Experimental Report

Proposal: **6-02-512** **Council:** 4/2012

Title: Dynamics of water inside hydrophobic nanotube

This proposal is continuation of: 6-02-486

Researh Area: Physics

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Samples: C12

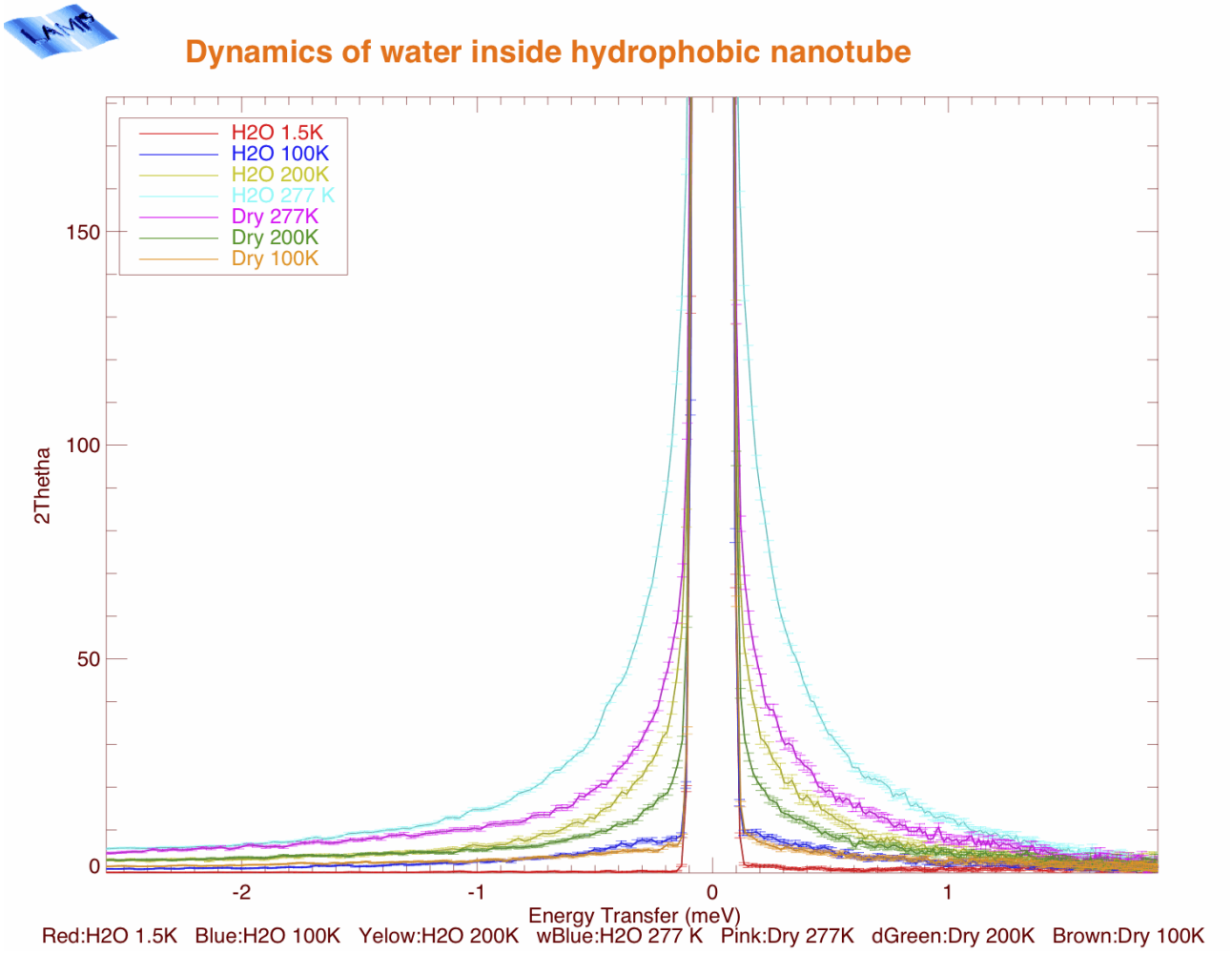
Instrument	Req. Days	All. Days	From	To
IN5	0	1	19/11/2012	20/11/2012

Abstract:

Liquids confined in nanotube environment possess anomalous static and dynamic properties which are of interest to a large scientific community. In particular, confined water is characterized by 1 and 2 dimensional structures (deep inside or facing the nanotube interface), faster diffusion than in bulk water, and possible collective modes associated with the specific HB networks. Despite many molecular dynamics simulations were focused on this topic, only a few experimental results can be found in the literature. With this proposal we want to investigate the dynamics of water confined in hydrophobic nanotubes of 1.5 nm diameter and 0.5-3 mm length in a temperature range from 2 to 280 K from ps to ns time scales. By comparing BS and TOF results from hydrated, partially hydrated and anhydrous nanotubes, we want to characterize the dynamics of the water inside the prototypical nanotubes. Such dynamics mirror the motion of water belonging to the clathrate-like structures, which shield the inner wall of the nanotube, and of the 1D water network that is present in the inner part of the nanotube.

Experimental report 6-02-512
"Dynamics of water inside hydrophobic nanotube"

The experiment was proposed to verify the presence, at very low temperature (10-100 K), of anomalous dynamics of water confined inside carbon nanotubes. The timescale we want to analyze ranges from ps to a few ns and, to this end, we ask for four days on IN16 and four days on IN5, but we obtain only one day on IN5. Some evidence of this anomalous dynamic was reported in the references 3, 4 and 5 (quoted on the proposal) as well on measurements we previously made on IN10. In this case the poor statistic did not allow to obtain quantitative evaluations, but some evidence of dynamics present at very low temperature (10-100 K) was observed.
On the base of the previous experiments we made with NMR, dielectric spectroscopy and on IN10, the measurements was suppose to be performed on i) powders of nanotubes hydrated in H_2O vapor for 20 days, ii) on dry nanotubes and, it was programmed finally, iii) on nanotubes hydrated for a few hours. In the case of the last sample the water can be adsorbed only on the surface of the nanotube grains and no water get into them, thus the relative spectra can be used to eliminate the contribution due to the external water. It has to be noted that the ratio between the amount of confined and surface water molecules can be roughly evaluated weighing the anhydrous, the partially hydrated and the fully hydrated samples. The internal/external water ratio results to be about 10%, thus the dynamic of the surface water is predominant compared to that of the confined one. Unfortunately the machine time was not enough to ran the last sample and we have had no data for the external water dynamics alone.
The results are reported in the following figure.



It is evident that, within the IN5 resolution, at very low temperature no dynamic is present, only above 100 K some dynamic can be seen. Since we have no data about partially hydrated nanotube, no conclusive statements can be made about the water dynamics inside the nanotubes.
For temperature above 100 K the analysis is on doing in connection with other measurements we made in August, since we have had other beam times in august on IN6 and IN16. The data collected in these experiments are under elaboration and concern the temperature dependence of: I) an anhydrous sample and ii) a sample hydrate only inside the nanotubes. Indeed in this experiment we followed a different procedure of sample preparation: first hydrated samples were made under water vapor at a temperature of 120 C and a pressure of 2 atm, with this procedure the preparation require about 1 day instead of 20. Second taking these hydrated powder at 40 C for about 4 ours the external water can be removed and the only water remains inside nanotube, allowing the measurements of their dynamics alone.
These data are analyzed together with those from the experiment on IN5 that concerns fully hydrated samples. In particular comparing, from IN5 and IN6 data, the Q dependence of the line width above 100 K it will be possible to disentangle the both the expernal and internal water dynamics.