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Carbide formation in tungsten coatings on carbon-fibre reinforced carbon substrates

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ABSTRACT

Tungsten coatings with molybdenum interlayer deposited on carbon-fibre reinforced carbon (CFC) substrates were selected as the first wall material for the divertor in the Wall Project at Joint European Torus (similar to the International Thermonuclear Experimental Reactor). For such a layered structure, diffusion of carbon from the CFC substrate towards the Mo and W deposits is expected during the operation of the reactor. As both molybdenum and tungsten form stable carbides, brittle compounds may form at the interface, thus strongly affecting the thermomechanical performance of the coated tiles. For the purpose of prediction of the operation time of such coated tiles, carbon diffusion and carbide formation kinetics need to be determined.
In the present study, W/Mo/CFC samples were subjected to heat treatment at 1470 K for various annealing times. The Focused Ion Beam technique was used for sample preparation for electron microscopy examinations. Transmission electron microscopy observations supported with diffraction pattern analyses revealed the both W_2C and WC carbides in the W coating, as well as that of Mo_2C carbide in the Mo layer. The results were used to estimate the kinetics of coatings degradation.

1. Introduction

The development in nuclear fusion science began in the 1950s and has since then been under constant progress. The next step on the road to fusion power plant is the International Thermonuclear Experimental Reactor (ITER) project whose main scientific goal is to deliver ten times the power it consumes [1] — and the first of all fusion experiments to produce net energy. During the first operation period of the ITER reactor according to the current design plasma facing components shall be made of beryllium (on the first wall) and of tungsten and carbon-fibre reinforced carbon (CFC) (in the divertor) [2]. Further operation of ITER, including the use of tritium as a fuel, might require a full W divertor [3]. However, so far there is no available data on the performance of the combination of these materials in a fusion environment. For that purpose the ITER-like Wall Project was launched at Joint European Tokamak with the goal to test the combination of these materials at the most ITER-relevant parameters accessible today [4–6]. In the current ITER-like wall project, tungsten lamellae stacks are installed in the load-bearing septum replacement plate and W coatings on CFC in the other divertor regions [7].

In order to verify feasibility of using W coatings, various coating techniques were investigated in a coordinated R&D program launched in 2005 [8–10]. As a result of this program 10–15 μm thick W coatings with 2–4 μm Mo interlayer produced by combined magnetron sputtering and ion implantation (CMSII) technique were selected as the most reliable [11]. Later, in 2009 this technique was modified to produce 20–25 μm W coatings. These technological achievements need to be supplemented with a proof of thermal stability of the W coatings on a carbon-rich substrate, particularly because no carbon diffusion barrier is foreseen in the present coatings concept. At the same time a strong effect of the tungsten carbides formed via precipitation driven by the carbon diffusion on the thermo-mechanical properties of the coatings has been observed [12]. In this context, the main motivation of this work was to investigate the carbide formation in these Mo/W coatings on CFC substrates exposed to high temperatures in a model laboratory environment. It has been assumed that such investigations shall provide estimates of the carbon diffusion rate in such a particular system and thus, in turn, of the safe operation time of such coated tiles.

2. Experimental details

Bi-directional CFC substrates were coated with 2–4 μm molybdenum interlayer and subsequently 10 μm tungsten film. The deposition was carried out by CMSII technology at the National Institute for Laser, Plasma and Radiation Physics in Bucharest. A detailed description of the process is given elsewhere [11].

Coated samples with dimensions 10×10×3 mm were annealed at 1470 K for 0.5 h, 1 h, 2 h, 5 h and 20 h under Argon atmosphere (the furnace was purged with Ar three times at room temperature and three times at 670 K) to avoid tungsten oxidation during annealing. Each sample was enveloped with a thin tungsten foil to eliminate possible contact

