Characterization of hydrogenated and deuterated silicon carbide films codeposited by magnetron sputtering

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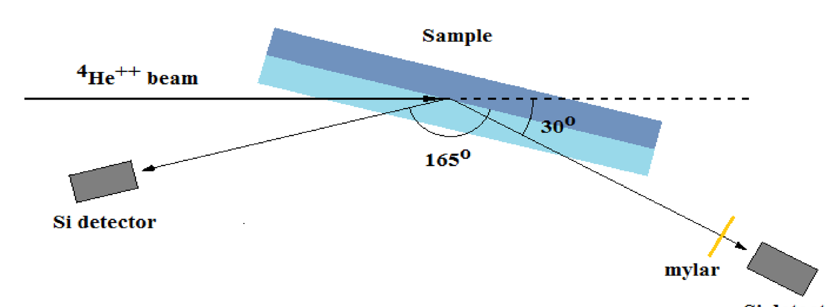
Introduction

Silicon carbide is a material with many applications, including fusion reactor technology [1] and also fission power reactors.

In the present work we will focus on investigations by Elastic Recoil Detection technique of the H/D content in silicon carbides thin layers deposited on silicon substrate. Deposition was performed by dual magnetron co-sputtering using Ar and mixtures of Ar/H_2/D_2 as working gases. Deposition using only Ar were performed at various discharge powers for identifying the parameters for deposition of SiC_X thin films with controlled stoichiometry. The material properties of the deposited thin films were investigated using specific techniques (profilometry, SEM, AFM, FTIR and spectroscopic ellipsometry).

RBS and ERDA Measurements:



