next generation magnetic confinement fusion devices some plasma particles can escape from the magnetic confinement region and impact the surrounding wall. Such plasma-wall interactions critically affect tokamak operation in many ways. Plasma erosion determines the lifetime of plasma-facing components and creates a source of impurities, which can play a role in cooling and diluting the plasma [1]. Deposition of eroded impurities onto plasma-facing materials alters their surface composition and via co-deposition may lead to long-term accumulation of large in-vessel tritium inventories. Among the erosion mechanisms, the disruptions phenomena are a major concern for future tokamak reactors. High-velocity gas jet injection will be probably the technology used to mitigate disruptions [2]. Noble gas species and nitrogen are being studied on fusion device to investigate the physics of gas jet penetration and the ability of the gas jet impurities to convert plasma energy into radiation [3]. Although the use of N_2 gas to reduce the edge plasma temperature has recently been successfully applied in fusion devices such as ASDEX [4] and JET [5], questions remained about effects of N on the W plasma-facing components and ammonia production. In particular, the knowledge of the dependence of the hydrogen retention mechanisms in the W-N system is still limited and requires further research [6,7]. It is thus important to determine the parameters that govern the retention process as a function of W surface structure and composition.

* Corresponding author.
E-mail addresses: vassallo@ifp.cnr.it (E. Vassallo), caniello@ifp.cnr.it (R. Caniello), angella@ieni.cnr.it (G. Angella), david.dellasega@polimi.it (D. Dellasega), granucci@ifp.cnr.it (G. Granucci), mellera@ifp.cnr.it (V. Mellera), minelli@ifp.cnr.it (D. Minelli), pedroni@ifp.cnr.it (M. Pedroni), ricci@ifp.cnr.it (D. Ricci), valentino.rigato@lnl.infn.it (V. Rigato), matteo.passoni@polimi.it (M. Passoni).

http://dx.doi.org/10.1016/j.jnucmat.2015.09.006
0022-3115/

The purpose of this study is to expose WN_x coatings with different properties (e.g. stoichiometry, crystallinity and N content) deposited using reactive Magnetron Sputtering (MS) to D plasmas with fluencies and energies that are expected in tokamak divertor areas ($\cong 10^{24}$ m^{-2}) and assess D retention level and distribution. The hydrogen retention in W exposed to low-energy deuterium plasma and ion beams has been studied [8–10] quite extensively by Thermal Desorption Spectroscopy (TDS) and Nuclear Reaction Analysis (NRA). The ERDA technique provides the depth profile of the deuterium concentration up to 200 nm in W using ^{7}Li ions [11] and up to 120 nm in WN_x samples using ^{4}He ions, furthermore the absolute concentrations for both isotopes, H and D, can be observed simultaneously.

2. Experimental details

2.1. WN_x coatings production

An RF plasma diode sputtering system [12] has been used to produce WN_x coatings as a function of seeding impurities (N). Coatings with thicknesses in the range of 1 μm were grown on Si substrates (1 × 1 cm^2, thickness ≅ 400 μm). The experimental apparatus consists of a parallel-plate, capacitive-coupled system, made up of a cylindrical stainless steel vacuum chamber with an asymmetric electrode configuration. A powered electrode is connected to an RF (13.56 MHz) power supply, coupled with an automatic impedance matching unit, while the other electrode, made up of stainless steel, is grounded. A 3-in diameter target of W (purity 99.9%) was placed on the powered electrode. Si substrates were placed on the ground electrode at 6.5 cm away from the powered electrode. No bias voltage was applied to the substrate holder. The substrates temperature was monitored by a thermocouple fixed directly on the substrate. Before the process, the substrates were cleaned with chemical etching solutions to remove surface contaminants. The process chamber was pumped to a base pressure below 1×10^{-5} Pa; high-purity gases were introduced into the vacuum chamber through a mass flow controller in order to establish the desired working pressure. Ar/N_2 mixtures were used as sputter gas. Total gas flow rate was fixed at 20 sccm. N_2 dilution was varied in the range 1–7 sccm. The RF power was fixed at 150 W (1500 V of DC self-bias voltage). The target was water cooled, and its temperature was kept below 20 °C during the coating process.