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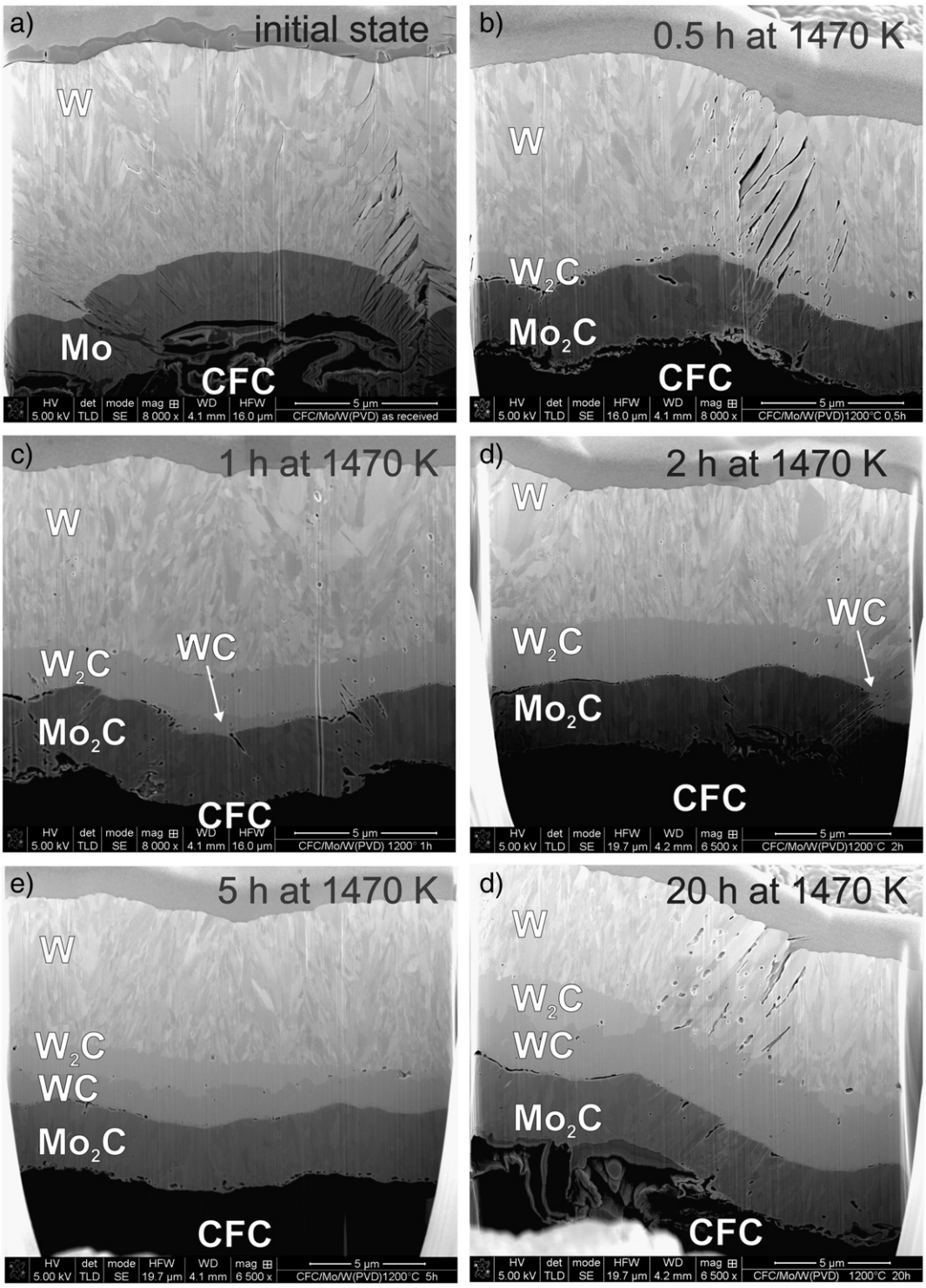

Fig. 1. Cross-section SEM images of the coatings: as-deposited (a) and after annealing at 1470 K for 0.5 h (b), 1 h (c), 2 h (d), 5 h (e) and 20 h (f).

with carbon. In order to estimate the kinetics of carbon diffusion from the CFC substrate to the W coatings, measurements were carried out of the thickness of tungsten carbides formed as a result of the annealing. To this end, cross sections were prepared and observed using a FEI Helios NanoLab 600 dual beam–focused ion beam/scanning electron microscopy (FIB/SEM). Since in the FIB/SEM dual beam column is at an angle of 52° to the sample surface, a correction factor was used equal 1/cos(38°). The platinum protection coating was deposited using 0.92 nA beam current for 7 min. The cross-sectioning was performed in a three step process — rough cross-section milling with maximum current of 21 nA, and two subsequent cleaning millings with ion current of 6.5 nA and 0.92 nA, respectively.

The mass contrast in the SEM was used to distinguish between tungsten and tungsten carbide in individual SEM images. The carbide phase composition was determined by nano diffraction in Hitachi HD-2700 Cs corrected dedicated STEM equipped with 200 kV Schottky electron source. STEM observations were carried out on thin lamellas prepared by the FIB lift out method.

To determine the degradation of coating's mechanical properties due to brittle carbide formation, a micro scratch test was performed