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Porosimetry with Positrons

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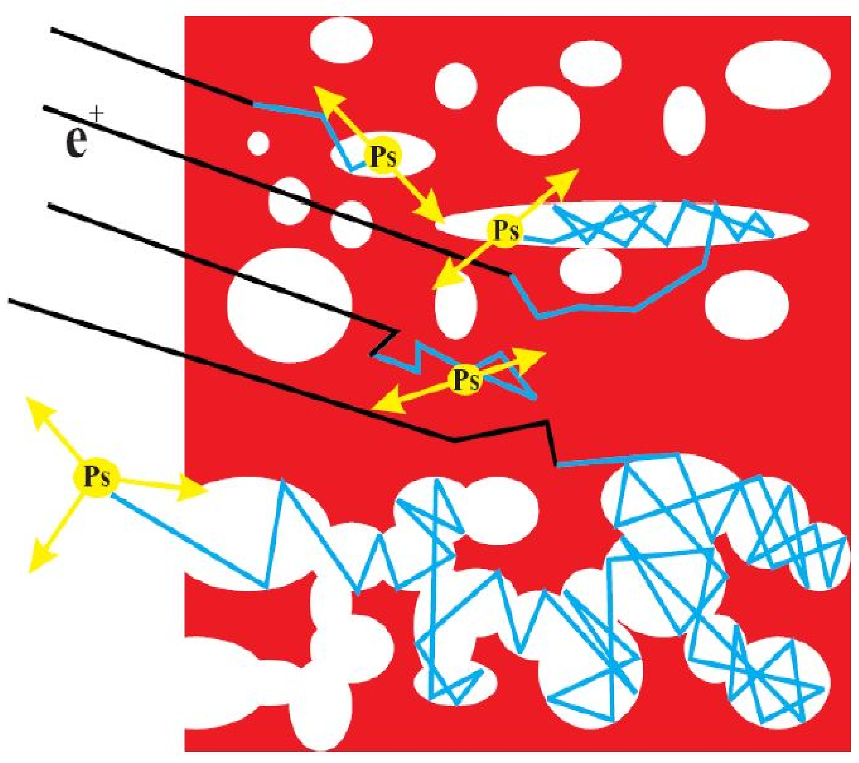
This poster shows how porosity can be detected by using **P**ositron **A**nnihilation **L**ifetime **S**pectroscopy (PALS).

Physics



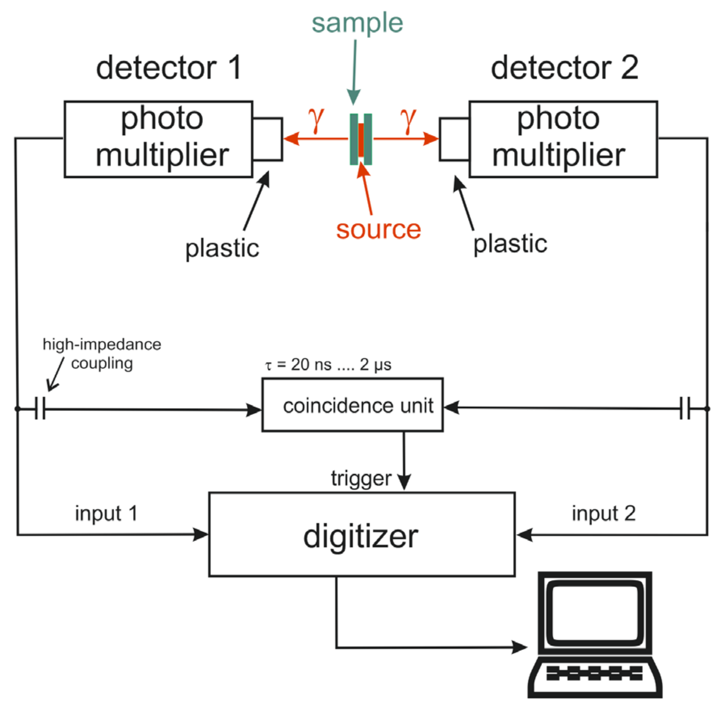
Positrons are emitted by a Na^{22} source and interact with the sample. The lifetime (T) of the positrons depends heavily on the electron density. They can be trapped in a pore or vacancy and live longer than in bulk material.

So the time until the positron annihilates gives us information about the porosity.

The fact that a 1.27 MeV gamma ray is emitted simultaneously with the positron and two 511 keV gamma rays emerge by pick-off annihilation make it possible to measure the lifetime of the positron. The bigger the pores are, the longer is the lifetime of the positrons.

