MINUTES OF THE PTOLEMY PROJECT MEETING

CIEMAT Office E70.P1.13, 18 June 2018, 12:30h

Attendees:

Alejandro Moroño, María Isabel García Cortés, Pablo García Abia, Santiago Cabrera, Antonio Molinero, Julio Cárabe, Isabel Rucandio

Objective of the project (Pablo García):

How our universe is today and how it will evolve in the future depends entirely on the amount of energy and matter it has had in the different phases of its development. Currently, the universe contains 5% conventional matter (Standard Model particles, chemical elements), 27% dark matter (we don't know what it is) and 68% dark energy (we don't know its origin either). About 13700 million years ago, a time when what we now see of the universe (a balloon 93000 million light years in diameter) was about the size of a melon, the density of matter and energy was very high and local fluctuations in this density have, over time, given rise to macro structures such as clusters and superclusters of galaxies, cradles of star formation and ourselves, ultimately. In this primordial sea of particles, neutrinos played an important role. These were abundant in that universe where, besides them, there were essentially only electrons, positrons and photons. The presence of these primordial neutrinos has been inferred (indirectly) from cosmological measurements such as fluctuations in the cosmic microwave background (CMB). The direct detection of these neutrinos is crucial to ascertain that they actually played an important role in the structure of the universe moments after inflation (the so-called Big Bang) and would shed light on the so-called leptogenesis, the primary process in which the first leptons (including electrons, positrons and neutrinos) emerged.

What are these neutrinos like and where do they come from? At the time of the early days of the high-density universe of electrons, positrons and neutrinos, these particle species were in thermal equilibrium. Electrons and positrons collided, disintegrating through a Z-boson into neutrino-antineutrino pairs. These in turn collided, giving rise to electron-positron pairs by the same mechanism. As the universe expanded, the temperature dropped below the electron mass, at which point the equilibrium was broken and the neutrinos were decoupled (freed). At that time (about 1 second after the Big Bang) the temperature of the universe was 1.95 K, about 1 MeV. With the subsequent expansion of the universe for nearly 14 billion years, neutrinos have been losing energy, dropping to the current 10^-4 to 10^-6 eV.

With these low energies it is practically impossible to detect the already elusive neutrinos. That's why we need a very massive detector, which maximizes the probability of a primordial neutrino producing a tritium-induced beta decay, and an ultra-precise measurement system, capable of detecting electrons and measuring their energy with an accuracy of eV fractions, to be sensitive to the effects of the tiny mass of neutrinos and thus be able to discriminate events induced by primordial neutrinos from the natural beta disintegrations of tritium.

Tasks performed (Alejandro Moroño):

For the comparison of deuterium absorption in different materials, the following five samples have been collected from the Chemistry Division:

- Graphite rod (Grafbarra)
- Graphite powder (Grafpolvo)
- Graphite oxide Exp.3 (Dried suspension in bottle) (GO Exp3)
- Graphite oxide GO-8 January 2016 (Chunks derived from synthesis) (GO-8)
- Reduced graphite oxide (Powder) (rGO)

As decided at the meeting of 21/05, these samples have been placed in a special chamber at a temperature of 50 C and an absolute pressure of 1 bar and exposed to deuterium gas for 6 hours to check the incorporation of deuterium into the material under these conditions.

This experiment has been repeated with an equal batch of samples, but in this case both the gas and the samples have been subjected at the same time to the bombardment of 1.8 MeV electrons generated in the Ciemat Van de Graaff accelerator with a current density of 5 microA/cm^2.

After these experiments, the initial sample of graphite, the initial sample of graphite charged with unirradiated deuterium (Unirr) and the sample charged with deuterium and irradiated (Irr) were studied using the techniques of TSD (Thermal Stimulated Desorption), SIMS (Secondary Ion Mass Spectrometry). The rest of the samples could not be studied by these techniques because they are not presented in solid form.

Using TSD, after the samples were loaded with and without radiation, they were heated to 10 degrees/minute and the amount of deuterium released per second was measured as a function of temperature, only the mass 4 corresponding to the deuterium molecule D_2 was measured and the results were shown in a graph, showing that the irradiated sample (e-Irr) releases more deuterium than the non-irradiated sample (Unirr), and also in three phases with maximums at about 250º, 420º and 630º indicating different forms of retention. Effusion results are, in the first instance, compatible with limited damage to the samples.

