
Experiment Title Hydrogen analysis in CNT, through RNR technique

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Short presentation of the scientific project

Nuclear resonant analysis for 1H determination in solid material like CNT, will be investigated using the following nuclear reaction 1H (19F, αγ)16O at the resonant energy in the range 6-18 MeV. The 19F reaction has the advantage that it can be conducted using natural (as opposed to isotopically enriched) F in the accelerator ion source. This experiment will be make on the 5 line of the Tandem accelerator.

History and production of CNT

The carbon nanotubes (CNT) are a new form of carbon made-up of graphene layers rolled-up into a cylindrical form (Figure 1). Discovered in 1991 by Sumio Iijima of the NEC Corporation, they immediately appeared as interesting materials because of their physico-chemical features. Their name is derived from their size, since the diameter of a nanotube is on the order of a few nanometers, while they can be up to several millimeters in length. The combination of their mechanical, thermal, chemical and electronic properties makes single-walled CNT (SWNT) and multi-walled CNT (MWNT) unique materials in nanoscience and nanotechnology

These cylindrical carbon molecules have novel properties that make them potentially useful in many applications in nanotechnology, electronics, optics and other fields of materials science, as well as potential uses in architectural fields. They exhibit extraordinary strength and unique electrical properties, and are efficient thermal conductors. Their final usage, however, may be limited by their potential toxicity and controlling their property changes in response to chemical treatment.

Fig. 1. By rolling a graphite sheet in different directions, two typical nanotubes can be obtained: zigzag (n,0), armchair (m,m) and chiral (n,m).

This CNT are produces by our partners, INFLPR, 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 home made 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 (C2H2) 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.

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 technique 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 15N (Lanford et al., 1976), 19F (Leich and Tombrello, 1973) and 7Li (Adler et al. 1974). Each of these reactions has its own advantages. For most applications, the 15N reaction has the advantage of having the best combination of analytic characteristics (depth resolution and sensitivity). The 19F reaction has the advantage that it can be conducted using natural (as opposed to isotopically enriched) F in the accelerator ion source. The 7Li 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 19F are listed, based on data from literature in table 1.

Table 1 Summary of nuclear parameters for hydrogen depth profiling

Reaction: 1H(19F,αγ)16O
Resonance: FL
FH
Resonance energy
Ecm(MeV)
Elab (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 Γlab (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 6.42 MeV 19F resonance has an excellent depth probe capability and moderate resolution as well as adequate sensitivity.

Beam properties for hydrogen analysis:
Numbers of days: 3 days
Incident beam: 19F4+
Energy: 6-18 MeV
Beam Current: 30nA
Line: 5

Justification of the requestment

This results will be reported in the third stage of a National Partnership Grant. (PNCDI2 72-191, acronym NUCNANO) where IFIN-HH is the project coordinator which has a deadline on 27 October this year.
The results will be also presented in MC meeting which will take in February 2011. This conference is hosted in a 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). Handbook of modern ion beam materials analysis, W.A. Lanford;
2). K. UMEZAWA, T. KUROI, J. YAMANE, F. SHOJI, K. OURA and T. HANAWA QUANTITATIVE HYDROGEN ANALYSIS BY SIMULTANEOUS DETECTION OF 1H(19F, αγ)16O AT 6.46 MeV AND 19F-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.

Beam time request(unit=8 hours) : 9 units
Desired Period : 15.09.2010- 15.10.2010

Desired beam properties

Type : 19F
Energy(MeV) : 6-18 MeV
Intensity(p/nA) : 30nA
Vacuum Requests : 10-5 torr

Special requirements for detectors, electronics,aquisition system
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Minimal information needed for the radiological risk evaluation:
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a)Source activity :
b)Use of open sources :

c)Estimate of the residual activity as a result of iradiation :
d)Means of storage/transportation for iradiated targets :