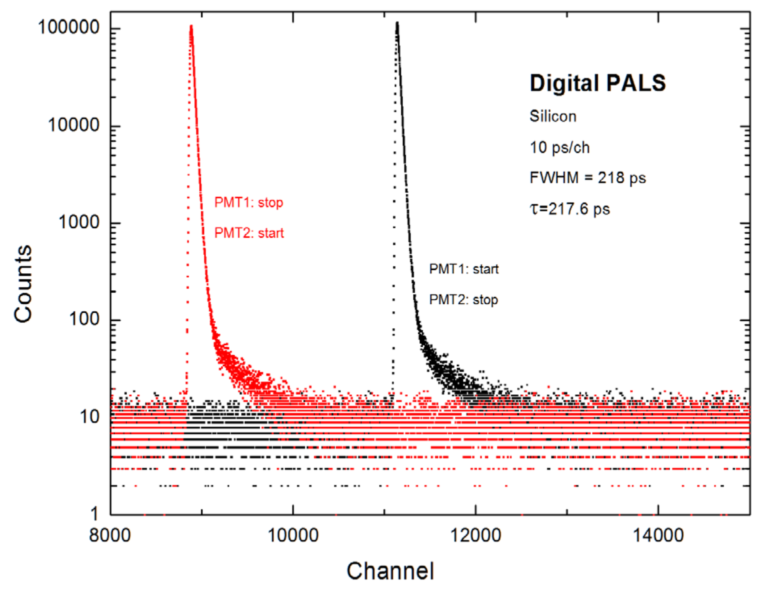
The picture shows the different ways how a positron can annihilate. Either a pick-off-annihilation takes place where two gamma rays are emitted or the annihilation with three gamma rays in vacuum. Only the pick-off-annihilation provides information of the material. Pick-off-annihilation means that the positron interacts with an electron in the sample and annihilates earlier than in vacuum.

Technology

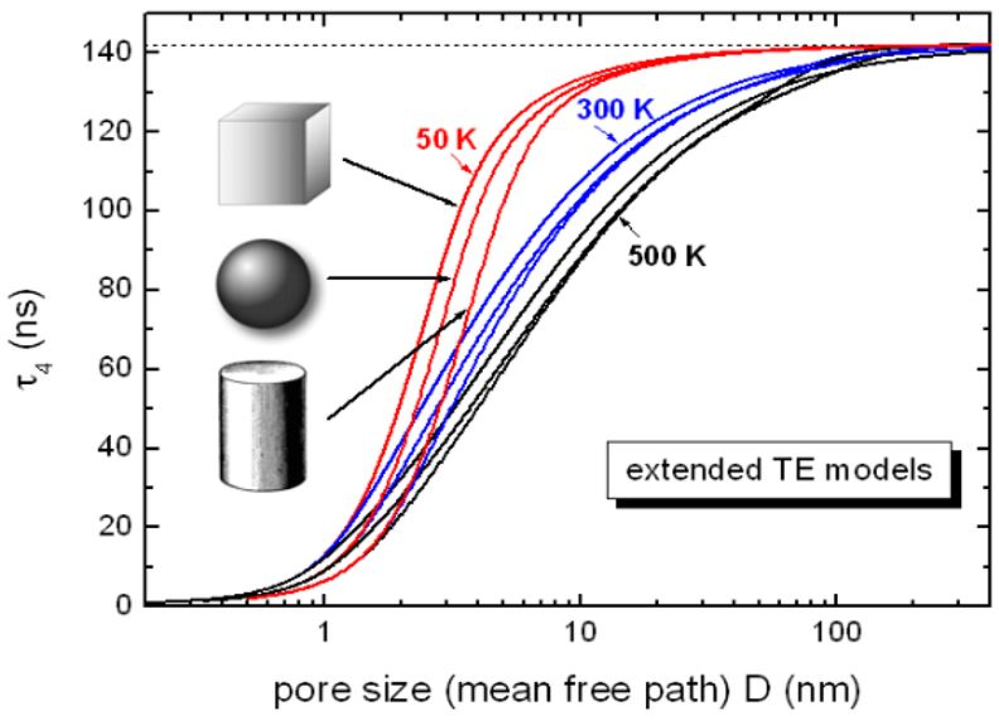


A scheme of the digital PALS is pictured above. To detect the gamma rays photomultiplier (PM) with plastic scintillators are used. Both function as start or stop pulse. The coincidence unit proves whether the signals fit in a set time range and actuates the trigger. The digitizer sends the data to a PC which arranges the pulse to start and stop. To get a full spectra we need to collect at least 10^6 events.

An example for the resulting spectrum is shown below. In principle there are two spectra in one because both PMs can work as start and stop detector. In these spectra the information about the lifetime of the positrons are included. A special software e.g. MELT or LT10 calculates the lifetimes and assigns the corresponding intensities.



