ELSEVIER

Journal of Nuclear Materials 301 (2002) 233–236

journal of nuclear materials

www.elsevier.com/locate/jnucmat

Letter to the Editors

Chemical analysis of flakes from the Joint European Torus

H. Kleykamp *

Institut für Materialforschung I, Forschungszentrum Karlsruhe, Postfach 3640, 76021 Karlsruhe, Germany

Received 28 September 2001; accepted 6 December 2001

Abstract

Measurements were performed on flakes from the JET with the Mark IIa divertor installed during the 1996 operation period by wavelength dispersive X-ray microanalysis in the element range from Be to the actinides and by X-ray diffraction. Metallic and ceramic particles up to 0.5 mm diameter were observed consisting of Al, Al_2O_3, Al–Mg alloys, austenitic steel, Mg_2Si and Ni base alloys. The major phase is stratified graphite up to 50 μm thickness with layers of Al and Fe up to 2 μm thickness on the surface and inside the redeposited flakes. The graphite contains further about 3% oxygen. The carbon fraction is higher than 80% of the total material. The origin of the redeposits is explained.

1. Introduction

The plasma–wall interactions in nuclear fusion machines result in erosion processes from the torus walls and the highly loaded divertors and limiters due to evaporation, arcing, sputtering, plasma disruption, etc. The material is subsequently redeposited on other spots of these components, part of which peels off in form of flakes and dusts and accumulates on the floor of the torus. To understand the origin and the composition, chemical analyses were carried out on these flakes, which had been produced in the JET machine during about 2000 plasma pulses under tritium operation in the period April to October 1996 [1]. The Mark IIa type divertor is provided with carbon fibre reinforced graphite (CFC) tiles and the poloidal limiters with fine-grained graphite. The torus consists mostly of Inconel 600 and is equipped with aluminum oxide coated antennas incl. Al_2O_3 insulator disks for plasma diagnostic purposes.

2. Experimental

The material was collected from the inner floor of the divertor region and contained about 10% of the total tritium inventory of JET. The flakes transported in capsules to Forschungszentrum Karlsruhe in 1997 amounted to about 2 g corresponding to 8 MBq tritium per g flakes. A local correlation of the inventory to distinct positions of the torus was not possible [2,3].

The element analyses on material from the capsules JET-2-5 (not annealed after collection) and JET-2-1 (annealed up to 1100 °C/3 h/air) were carried out with the β–γ-shielded X-ray microanalyser JRXA 50/JSM 6400 of IMF I. The instrument facilitates quantitative analyses in the range between beryllium and the actinides. The detection limit of the elements is about 0.02% with the exception of beryllium and increases up to 3% if this element is present as BeO and Be_2C due to the high mass absorption coefficients of the Be Kα radiation in oxygen and carbon. Therefore, Be cannot be detected in form of small BeO inclusions or of a diluted dispersion in carbon [4]. Further, an X-ray diffraction analysis was made with the β–γ-shielded X-ray diffractometer of the Hot Cells of Forschungszentrum Karlsruhe on non-annealed flakes of the capsules JET-2-9 and JET-3-3, A beryllium containing crystalline phase with a mass fraction less than 3% should be detectable by this method [5].

* Tel.: +49-7247 82 2888; fax: +49-7247 82 4567.
E-mail address: heiko.kleykamp@imf.fzk.de (H. Kleykamp).

0022-3115/02/$ - see front matter
PII: S0022-3115(02)00715-8

3. Results

3.1. Non-annealed sample JET-2-5

The light-optical microstructure and an electron-optical section of embedded and polished material are illustrated in Fig. 1. The phases up to 500 μm in diameter consist of metallic Al, Ni-base alloys (phase d in Fig. 1), Al–Mg and two-phase Al–Mg_2Si precipitates as well as of Al_2O_3, Ca–K alumino silicates and Fe silicides. The melt dripped down along the Inconel 600 and other surfaces to the torus floor during the plasma disruptions. The quantitative analysis of selected droplets and fragments is compiled in Table 1. The dominating fraction of the flakes is composed of redeposited stratified carbon layers up to 500 μm length and 50 μm thickness containing very thin (<2 μm) intermediate metallic layers. The dominating metals are Al and Fe with an overall composition of about 1% of the carbon flakes, both metals in pyramidic cake structure. Other laminated particles are carbon fibre reinforced carbon fragments. The carbon flakes contain on an average 3% oxygen probably bonded to the metals or chemisorbed to carbon. The origin of the Al and Fe layers and of oxygen inside the graphite are Al_2O_3 and steel which were atomised to the elements during the plasma pulses.

