Preparation of Ti-doped carbons for fusion applications

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INTRODUCTION

Carbons materials can be used as plasma-facing components of fusion devices because they exhibit high thermal shock resistance, absence of melting, low radiation of carbon atoms from the central plasma, high thermal conductivity, low erosion due to energetic atoms of hydrogen and oxygen, etc.

The production of self-passivating materials using graphites doped with metallic carbides is a good method to improve properties.

Metal doped graphites can be obtained mixing Mesocarbon Microbeads with carbides particles, the properties improving as particle size decreases because a more homogeneous material is achieved.

However, the production of doped-carbon materials via copyrolisis of a carbon precursor with an heteroatom source soluble in the carbon precursor should produce doped carbons of an homogeneous nanosize particle distribution.

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EXPERIMENTAL

Petroleum residue

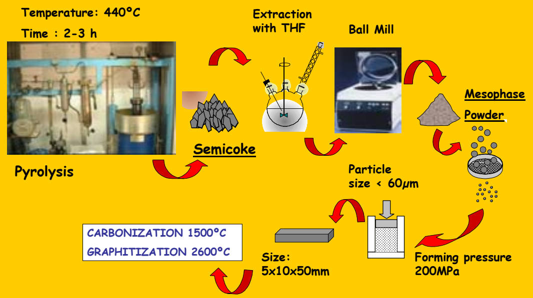
Titanium Butoxide [$Ti(OC_4H_9)_4$] → Mixtures with 0-2 wt% Ti

Pyrolysis conditions

Pressure : 1 MPa

Temperature: 440°C

Time : 2-3 h



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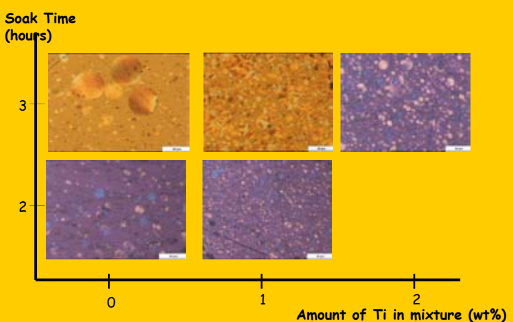
RESULTS (Semicoke analysis)

Sample	Temp (°C)	Pressure (MPa)	Time (h)	Ti Mixture (wt%)	THFI (wt%)	Mesophase content (%)	Ti semicoke (wt%)	FTIR CHar/CHal
440-1/3 0% Ti	440	1	3	0	56	50	0	1.38
440-1/3 0.5 % Ti	440	1	3	0.5	61	55	1.2	1.40
440-1/3 1 % Ti	440	1	3	1	67	54	2.6	1.45
440-1/3 2 % Ti	440	1	3	2	74	59	5.6	1.74

- Titanium Butoxide (Ti $(OC_4H_9)_4$ decomposes during pyrolysis (300-350°C) to give TiO_2 particles



- The presence of TiO_2 particles favours the nucleation of mesophase, leading to a more homogeneous distribution of mesophase spheres

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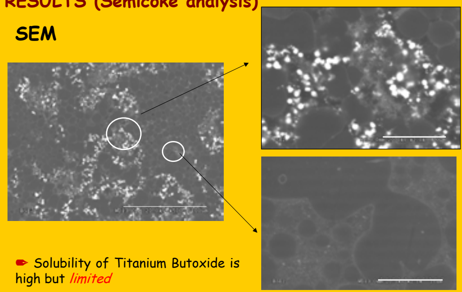
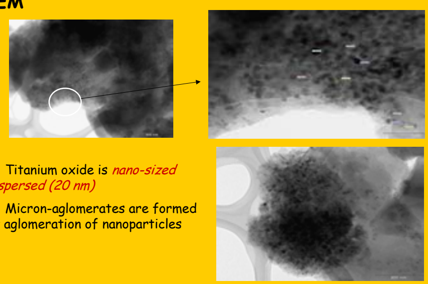
RESULTS (Semicoke analysis)

SEM



- Solubility of Titanium Butoxide is high but *limited*
- Titanium oxide is homogeneously distributed but it *forms aglomerates < 2 μm*
- A better dispersion is achieved using ultrasonic stirring

TEM



- Titanium oxide is *nano-sized dispersed (20 nm)*
- Micron-aglomerates are formed by aglomeration of nanoparticles

