

Ion beam micro analysis of deposits at tokamak divertor surfaces

P. Petersson^{a,*}, H. Bergsaker^b, G. Possnert^a, J.P. Coad^c, S. Koivuranta^d, J. Likonen^d

^a Tandem Laboratory (Association EURATOM-VR), Uppsala Universitet, P.O. Box 256, 751 05 Uppsala, Sweden

^b Div. of Fusion Plasma Physics (Association EURATOM-VR), KTH, SE-10044 Stockholm, Sweden

^c EURATOM/UKAEA Fusion Association, Culham Science Centre, Abingdon, OX14 3DB, UK

^d Association EURATOM-TEKES, VTT, P.O. Box 1000, 02044 VTT, Espoo, Finland

ARTICLE INFO

Article history:

Available online 10 March 2010

Keywords:

Nuclear reaction analysis

Micro-beam

Deuterium

Beryllium

ABSTRACT

Cross sections of deposited layers in the Joint European Torus (JET) were analysed using the nuclear micro analysis at the Tandem Laboratory, Uppsala University. For deuterium and beryllium the nuclear reactions $^2\text{D}(^3\text{He,p})^4\text{He}$ and $^9\text{Be}(^3\text{He,p}_n)^{11}\text{B}$ were exploited for analysis. Typically the analyses have been made with 10 μm spatial resolution and a sensitivity of better than one atomic percent for beryllium or deuterium in carbon matrix. Comparing several different surface treatment techniques shows that polishing the sample surface give very good optical surface information but that some amount of deuterium and beryllium probably is removed. For good quantitative results the measurement can either be done on a rough surface or the top of the polished surface can be cut off.

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1. Introduction

Plasma-surface interactions are a major area of investigation in the development of controlled nuclear fusion as a source of energy. The materials that are considered for plasma facing components in the international tokamak experimental reactor (ITER) [1] are tungsten, beryllium and carbon fiber composites (CFC) [2,3]. The use of more than one plasma facing material entails a problem in that materials that are eroded in some wall areas are re-deposited and mixed in other areas. The formation of thick deposited layers is of great concern, firstly since they may incorporate large amounts of tritium that is hard to locate and remove, secondly since the break up of thick deposited layers leads to dust production, which in turn constitutes a safety problem, as well as a potential operational problem [2]. Mixing of materials may also alter the surface properties in undesired ways.

The present aim of the JET tokamak program is mainly to support ITER [3]. JET has operated since 1992 with an internal divertor at the bottom of the vessel, which has been redesigned several times. Open magnetic field lines intersect surfaces in the divertor and this is where plasma-surface interactions are most intense. In operations intervals in 1999, 2001, 2004 and 2007, divertor tiles were replaced and samples of the surfaces could be used for studies of the effects of erosion and deposition. The plasma facing wall in JET in this period was CFC, with periodic deposition of beryllium by evaporation. Normally erosion is dominating in the outer regions of the divertor, while deposited layers are built up on the in-

ner parts [4]. Due to chemical re-erosion of carbon in the plasma exposed deposited layers, they are enriched in beryllium [5]. The layers incorporate the main plasma constituent deuterium, at varying levels of concentration, as well as oxygen, hydrogen and traces of stainless steel components. Since the deposition is cumulative the concentration profiles throughout the layers should be correlated with the history of plasma operations.

Ion beam analysis methods have always been extremely useful for surface studies in fusion research. The accessible depth limits their usefulness for very thick deposits. Secondary ion mass spectrometry (SIMS) as well gets impractical with layers that are many tens or even hundreds of μm thick. In order to access thick layers it is convenient to cut cross sections of the layers. Optical micrographs of polished layer cross sections from the JET divertor reveals a rich phenomenology [6]. For quantitative elemental analysis of the cross sections, nuclear reaction analysis (NRA) with ion micro-beam is an attractive option [7]. For simultaneous detection of hydrogen isotopes also elastic recoil detection (ERD) with micro-beam is useful [8]. However, as the first measurements on polished cross sections showed lower beryllium and deuterium content than ion beam analysis of the original surfaces, it was felt that the sample preparation needed further attention. This report aims at clarifying the influence of cutting and polishing on the elemental composition in the layer cross sections.

2. Experimental

Tile four in octant one of the JET divertor was removed from the JET divertor during the JET shut down in 2007, having been exposed to JET plasma operation since 1998. Cross-sectional samples

* Corresponding author.

E-mail address: Per.petersson@angstrom.uu.se (P. Petersson).

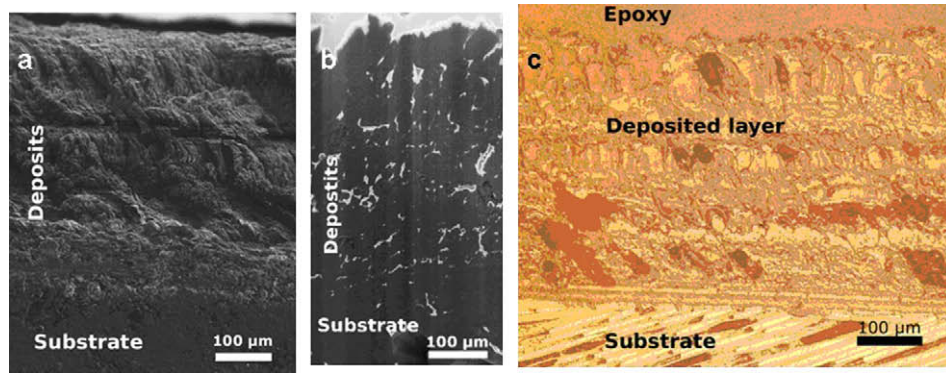


Fig. 1. Images of the cross sections from the same sample. SEM image of unpolished rough surface (a) and polished surface (b) where bright regions are related to the insulating properties of epoxy. Optical images of polished sample (c) where a lot of features are visible.

for microscopy and micro-beam analysis were cut out at position 4/7 in the sloping part of tile four [6]. One of the samples was placed into cold mounting epoxy (Epofix, Struers) and polished with the Struers Tegrast system as described in [6], using a virtually water free diamond suspension (DP-suspension). An adjacent piece was left with raw cut edges.