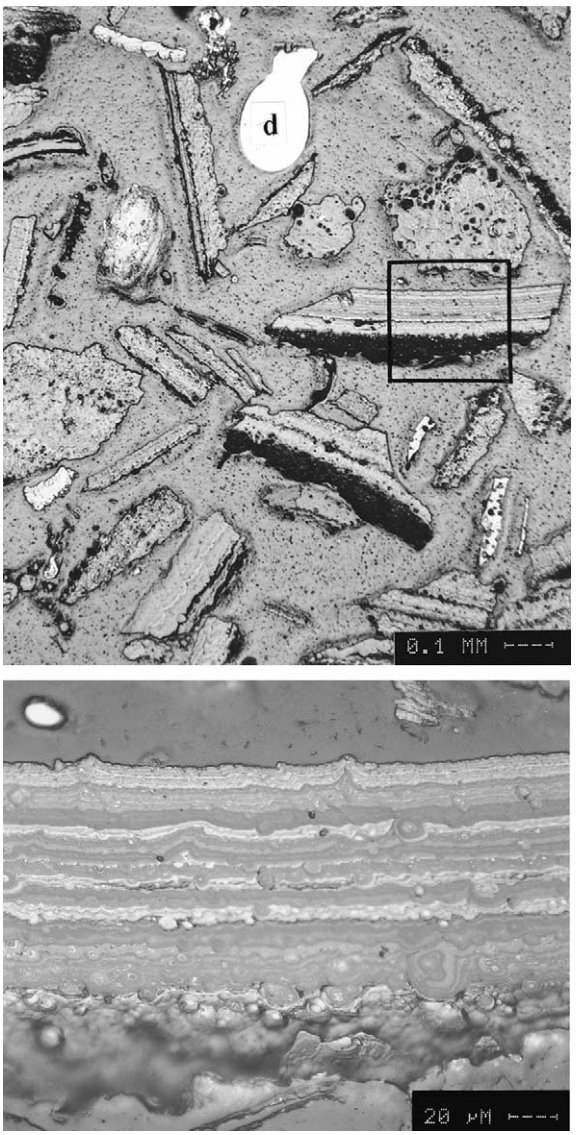

Fig. 1. Light-optical microstructure and electron-optical section of carbon flakes and metallic Al and Ni–Cr (phase d) droplets, non-annealed sample JET-2-5.

Table 1
Quantitative analysis of precipitates in flakes of Fig. 1, sample JET-2-5, concentrations in mass%

Element	Composition						
	Al	Al	Al_2O_3	≈Inconel 600[a]	Al(Mg)	Al–Mg_2Si	Al–Mg_2Si
Al	99	99	52	0	94	91	91
Mg	0	0	0.1	0	3.7	4.6	3.3
Si	0.1	0.2	0	0.6	1.0	3.0	4.2
O	0	0	47	0	0	0	0
Fe	0.7	0.6	0	4.9	1.2	0.9	0.9
Cr	0.3	2	0	23	1	0.6	0.8
Ni	0.1	0.1	0	72	0	0	0

[a] Phase d in Fig. 1.

3.2. Annealed sample JET-2-1

Morphology and composition of the air-annealed flakes are similar to those of the non-annealed flakes, however, the total mass was reduced by 32% after annealing due to carbon combustion. Fig. 2 illustrates the laminated pyramidic cake structure of redeposited carbon with intermediate Al layers inside the flakes. Single-phase and multi-phase Al–Cr–Fe–Ni droplets are the dominating redeposits.

3.3. Non-annealed samples JET-2-9 and JET 3-3

The X-ray diffraction analysis was conducted on two non-annealed samples of the capsules JET-2-9 and JET-3-3 using Cu $K\alpha_1$ radiation and an additional monochromator between sample and detector. The results are compiled in Table 2. The positions of the X-ray diffraction lines evidence the presence of graphite

(0 0 2 reflex), Al, Al_2O_3 and Ni-base alloys (Inconel 600). The existence of BeO could not be proved by X-ray diffraction.