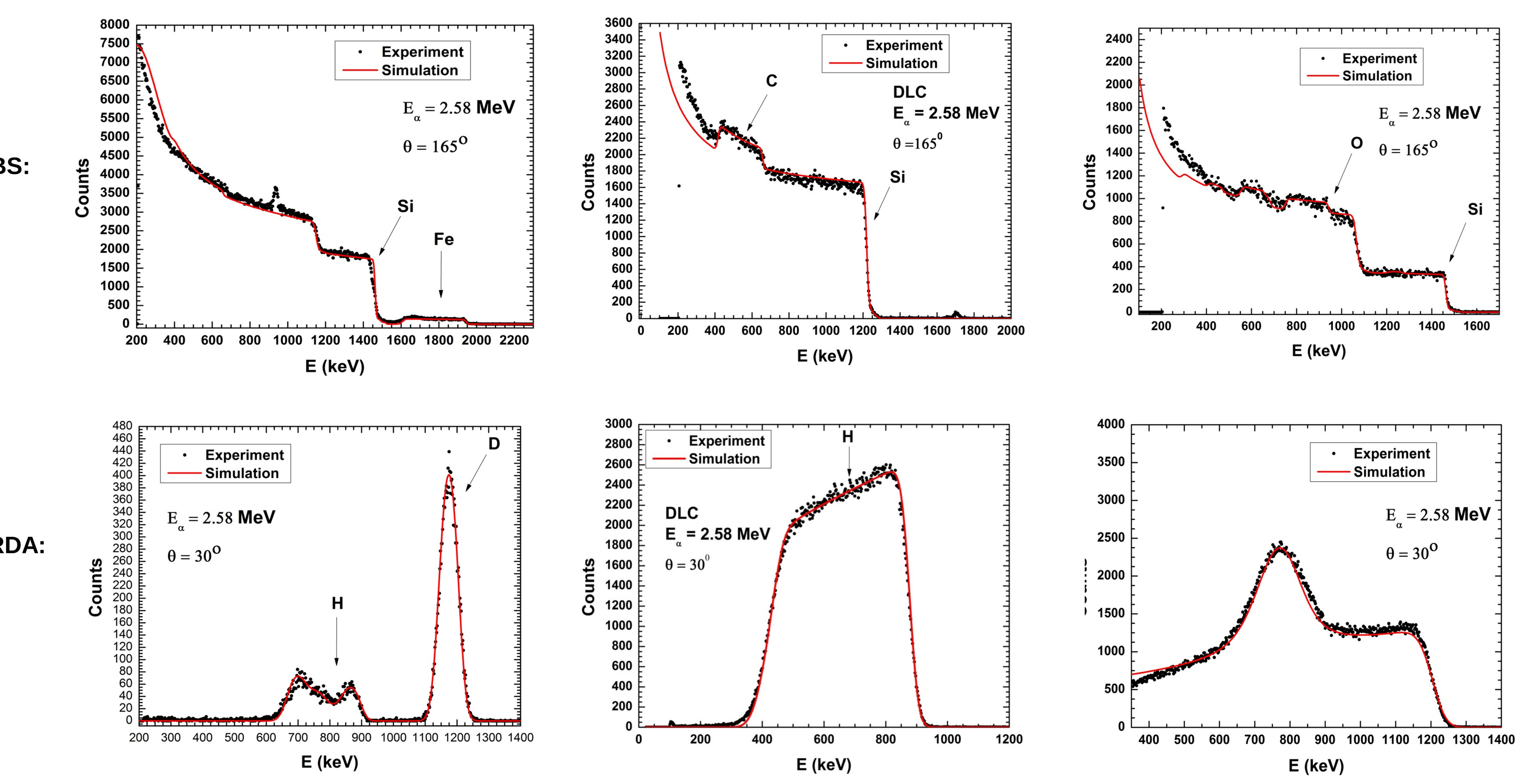
Mylar thickness: 11,35 µm

The SIMNRA code [2] was used for fitting the data

Non Rutherford calculated cross section tables [3] were used in the simulation code for H and D characterisation

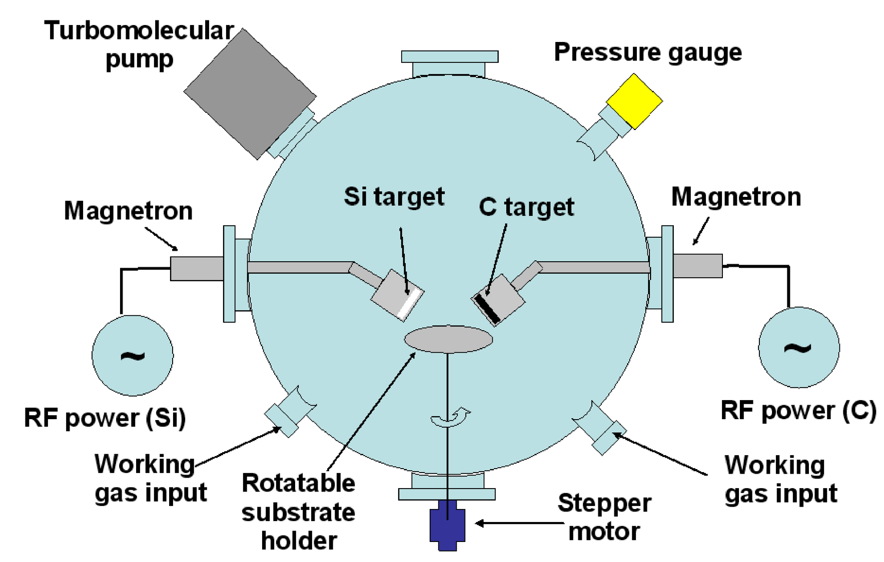
RBS:

ERDA:



Samples preparation:

Deposition setup:



- Deposition of SiC thin films is performed by simultaneously co-sputtering of two targets (Si and C);
- The substrate is located on a rotateable holder.

Deposition parameters:

Samples deposited in Ar:

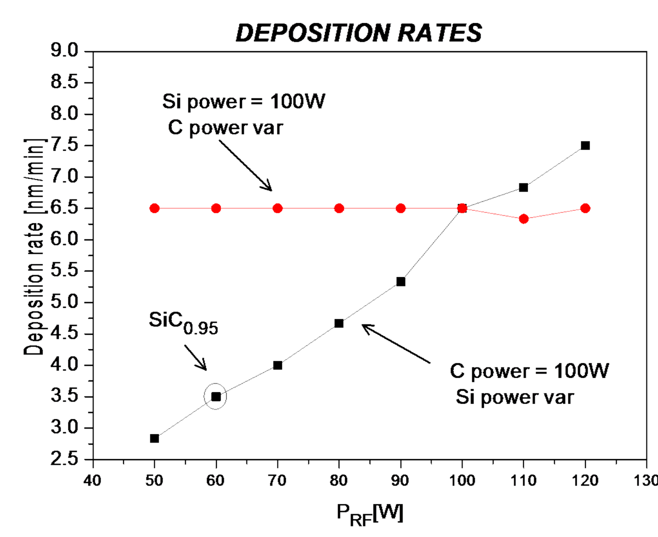
(40sccm Ar, 4.5*10^{-2}Pa, 3 hours deposition)

Batch 1 :
- P_{RF}Si =100W;
- ~~P_{RF}C =(50-130W, steps of 10W);~~

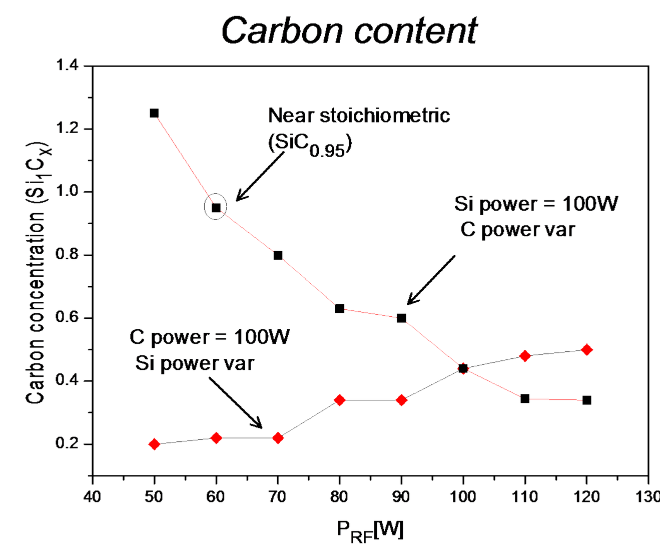
Batch 2:
- P_{RF}C =100W;
- P_{RF}Si =(50-130W, steps of 10W);

Samples deposited in Ar mixed with H_2 and D_2 :
(mixture pressure: 4.5*10^{-2}Pa;
P_{RF}Si=60W, P_{RF} C=100W, i.e $SiC_{0.95}$)

- Ar + 10%, 20%, 40% and 60% H_2;
- Ar+20% D_2;
- Ar+10% D_2+10% H_2.

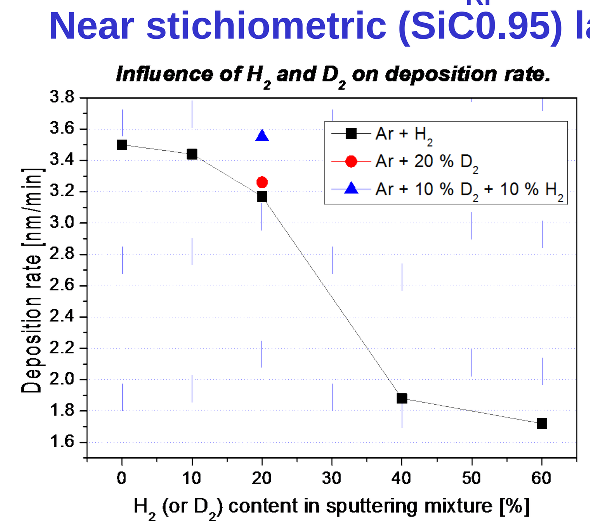


Tuning of deposition rate is possible varying the power applied to Si target.