Fig. 1a shows a SEM micrograph of the raw, unpolished layer cross section. There are two discontinuities parallel to the surface, which may correspond to the JET shut downs in 2001 and 2004. Fig. 1b shows a SEM image of the polished cross section. The bright spots within the layer are probably epoxy. Fig. 1c is an optical image of the polished surface, showing very clearly a laminar, layered structure of the deposit. The micro-beam analysis was performed for comparison on the raw cross section of the deposited layer, on the original top surface, on the polished cross section and on a small area after removing some of the polished cross section surface by cutting with a sharp razor blade. Fig. 2 shows a SEM image of the cross section after the cutting where the substrate has a rougher structure compared to both the deposits shown in the same figure and the polished surface seen in Fig. 1b. The white spots related to epoxy have also been removed by the cutting away the top surface.

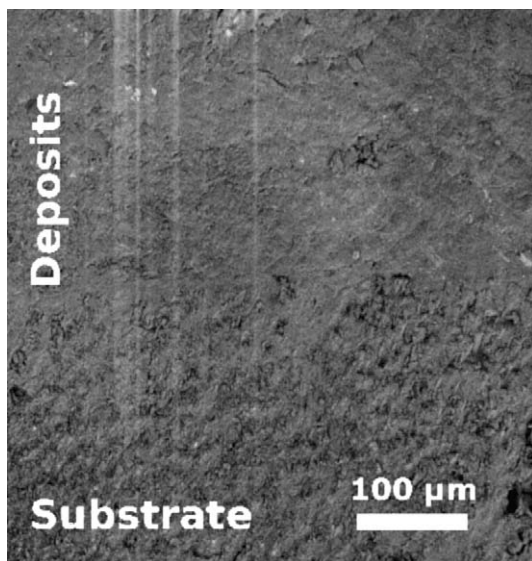


Fig. 2. SEM image of an area of a polished sample where the top layer has been cut off using a razor blade. The carbon fibers in the substrate give a rougher surface compared to the deposited layer.

The ion beam analysis facility is set up at the 5 MV tandem pelletron accelerator at the Ångström Laboratory in Uppsala. The micro-beam line consists essentially of an adjustable rectangular aperture followed by a transport distance of about 6 m and a triplet quadrupole magnetic lens, which focuses a reduced image of the aperture onto the sample [9]. The beam was scanned stepwise over 256×256 pixels with $30 \mu\text{s}$ dwell time at each spot. The control signals for the sweep were sampled in coincidence with collected particles (nuclear reaction products from the sample surface). For spatial calibration the beam was scanned over Au or Cu grids of known dimensions. Typically the achieved spatial resolution was $10 \mu\text{m}$ in the direction perpendicular to the original surface layer and $20\text{--}100 \mu\text{m}$ in the tangential direction. With that beam size the achievable beam current was in the range of 100 pA . For simultaneous measurements of beryllium and deuterium and carbon, the nuclear reactions $^3\text{He}(^9\text{Be}, \text{p}_n)^{11}\text{B}$, $^3\text{He}(^{12}\text{C}, \text{p}_n)^{14}\text{N}$ and $^3\text{He}(^2\text{D}, \text{p})^4\text{He}$ are used, with 3 MeV ^3He beam and the different peaks and their corresponding reactions shown in Fig. 3. Depending on the exact sample composition and density the accessible depth is $8\text{--}12 \mu\text{m}$ for D analysis and roughly half that for Be.

All data were collected in list mode, to allow for off line sorting of the data. To create two-dimensional maps, all events were selected in the energy ranges corresponding to the different elements in the plot. The elemental concentrations were then calculated making the assumptions that carbon is the dominant material in the deposited material and that deuterium and beryllium only occurs in small amounts. This assumption should be valid for most of

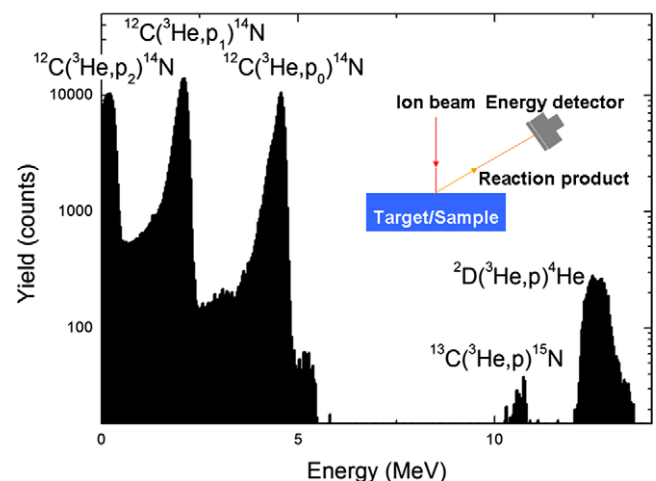


Fig. 3. Typical energy spectrum for ^3He interacting with a target containing carbon and deuterium.

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