Using SIMS, the sample surface is bombarded with high-energy ions, producing ejection (or sputtering) from the surface of neutral, charged species (atoms, groups of atoms, molecular fragments) that are detected by mass spectrometry. The graph obtained shows that the original sample and that of the first experiment (Unirr) are very similar. However, there is an important difference from the irradiated sample for the deuterium mass, which means that the irradiation of that sample favours the retention of deuterium.

If we compare the results obtained by SIMS and TSD, we observe that SIMS shows that there is much more deuterium in the radiation charged (a signal of the order of 10 higher), this is also observed by TSD although the relationship in this case is approximately double. This fact seems to suggest that some of the deuterium that is released during progressive heating to 1000 does not release in molecular form, i.e. as D_2. After repeating the SIMS analysis of the sample analyzed by TSD the result revealed that the amount of atomic deuterium is similar to that of the unirradiated sample, indicating that all retained deuterium has been released during heating by TSD. If the deuterium released in molecular form is only twice that of the unirradiated sample, and according to SIMS there was about 10 times more, it can be deduced that a large part of the deuterium released in this treatment was bound to other atoms, which could be with a carbon-deuterium bond (C-D), which would be the most favorable for the objectives of the project.

Other topics:

These early achievements are considered significant and the desirability of further experimental work is raised for:

This characterization seeks to identify and quantify the adsorbed deuterium (physiosorption) and the one bound to carbon (chemisorption). That will be done by getting to know each other, if possible:

- the amount of deuterium incorporated in the basic material,
- the damage caused to it during the deuterium loading process and/or if the material is recrystallized, and
- Check whether and to what extent a reduction in graphite oxide occurs.

It would be convenient to characterize the three series of samples: original, unirradiated but exposed to deuterium (Unirr) and those loaded with deuterium and irradiated (Irr) by various techniques. In the first stage, we will try to study using the most available techniques at CIEMAT, which are:

- Fourier Transform Infrared Absorption (FTIR), provides information on the vibration spectrum and existing functional groups. They can help identify and quantify deuterium-carbon bonds.
- Raman spectrometry, studies low frequency modes (vibrational, rotational, etc). They can provide information on the structure of graphite or graphite and its possible modification as a result of treatments, mainly electron irradiation. This is a complementary technique to FTIR.
- X-ray diffraction (XRD), detects the crystalline species present.
- Thermogravimetry (TG), studies the variation of the weight of the sample as a function of temperature.
- Spectroscopy of X-ray photoelectrons (XPS), which studies the surface of a material to determine its elemental composition (qualitative and quantitative), empirical formula, and the oxidation states of the elements present in the material.
- The B.E.T. method, developed by Brunauer, Emmett and Teller, makes it possible to determine the surface of a solid based on the adsorption of an inert gas, usually N2, at low temperature.

In a second phase we could also study the samples by SEM (scanning electron microscopy), TEM (transmission electron microscopy, STM (tunneling electron microscopy) or AFM (atomic force microscopy), some of which are available at

CIEMAT and others off-center, which can help to visualize the possible fixation of deuterium atoms in the graphite or graphite network.

If it is possible to obtain samples of 2D graphene on copper, they will be used for Raman and STM (FTIR measurements cannot be made with metal substrates).

To establish the sequence of analysis, the samples will first be pressed in powder form (a small press must be found), leaving a small quantity for XPS, Raman and TG, and will be analyzed by non-destructive techniques (FTIR, XRD, SIMS) and then destroyed if necessary to perform the rest of the analyses.

In order to share all the information that came out of these studies, it was proposed that a folder be opened on the common graphic disk (\\cendat2\gtg\Ptolemy), that it would contain all the information on the project and the data and results of the different studies that were carried out and, of course, that it would give access to the people who participated in this project. (A. Molinero).

On the other hand, it was reported that there is a tritium analysis laboratory at CIEMAT, in building 3, and an attempt will be made to contact them to see if they can contribute their knowledge to this project (I. Rucandio).

It was also reported that a new call for research projects funded by the Autonomous Community of Madrid is about to be launched. Consideration should be given to applying for a project on this subject to secure funding.