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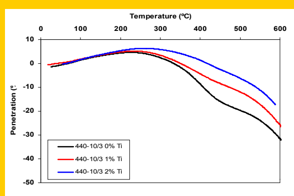
RESULTS (Mesophase powder analysis)

Sample	TI (wt%)	NMPI (wt%)	ß-resin (wt%)	Ti (wt%)	Volatile matter (1000°C) (wt%)	C/H_{at}	FTIR CHar/CHal
440-1/3 0% Ti	91	80	11	0	12	2.03	2.3
440-1/3 0.5 % Ti	91	85	6	1.9	12	2.04	2.4
440-1/3 1 % Ti	92	85	7	3.7	12	2.04	2.5
440-1/3 2 % Ti	98	85	13	7.2	12	2.14	3.1

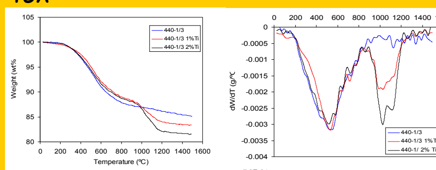
- Ti-doped mesophase powders have higher aromaticity and C/H_{at} ratio

TMA



- The amount of Titanium affects the viscous flow behavior during thermal treatment

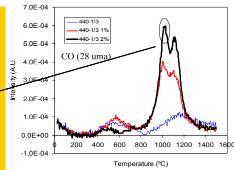
TGA



Reaction:

$TiO_2 + 3\ C \rightarrow TiC + 2\ CO$ (900-1200°C)

Intermediate compound ($Ti_xO_yC_z$)



- During thermal treatment *TiO_2 reacts with C to give TiC and CO*. The analysis of CO by mass spectrometry indicates that the reaction takes place between 900-1200°C. The formation of an intermediate TiOC compound is detected.
- This reaction encreases the amount of volatiles evolved and generates porosity, the effect being larger with increasing $Ti(OC_4H_9)_4$ content

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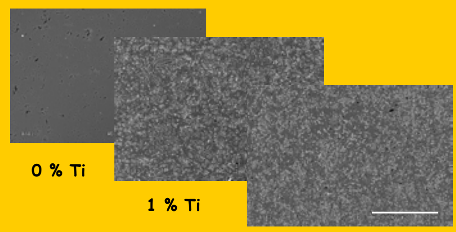
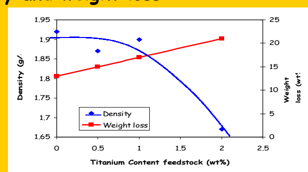
RESULTS (Graphitised samples 2600°C)



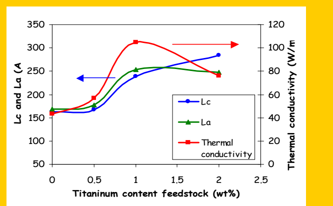
- A good dispersion of Titanium is achieved
- TiC particle size is lower than 2μm
- Porosity of the graphite increases for initial Ti content >2 wt%.
- Flexural strength for the graphites is higher than 90 MPa.

Density and weight loss



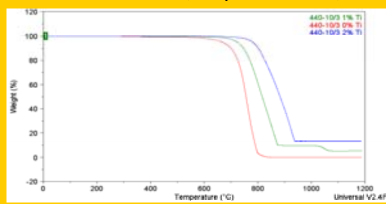
- Density decreases for Ti content > 2% due to high volatile matter evolution caused by TiO_2 reaction with C

Thermal conductivity and Lc



- The coherent crystal stack height (Lc) and thermal conductivity increase with titanium content
- The decrease in thermal conductivity for 2wt% Ti is caused by the defects in the graphite (lower density, higher porosity)

Oxidation resistance (samples 1500°C)



- Preliminary oxidation resistance tests (samples 1500°C) indicates an enhancement of the oxidation resistence with Ti content

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CONCLUSIONS

- $Ti(OC_4H_9)_4$ decomposes during pyrolysis to form in-situ TiO_2.
- TiO_2 acts a nucleating agent to form mesophase spheres
- Titanium remains in the carbon after extraction to separate mesophase spheres
- TiO_2 reacts with C at 900-1200°C to give TiC and CO.
- Loss of CO generates porosity, the effect increasing with titanium content
- Doped graphites have a higher Lc and thermall conductivity

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