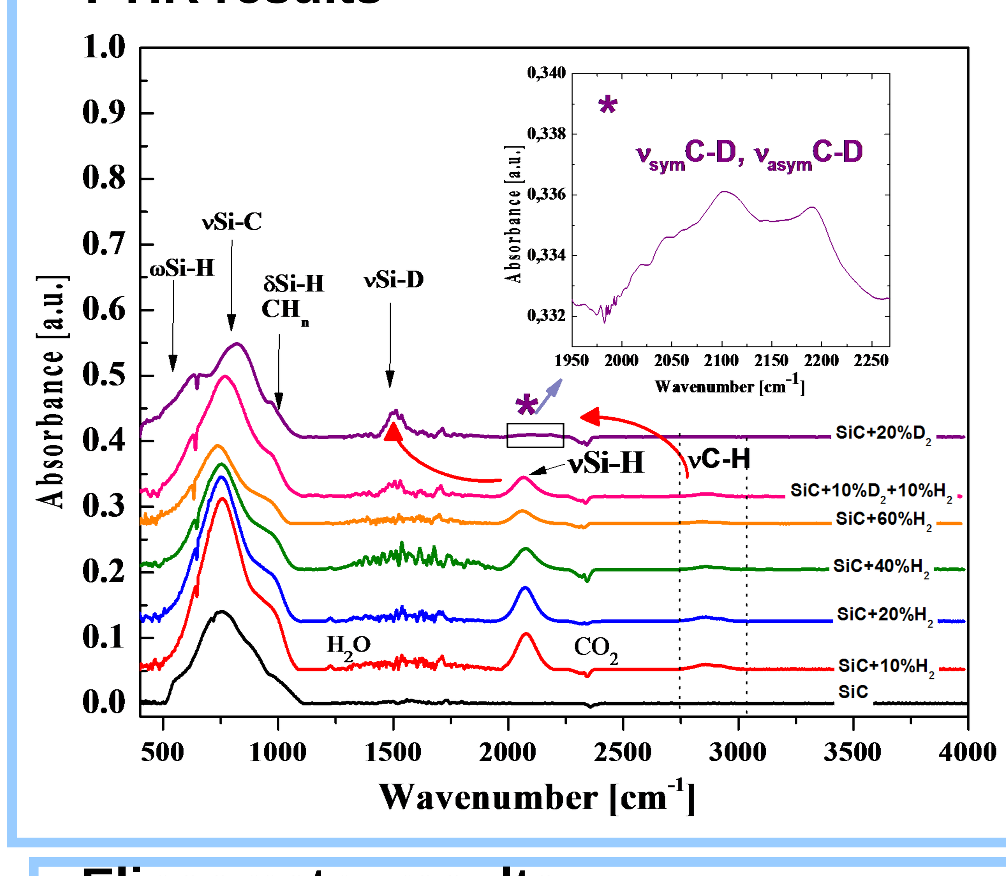
Stoichiometry of the SiC_X layers is more efficient varied adjusting the power applied to C target

P_{RF}Si=60W and P_{RF}C=100W :
Near stichiometric (SiC0.95) layers are grown with a rate of 3.5 nm/min.