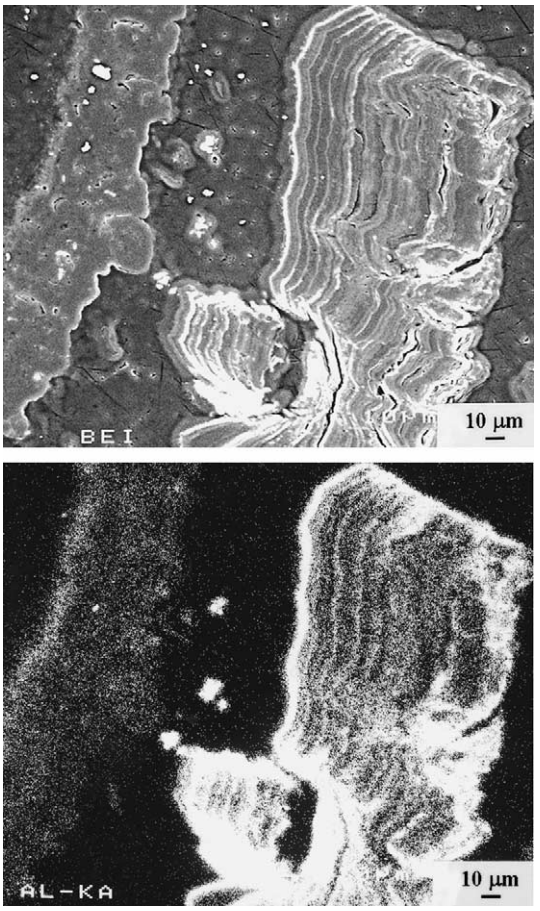

Fig. 2. Electron-optical (BEI) microstructure and Al distribution image of the pyramidic cake structure, annealed sample JET-2-1.

4. Discussion

The Mark IIa divertor consists of an Inconel 600 support structure containing an arrangement of large carbon fibre reinforced carbon composite tiles [1]. Inconel and aluminium droplets, laminated graphite flakes and CfC fragments have been observed preferably in the divertor range of the inner torus floor. The existence of Be or BeO could not be proved due to the poor detection limit of beryllium by X-ray microanalysis [4]. The fracture process of the Al_2O_3 debris is probably caused by thermal expansion differences with the supports. The considerable amounts of aluminium and oxygen originate form Al_2O_3 which is atomised to the elements during the plasma pulses. The oxygen atoms are trapped in the graphite flakes, also probably dissociated H_2O vapour which has been incorporated into the torus during inspection could be a reason for contamination. The total composition of the flakes was estimated from the multitude of the microanalyses on sample JET-2-5. The result is compiled in Table 3. The flakes contain more than 80% carbon and about 3% oxygen. The alternating C and Al layers in Fig. 2 reflect the time-shifted erosion–redeposition processes during the plasma pulses.

Table 3
Composition of non-annealed flakes on the floor of the divertor region, sample JET-2-5, estimated results from X-ray microanalysis

Element	Concentration (mass%)
C	>80
O	≈3
Al	5
Ni	3
Fe	2
Cr	2
Mg	0.2
Si	0.1
Be	<Detection limit

Table 2
X-ray diffraction analysis of flakes, samples JET-2-9 and JET-3-3, Cu Kα_1 radiation, lattice spacing d in pm

JET-2-9			JET-3-3			$h\,k\,l$	Phase
d (pm)	Relative intensity	Lattice parameter (pm)	d (pm)	Relative intensity	Lattice parameter (pm)		
335.20	100	$c = 670.4$	335.62	100	$c = 671.2$	0 0 2	Graphite
233.69	72	$a = 404.8$	233.33	74	$a = 404.1$	1 1 1	Al
205.02	74	$a = 355.1$	205.34	72	$a = 355.7$	1 1 1	Ni(Cr)
177.72	40	$a = 355.4$	–	–	–	2 0 0	Ni(Cr)
159.85	44	(Hexagonal)	–	–	–	1 1 6	Al_2O_3
139.80	–	–	–	–	–	–	–
125.37	37	$a = 354.6$	–	–	–	2 2 0	Ni(Cr)
121.71	33	$a = 403.7$	122.02	28	$a = 404.7$	3 1 1	Al
93.91	31	–	–	–	–	–	–

References

[1] A.T. Peacock, P. Andrew, P. Cetier, J.P. Coad, G. Federici, F.H. Hurd, M.A. Pick, C.H. Wu, J. Nucl. Mater. 266–269 (1999) 423.
[2] H.D. Roehrig, Forschungszentrum Karlsruhe, personal communication, 23 June 1998.
[3] R. Rolli, H. Werle, E. Kaiser, R. Pejsa, Forschungszentrum Karlsruhe, internal report, 1998.
[4] H. Kleykamp, J. Anal. At. Spectrom. 14 (1999) 377.
[5] R. Pejsa, B. Neufang, A. Wacker, Forschungszentrum Karlsruhe, internal report, 1998.