Hydrogen analysis in CNW and WS2 nanotubes, using NRA technique

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Beam time request (unit=8 hours) - 18 units (6 days)

Desired Period- 01.10.2011-15.11.2011

Desired beam properties:

Type- $^{19}F^{4+}$, $^{15}N^{3+}$

Energy (MeV)- 16-18 MeV for $^{19}F^{4+}$ and 13-15 MeV for $^{15}N^{3+}$

Intensity(p/nnA)- 30 nA for $^{19}F^{4+}$ and 5 nA for $^{15}N^{3+}$

Vacuum Requests- 10-6 Torr

Nuclear reaction analysis(NRA) for ^{1}H determination in solid material like carbon nano-walls(CNWs), and WS2 nanotubes will be investigated using the following nuclear reactions: ^{1}H (^{19}F, αγ)^{16}O at the resonant energy in the range 16-18 MeV and ^{1}H(^{15}N αγ,)^{12}C in the range 13-15 MeV. The ^{19}F reaction has the advantage that it using natural (as opposed to isotopically enriched) F in the accelerator ion source while the ^{15}N reaction has the advantage of having the best combination of analytic characteristics (depth resolution and sensitivity). This experiment will use the 5 line of the Tandem accelerator.

Introduction

Carbon-nanowalls (CNWs) can be described as two-dimensional graphite nanostructures with edges comprised of stacks of plane graphene sheets standing almost vertically on the substrate. These sheets form a wall structure with a high aspect ratio. The thickness of CNWs ranges from a few nm to a few tens of nm. The large surface area and sharp edges of CNWs may prove useful for a number of applications such as electrochemical devices, field electron emitters, storage materials for hydrogen gas, catalyst support. In particular, vertically standing CNWs with a high surface-to-volume ratio, serve as an ideal material for catalyst support for fuel cells and in gas storage materials.

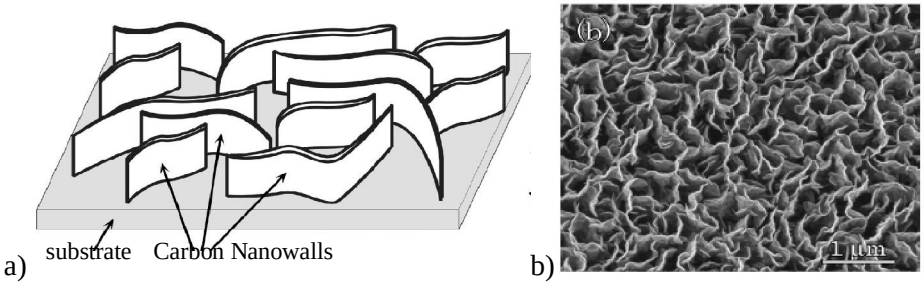

Fig. 1. a) Schematic diagram of CNWs, b)SEM image of CNWs.

The CNWs are produced by our partner, National Institute for Laser, Plasma and Radiation Physics, using the plasma enhanced chemical vapor deposition technique, fig 2.

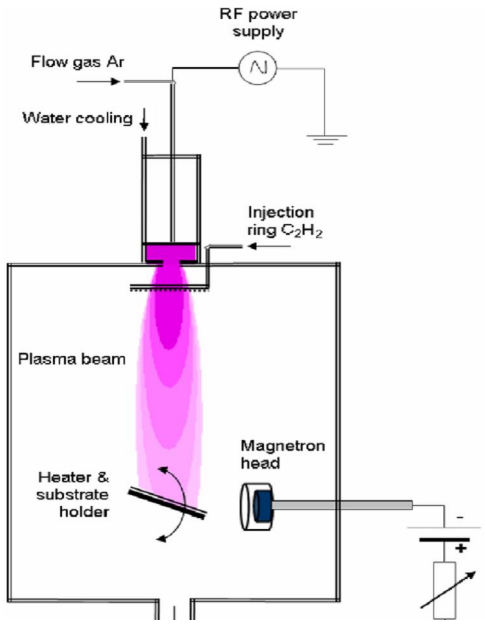

Fig. 2. Scheme of the experimental setup

A radiofrequency (RF) plasma source (13.56 Mhz, RF power 50-500 W) is mounted on the top of a cylindrical reaction chamber and generates a plsma beam oriented along the vertical axis. A homemade DC magnetron is mounted laterally with its axis oriented horizontally. A substrate holder is placed at the crossing of the two axes and comprises a substrate heater. The substrate holder can be rotated to expose the substrate either to the magnetron plasma or to the radiofrequency plasma beam. Details on the plasma beam source were given elsewhere. The RF discharge is generated by introducing argon (Ar) in a small discharge chamber containing two electrodes, one of them acting as a nozzle. The discharge expands through the nozzle from the generation zone into the deposition chamber due to a pressure gradient as a bright, long plasma beam. Acetylene (C_2H_2) diluted with hydrogen (H2) is introduced in the expanding Ar plasma beam via an injection ring. The injection ring is positioned 6 cm above the substrate holder. The peculiarity of this deposition system is that the discharge and the deposition regions are spatially separated. The carbon containing radicals are formed in the injection zone and are deposited onto the heated Ni catalyst.