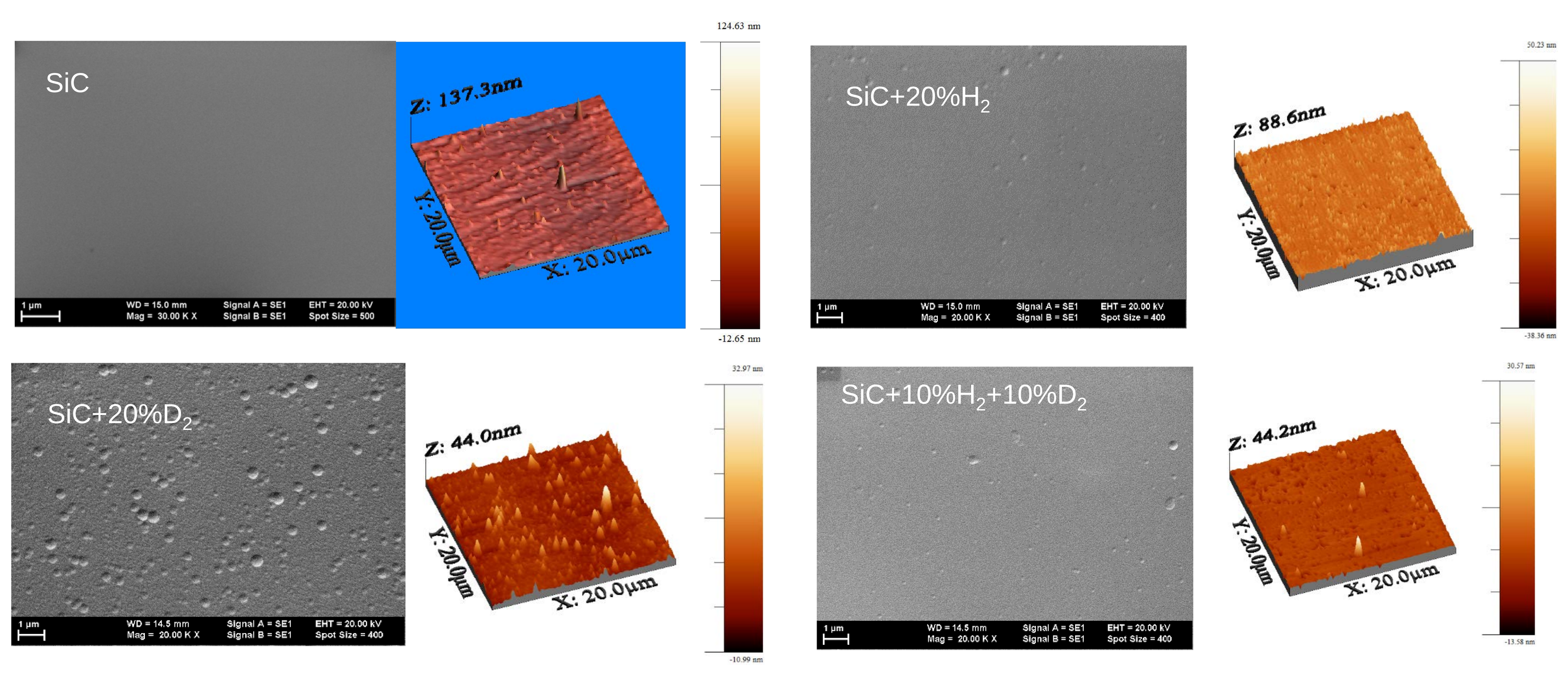
- **The increase of the H_2 content in the sputtering mixture leads to a slow decrease of the deposition rate;**
- **H_2 and D_2 seems to produce the same effect over the deposition rate (compare the rates at 20%H_2, 20%D_2 and 10%H_2+10%D_2).**

FTIR results



- **The sample deposited only in Ar present specific spectral signatures [4,5]:**
  - 600-650 cm^{-1}: ω Si-H wagging mode
  - 650-900 cm^{-1}: ν SiC stretching vibrations.
  - 950-1100 cm-1: CH_n rocking or wagging modes;
- **Addition of H_2 to deposition mixture leads to:**
  - 2700-3150 cm^{-1}: ν C-H stretching modes;
  - 2090 cm^{-1}: ν Si-H stretching band.
- **Addition of D_2 produce the following isotopic shifts (represented by red thick curved arrows on the graph):**
  - ν Si-H 2090 cm^{-1} →ν Si-D (1550cm^{-1});
  - ν C-H (2700-3150 cm^{-1}) → ν C-D (1950-2250 cm^{-1}).

