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The impact of specific surface area on the retention of deuterium in carbon fiber composite materials

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Abstract –

In this study, the PISCES-A linear plasma instrument has been used to characterize retention in several carbon fiber composites in order to better understand the factors which lead to elevated retention levels in these materials. The PISCES instrument is capable of subjecting materials to intense fluxes (up to 10^{22} $m^{-2}s^{-1}$) of low energy (150 eV) D^+ ions, producing conditions similar to those encountered by plasma facing components in a fusion reactor. In this investigation, three CFCs (fabricated with different manufacturing processes) are compared with the N11 composite used in the Tore Supra reactor. The specific surface areas for these materials were within the range of 0.14–0.55 m^2/g. The plasma bombardment conditions were adjusted to provide doses on the order of 10^{25}–10^{26} $m^{-2}s^{-1}$ at a sample temperature of 200 °C. After removal from PISCES-A, the amount of D retained in the sample surface was determined via thermal desorption spectroscopy. The measured retention showed a strong correlation with the type of material used and the corresponding BET surface area. By using a CFC with a lower internal porosity, one could expect a reduction in retention by a factor of 5 or more.

Motivation and Background

Recent studies of retention by Roth et al. [1] in the PISCES-A instrument show no saturation with increasing fluence in the NB31 and N11 composites. Depth profiles from these studies indicate a long diffusion tail into the bulk. Would CFC materials with lower surface-connected porosity yield better results? To compare directly with the results of Roth, we attempted to reproduce the experimental conditions of Ref. 1 as closely as possible.

Experimental Configuration:

- We used the PISCES-A instrument at UCSD to expose three samples to a low energy, high flux plasma.
- Retention determined by TDS.
- Sample analysis included SEM and BET measurements performed at Idaho National Laboratory.

Exposure Conditions:

- Flux: 2×10^{22} $m^{-2}s^{-1}$
- Ion energy: 130 eV
- T_{sample} = 200 °C

[1] J. Roth, V.Kh. Alimov, A.V. Golubeva, R.P. Doerner, J. Hanna, E. Tsitrone, Ch. Brosset, V. Rohde, A. Herrmann, and M. Mayer, *J. Nucl. Mater.*, **363-365**, 822-826 (2007).

Carbon Fiber Composite Test Matrix

- We exposed three different carbon fiber composites to the PISCES-A plasma.
- These samples included K-Karb (manufactured by Kaiser Compositek) and Hitco CC139C and CC389L.
- We also measured the BET surface area of CFC N11.
- SEM indicated small gaps between matrix and fibers, which may contribute to surface connected porosity.

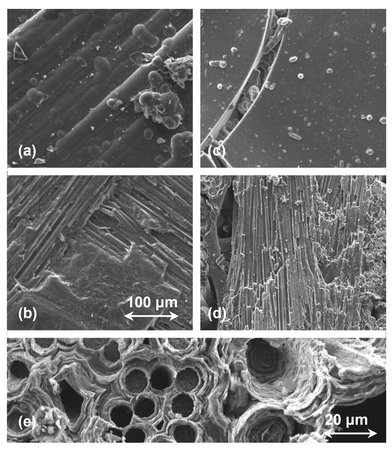

Fig. 1: SEM images showing the fiber structure of the plasma exposed surfaces of the CFC tiles: (a) Hitco CC139C (b) Hitco CC389L (c) K-Karb (d) N11, showing side view of fiber bundle (e) N11, showing end view of fiber bundle. The 100 µm scale applies to images (a)-(d), and the 20 µm scale applies to image (e).

Thermal Desorption Spectroscopy

We used thermal desorption spectroscopy (TDS) to ascertain the amount of D retained in each of the CFC samples.

- Final temp: 1000 °C; 8 °C/s ramp.
- K-Karb and Hitco CC139C samples showed similar desorption behavior, with a peak at approximately 475 °C.
- The main desorption peak shifts to a lower temperature for Hitco CC389L (380 °C), although a component of the compound desorption curve also peaks at 475 °C.
- This could suggest a different trapping mechanism associated with higher BET surface area.

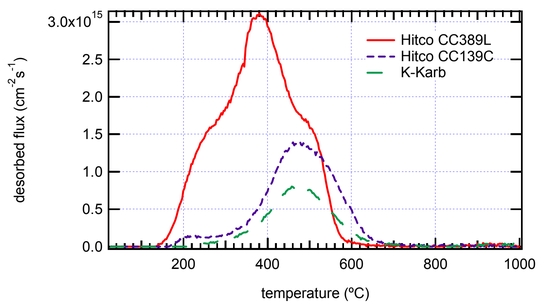

Fig. 2: Thermal desorption profiles for CFC materials exposed in PISCES-A. Experimental conditions were: flux: 2×10^{22} $m^{-2}s^{-1}$; fluence: 5×10^{25} m^{-2}; T_{sample}=200°C

BET Measurements

The Brunauer-Emmett-Teller (BET) gas adsorption technique was used to determine the specific surface area of the samples.