2.2. Coatings characterization

The morphological properties and physical structure of the films were investigated by Scanning Electron Microscopy (SEM). Measurements were performed using a ZEISS Supra System with an accelerating voltage of 5 kV. The structural properties studied by X-ray diffraction measurements were performed with a wide angle Siemens D-500 diffractometer (WAXD) equipped with a Siemens FK 60-10 2000W tube. The radiation was a Kα beam with wavelength λ = 0.154 nm. The operating voltage and current were 40 kV and 40 mA, respectively. The data were collected from 20 to 50 2θ° at 0.02 2θ° intervals by means of a silicon multi-cathode detector Vortex-EX (SII). The roughness was investigated by Atomic Force Microscopy (AFM in air by means of a Nano-RTM System (Pacific Nanotechnology, Santa Clara, CA, USA) operating in contact mode. The deposition rates were measured by a P15 surface profiler (KLATencor San Jose, CA) and by SEM.

2.3. D plasma exposure of coatings

WN_x samples were exposed to deuterium plasmas in the linear plasma device GYM [13]. It consists of a vacuum chamber (radius R = 0.125 m, length L = 2.11 m) mounted in a 0.13 T linear magnetic field (Fig. 1), in which a highly reproducible deuterium discharges are obtained and steadily sustained by microwaves (power up to 1.5 kW CW) in the electron cyclotron frequency range (2.45 GHz), injected perpendicularly to the magnetic field lines in O-mode polarization. The resonance at 0.0875 T is located in a single position close to the end of the vessel, opposite to the RF power launcher. The diagnostic setup is based on electrostatic probes spatially distributed for coherent structures detection (at present it is with 8 tips), on an imaging system using a fast framing camera and an image intensifier unit for direct visualization of the plasma structures (up to 200 kframes/sec). The plasma column has a radius of ~10 cm. The coatings have been exposed by a sample insertion system (Fig. 1) that consists of the sample head, the sample manipulator and the sample exchange chamber. The sample head is attached to the manipulator allowing the withdrawal of the sample from its exposure position to the sample exchange chamber. This sample exchange chamber can be isolated from the main vacuum chamber via a gate valve. The sample temperature was monitored by a thermocouple fixed directly on the sample holder. In all experiments, the measured temperature of the sample holder during the exposure process was in the range of 300–330 K.

All samples have been exposed to D_2 plasmas at a working pressure of 6.7×10^{-2} Pa with a total gas flow rate fixed at 30 sccm. The plasma state was driven by a 2.45 GHz RF generator at 1.5 kW of power. In this experimental condition, by mean of the Langmuir probe I–V characteristics, the following plasma parameters were found: plasma density ≅ 5.6×10^{16} m^{-3}, plasma potential ≅ 12 V, electron temperature ≅ 4.4 eV. The ion flux Γ_i was calculated by the Bohm criterion [14,15] which yields

$$\Gamma_i = n_+ \exp(-0.5)\sqrt{\frac{kT_e}{m_+}}$$

where n_+ is the ion concentration, T_e is the electron temperature, m_+ is the mass of incident ion and k is the Boltzmann's constant. The ion flux was 5×10^{20} m^{-2} s^{-1} and with 1 h of plasma exposure the ion fluence was determined in ≅ 10^{24} m^{-2}.

2.4. Deuterium retention analysis

The near surface deuterium retention was determined with ex-situ ion beam analysis using Elastic Recoil Detection Analysis (ERDA) by the D(^{4}He,D)^{4}He reaction for near surface depth profile determination. In this technique, a monoenergetic He^+ beam with incident energy of 2.15 MeV impinges on the sample at grazing incidence (15° from the surface), and the energy spectrum of the forward-scattered deuterons and protons at 30° from the beam is obtained using a surface barrier detector. In this process, forward scattered He^+ are filtered out using a Mylar stopping foils (≅ 12 μm). Parallel to the ERDA also the Elastic Backscattered particles were collected at 160° from the beam to determine the stoichiometry of the WN_xO_y surface layer.

3. Results and discussion

3.1. Deposition and characterization of WN_x coatings

Tungsten nitride coatings were prepared by diode sputtering in Ar + N_2 gas mixtures obtained by setting the total gas flow to 20 sccm at different Ar/N_2 flows. Based on our previous experience [16] in delamination mechanisms of thin films, in order to have mechanically stablecoatings, all coatings have been deposited in a bi-layer structure. The underlying layer is 800 nm thick and shows