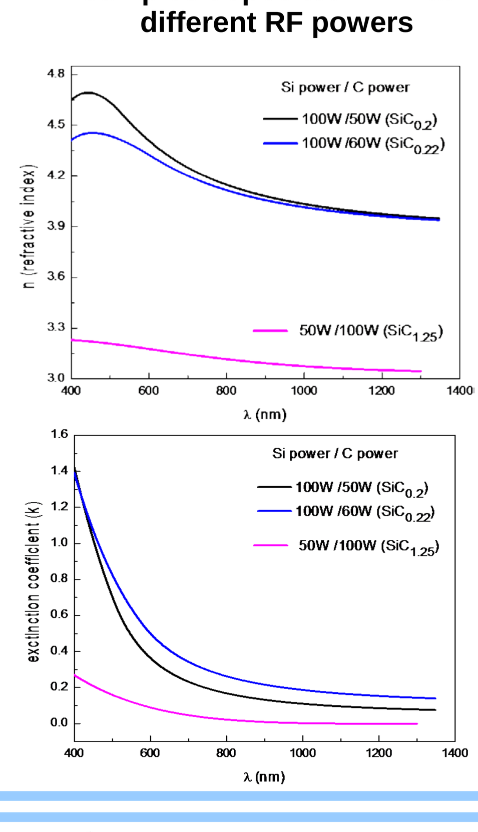
SEM and AFM results



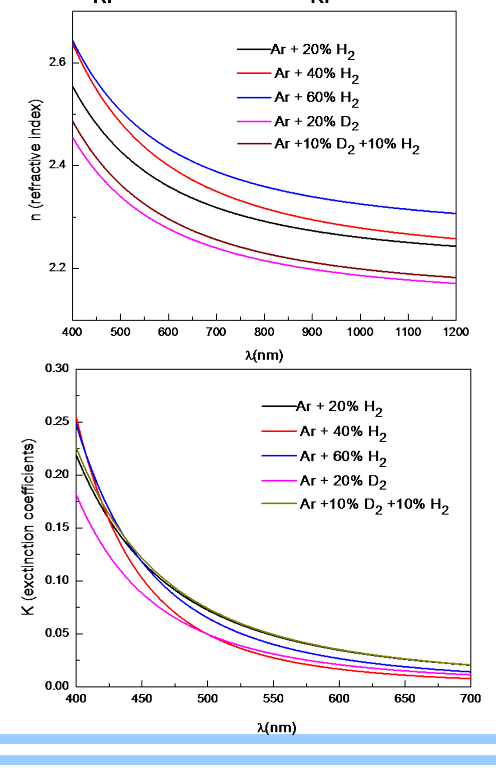
- The sample deposited only in Ar present a pretty smooth surface, slightly influenced by variation of the deposition process parameters (namely RF applied powers);
- Addition of both H_2, D_2 in the deposition gas leads to roughening of the samples surface, possible due to etching effect of the hydrogenoid species produced in the discharge.

Elipsometry results

Samples deposited in Ar at different RF powers



Samples deposited in Ar+H_2+D_2 mixtures at P_{RF}Si=60W and P_{RF} C=100W



By proper choice of the RF powers applied to both Si and C targets or of the deposition gaseous mixture, the refractive index and extinction coefficient (right) of the SiC_X thin films deposited only in Ar can be easily adjusted over a large range:

n= 2.2-4.5 and k= 0.02-1.6.

Conclusions:

- Elemental composition and thickness of silicon carbide samples were investigated by RBS. The hydrogen and deuterium content were determined by ERDA;
- The samples were prepared by simultaneously sputtering of two targets (silicon and graphite) using Ar or mixtures of Ar with H_2 and D_2 as working gases;
- The dependence of the SiC_x layers stoichiometry over the experimental deposition parameters was investigated;
- The sample deposited only in Ar present a pretty smooth surface, slightly influenced by deposition process parameters; addition of both H_2, D_2 leads to roughening of the samples surface, possible due to etching effect of the hydrogenoid species produced in the discharge.
- FTIR investigations of the samples revealed the presence of D-C-D and H-C-H chemical bonds in the samples codeposited using Ar/D_2 and Ar/H_2 mixtures, respectively;
- Spectroscopic Elipsometry investigations revealed that optical properties of the deposited SiC layers can be easy adjusted by proper choosing of the deposition process parameters.

References:

[1] H Nakamura, J Dietz, P Ladd, Wall conditioning in ITER, Vacuum, 47, 6-8, 653-655, (1996)
[2] SIMNRA home page, http://home.rzg.mpg.de/~man/
[3] Nuclear Instruments and Methods in Physics Research B 261 (2007) 401–404
[4] George Socrates, *Infrared and Raman Characteristic Group Frequencies*, Third Edition, John Wiley &Sons, LTD, 2001.
[5] L. Calcagno, F. Giorgis, A. Makthari, P. Musumeci, F. Pirri, Philosophical Magazine B 79 (1999) 1685-1694.

Acknowledgements

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