WS2 nanotubes are very interesting structures, with various electronics applications. The observed strength with respect to the Young's modulus was found to be exceedingly high in comparison to conventional high strength materials. WS2 nanotubes can be implemented in nanoelectronics, fuel cells, ultra-filtration membranes and catalysts. For example, since these nanotubes are semi-conductors they can be used in products such as advanced high resolution flat panel displays and as tips for atomic force microscopes (AFMs). The optical properties of

the inorganic nanotubes enable numerous other applications in the fields of nanolithography, photocatalysis and other fields. These materials are bought from NanoMaterials Ltd (Israel).

Hydrogen depth profiling in solids

Over the past decade, due to the development of improved hydrogen analysis methods, extensive studies of hydrogen in materials have found that the presence of hydrogen can have dramatic effects on the electrical, mechanical and chemical properties of many materials. Among many possible techniques for hydrogen analysis, ion beam techniques have become popular and satisfy most analysis needs. However, the NRRA (nuclear resonant reaction analysis) technique is particularly attractive and powerful because of its inherent capability of providing a nondestructive and simple analysis of the total quantity of hydrogen in a sample.

The three resonant reactions that have been used far more than others aro those induced by ^{15}N (Lanford et al., 1976), ^{19}F (Leich and Tombrello, 1973) and ^{7}Li (Adler et al. 1974). Each of these reactions has its own advantages. For most applications, the ^{15}N reaction has the advantage of having the best combination of analytic characteristics (depth resolution and sensitivity). The ^{19}F reaction has the advantage that it can be conducted using natural (as opposed to isotopically enriched) F in the accelerator ion source. The ^{7}Li reaction has the advantage of allowing profiling to much greater depths in a sample than either of the other reactions mentioned above.

The summary of the parametres used for hydrogen profiling for ^{19}F are listed, based on data from literature in table 1.

Table 1 Summary of nuclear parameters for hydrogen depth profiling

	Reaction: $^{1}H(^{19}F,\alpha\gamma)^{16}O$	
	Resonance: FL	*FH*
Resonance energy E_{cm}(MeV) E_{lab} (MeV)	 0.324 6.420	 0.829 16.44
γ-ray energy (MeV)	6.13, 6.98, 7.12	6.13, 6.98, 7.12
Branching ratio (%)	96.85, 0.55, 2.60	73.73, 20.67, 6.0
Resonance width Γ_{lab} (KeV)	44	86
Resonace cross section σ (mb)	88	440
Yield α Γx σ	3870	38000
(Relative yield)	1.30	12.5
Energy gap to next resonance (MeV)	2.70	1.2

The results from literature show that the method using the 16.44 MeV ^{19}F resonance has an excellent depth probe capability and moderate resolution as well as adequate sensitivity.

Beam properties for hydrogen analysis:

Numbers of days: 6 days (3 days for $^{19}F^{4+}$ and 3 days for $^{15}N^{3+}$)

Incident beam: $^{19}F^{4+}$, $^{15}N^{3+}$

Energy: 16-18 MeV for $^{19}F^{4+}$ and 13-15 MeV for $^{15}N^{3+}$

Beam Current: 30 nA for $^{19}F^{4+}$ and 5 nA for $^{15}N^{3+}$

Line: 5

Justification of the requestment

This results will be reported in the final stage of a National Partnership Grant. (PNCDI2 72-191, acronym NUCNANO) where IFIN-HH is the project coordinator which has a deadline this year, 10 December 2011.

The results will be also presented in MC meeting of COST action „Composites of Inorganic Nanotubes and Polymers" (COINAPO) (End date: May 2013) – MP0902. IFIN-HH is part of the romanian team involved in this project.

References

1). Nuclear Reactions for Hydrogen Analysis, W. A. Lanford, in the Handbook of Modern Ion Beam Analysis ed. by J. Tesmer and M. Nastasi, Materials Research Society (1995)193.
2). K. Umezawa et al. Quantitative hydrogen analysis by simultaneous detection of $^{1}H(^{19}F, \alpha\gamma)^{16}O$ at 6.46 MeV and ^{19}F-ERDA.
3). Combined growth of carbon nanotubes and carbon nanowalls by plasma-enhanced chemical vapor deposition Alexander Malesevic, Sorin Vizireanu, Raymond Kemps, Annick Vanhulsel,Chris Van Haesendonck, Gheorghe Dinescu, Carbon 45 (2007) 2932–2937
4). D. Endisch et al. ^{15}N analysis ofH in polymer films