Experimental Analysis:

- Measurements obtained with a Micromeritics ASAP 2000 instrument with Kr gas serving as the adsorptive.
- Adsorption isotherms for Hitco and K-Karb composites show a marked increase in the volume of gas adsorbed (V_a) at a pressure ratio $P/P_o\approx0.3$. (A non-porous material would be characterized by a linear increase in V_a until $P/P_o\approx0.9$.)
- Composite N11 shows a much more abrupt change in V_a at $P/P_o\approx0.3$.

BET isotherms indicate each material contains a population of mesopores (>2 nm in diameter) in addition to a population of macropores (>50 nm in diameter).

BET Results:

- The specific surface area was determined from the linear region of the transformed isotherms; see figure (b) to the right.
- BET surface areas for the materials described in this study ranged from 0.14-0.55 m^2/g (CFC N11 had a BET surface area of 0.37 m^2/g.)
- This value is large compared to the specific surface areas reported for pyrolytic graphite.

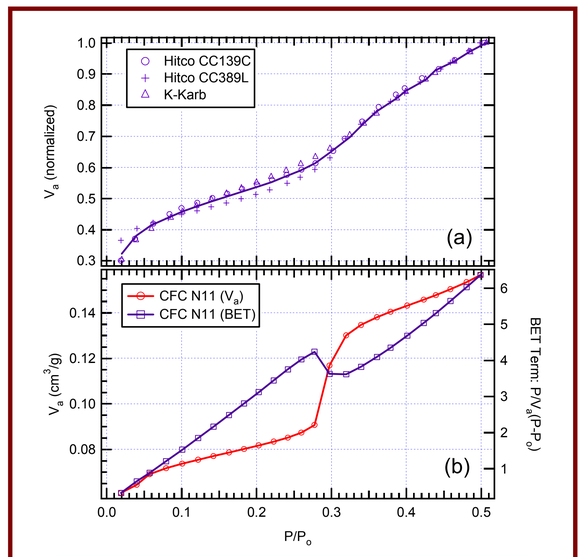

Fig 3: BET isotherms for (a) Hitco and K-Karb composites and (b) CFC N11. Also shown in (b) is the transformed isotherm. The specific surface area is determined from the linear region ($0<P/P_o<0.25$) of this plot.

Retention Results

The retention data show a strong correlation with BET surface area. These results are summarized in the table below.

- The Hitco CC389L composite, which has the highest specific surface area, also retains the most D during an exposure.
- Retention in Hitco CC139C and K-Karb under the same conditions is lower by factors of 2.6 and 4.6, respectively.

Comparison with CFC N11:

- The composites used here are compared with NB31 and N11 in Fig. 4. Data from Roth et al. [1].
- We note that both Hitco CC139C and K-Karb perform much better than N11 from a retention standpoint and have a much lower BET surface area.
- This result suggests that materials with low BET surface areas (~0.15 m^2/g) could reduce tritium inventory in PFCs.
- Comparison with ion beam data is more difficult due to lower fluxes. If extrapolated to higher fluences, NB31 data from Roth et al. suggests very high retention.
- Further high flux measurements are needed for the NB31 material.

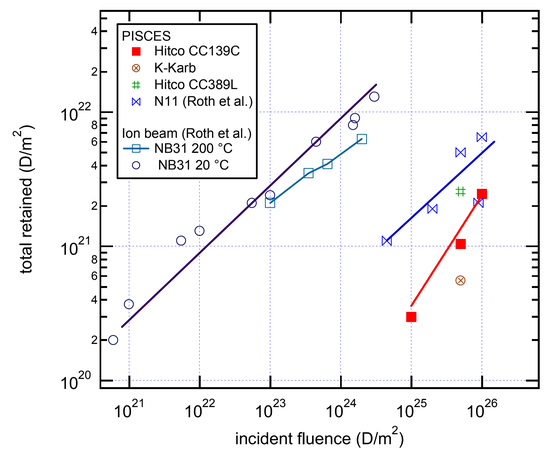

Fig. 4: Fluence dependence of retention in CFC materials. Data from present work and prior study by Roth et al.

CFC type	BET surface area (m^2/g)	Fluence (m^{-2})	Retention (m^{-2})	Retained Fraction
K-Karb	0.14	5×10^{25}	5.6×10^{20}	1.1×10^{-5}
Hitco CC139C	0.15	1×10^{25}	3.0×10^{20}	3.0×10^{-5}
		5×10^{25}	1.0×10^{21}	2.0×10^{-5}
		1×10^{26}	2.5×10^{21}	2.5×10^{-5}
CFC N11	0.37	--	--	--
Hitco CC389L	0.55	5×10^{25}	2.6×10^{21}	5.2×10^{-5}

Summary

In this study, we have characterized the specific surface area of several CFC materials. For the PISCES-A exposure conditions considered, a strong correlation with BET surface area was observed. This indicates that surface connected porosity is an important factor in determining the overall retention and suggests that tritium inventory issues may be mitigated to some extent through a careful consideration of specific surface area.

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