

Carbon deposition on beryllium substrates and subsequent delamination

R. Mateus^{1,*}, P.A. Carvalho^{1,2}, N. Franco^{1,3}, L.C. Alves^{1,3}, M. Fonseca⁴, E. Alves^{1,3}

*Corresponding author: rmateus@ipfn.ist.utl.pt

Associação Euratom/IST

¹IPFN, Instituto de Plasmas e Fusão Nuclear - Laboratório Associado, Instituto Superior Técnico, Av. Rovisco Pais, 1049-001 Lisboa, Portugal

²ICEMS, Departamento de Engenharia de Materiais, Instituto Superior Técnico, Av. Rovisco Pais, 1049-001 Lisboa, Portugal

³ITN, Instituto Tecnológico e Nuclear, Estrada Nacional 10, 2686-953 Sacavém, Portugal

⁴CFNUL, Centro de Física Nuclear, Universidade de Lisboa, Av. Prof. Gama Pinto 2, 1649-003 Lisboa, Portugal

Introduction

Pure beryllium, carbon and tungsten parts will be used as plasma facing components in future fusion devices. However, mixed layers and compound formation will occur in the component surfaces during reactor operation due to erosion, re-/co-deposition and heat-load events. As result, the properties of the exposed surfaces will evolve in time and depend on the phase transformations arising from the interaction with the plasma [1,2,3]. The experimental results also evidence that re-/co-deposits tend to flake and peel-off from the substrate [4,5] and contribute to the formation of dust agglomerates in the plasma [5]. The present work studied the Be-C system and shows that the compound formation promotes by itself the occurrence of dust emission events.

The Be-C system was studied previously by depositing C thin layers in Be substrates. The formation of beryllium carbide (Be₂C) starts at about 557 K at the interface and is strongly promoted at higher temperatures [2,3]. A fast formation of beryllium oxide (BeO) at the surface is also evident in the same temperature range [2,3]. Heat loads can induce a compressive stress and the blistering of deposited thin films [7]. Compounds are easily formed at the blisters due to their lower thermal conductivity. The presence of distinct phases presenting different lattice parameters and thermal expansion coefficients lead to the fracture and removal of the blisters in the surface. The phenomena could be visualised by following the annealing route via electron microscopy.

Sample preparation

120 nm carbon films were evaporated in pure beryllium plates

with an electron beam evaporator
polished beryllium plates (Goodfellow, 99.8+ at.% / SiC paper 4000 mesh)
graphite rod (99.0+ at.%) used as carbon source

Samples were annealed

in the 373 to 1073 K range during 90 min with temperature steps of 100 K

Analytical techniques

Rutherford Backscattering (RBS)
X-ray Diffraction (XRD) – grazing geometry
Scanning Electron Microscopy (SEM)
Secondary Electron (SE) and Backscattered Electron (BSE) analysis
Energy-dispersive X-ray spectroscopy (EDS)

Results and discussion

Rutherford Backscattering (RBS) and X-ray Diffraction (XRD)

- Slight modifications of the Be, C and O RBS yields between 373 and 773 K.
- The Be-C reaction becomes very fast at higher temperatures and it is complete after the annealing at 1073 K.
- Be seems to diffuse through the carbide layer and oxidises at the surface.
- SEM analysis detected a blistered and an unblistered zone in the sample annealed at 973 K (see Fig.3.c). RBS data reveal that the blistered zone presents a higher O content.

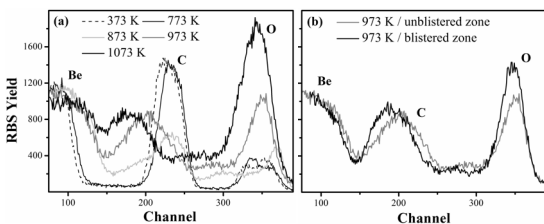
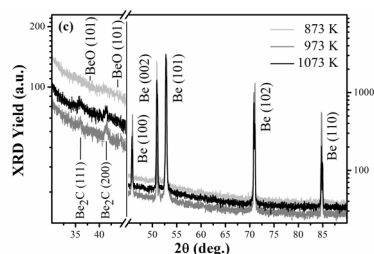


Fig. 1. Typical RBS spectra of the superficial films after the annealing route: at 373 K and in the 773 to 1073 K range (a); at 973 K in the blistered and unblistered zones of this sample (b);

Annealing temperature (K)	Thickness of the reactive layer (nm)	Elemental content in the film for	
		Be (10 ¹⁵ at./cm ²)	O (10 ¹⁵ at./cm ²)
373	118	0	121
773	117	94	138
873	330	2891	217
973	392	3060	455
1073	584	4238	993



- The presence of Be₂C is confirmed in the films annealed above 873 K by XRD analysis.
- However, the BeO signature is not visible in the spectra, suggesting a different preferential location for the BeO component in the films.

Fig. 2. Be₂C signature in the X-ray diffraction pattern at higher temperatures (c).

Microstructural analysis by SEM (SE, BSE and EDS mapping)

- The annealing route induced the formation of *telephone-cord like* blisters resulting from a thermal compressive stress of the deposited films (Fig.3).
- A carbide formation reaction front became apparent at 873 K and higher temperatures (see Fig.3b to 3d).
- The blisters delaminates from the films annealed above 873 K (Fig.3e and 3f), whereas the films annealed at lower temperatures remain unchanged.
- SE/BSE images in Fig.4 confirm that C is the only component of the films after annealing at 773 K (see Fig.4c), and BeO formation starts at 873 K due to Be diffusion through the C layer (see Fig.4f).

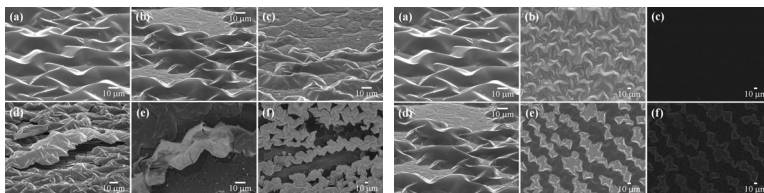


Fig. 3. SE images of the blisters formed in the films after the annealing route at 773 K (a), 873 K (b), 973 K (c) and 1073 K (d); BSE images revealing a long term delamination behaviour of the films annealed at 973 K (e) and at 1073 K (f).

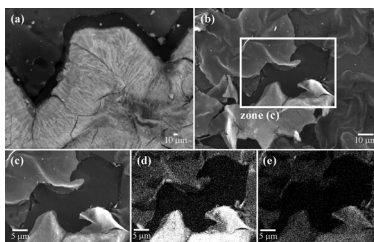


Fig. 4. Films surfaces after annealing: 773 K: SE image (a), equivalent SE (b) and BSE (c) top views; 873 K: SE image (d), equivalent SE (e) and BSE (f) top views.

Conclusions and future work

From the present work becomes evident that the formation of compounds in the C-Be (and O) system promotes by itself the delamination of re-deposited layers.

It is foreseen the use of carbon parts in the divertor components of future fusion reactors [1,5] and it will be advantageous to study the reverse Be-C system by depositing and annealing Be films in graphite substrates.

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