Theory

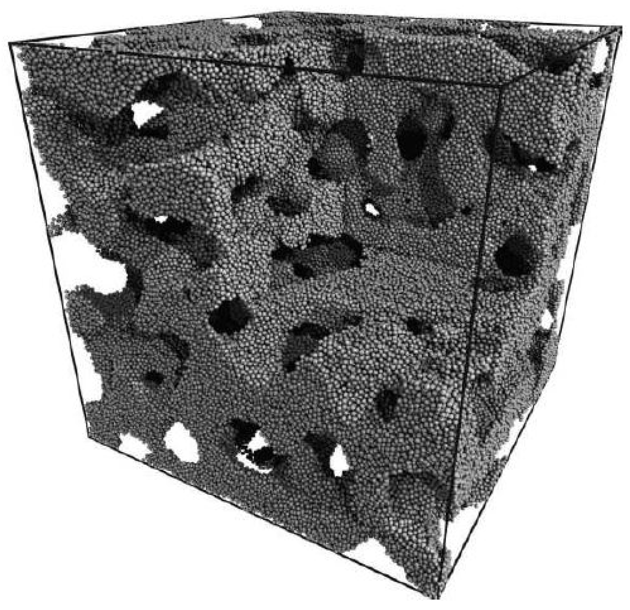


To connect the lifetime with the pore size it is necessary to use a mathematical model for the pore properties. The lifetime depends on temperature and geometry of the pore. A possible relation is described by the extended Tao-Eldrup-Model. Obviously the PALS is very sensitive of differences in pore sizes between 1nm and 30nm.

Possibilities and Limits

With PALS it is possible to discover vacancies e.g. in semiconductors or determine the pore size distribution of porous materials as CPG *(picture on the right),* for example. Furthermore open volumes in polymers can be detected. The advantage to other techniques is that even closed pore systems are visible to positrons and the measurement process is non-destructive.

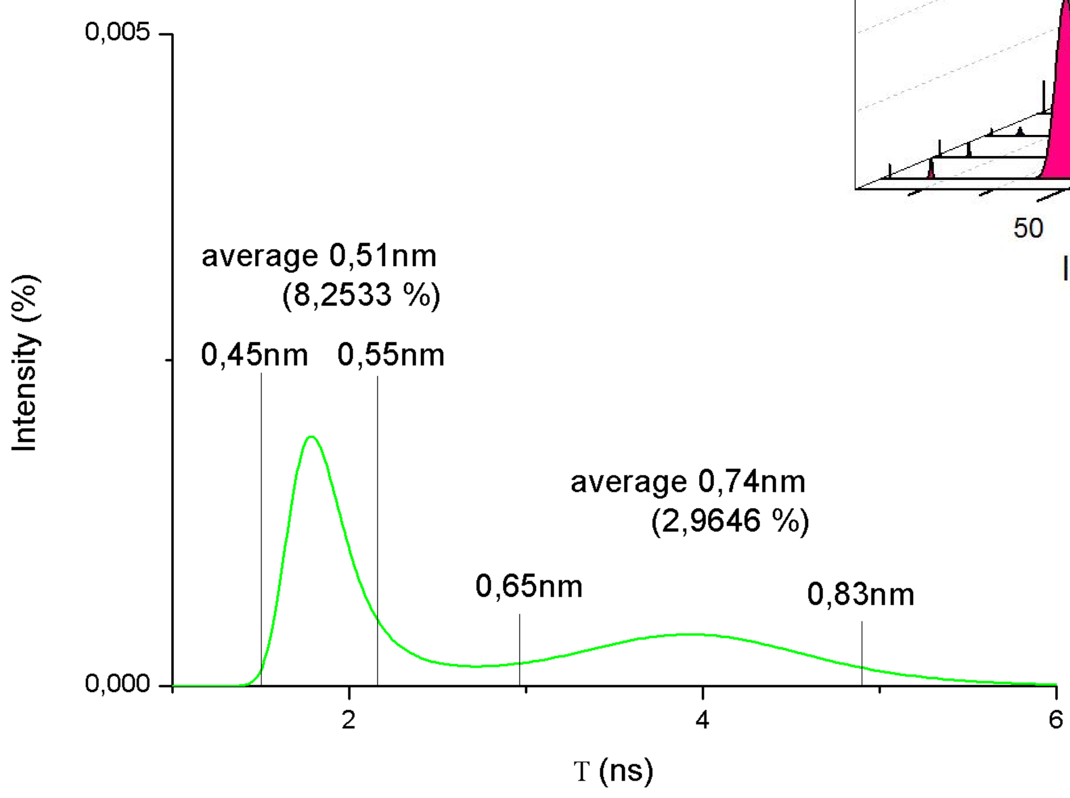
The limit is the lifetime of the positron in vacuum. If the open volume is too big, the PALS can't deliver any information abut it. The horizontal dashed line in the picture on the left side stands for the physical upper limit (vacuum lifetime). Thus, the practical upper limit is around 60nm pore diameter.

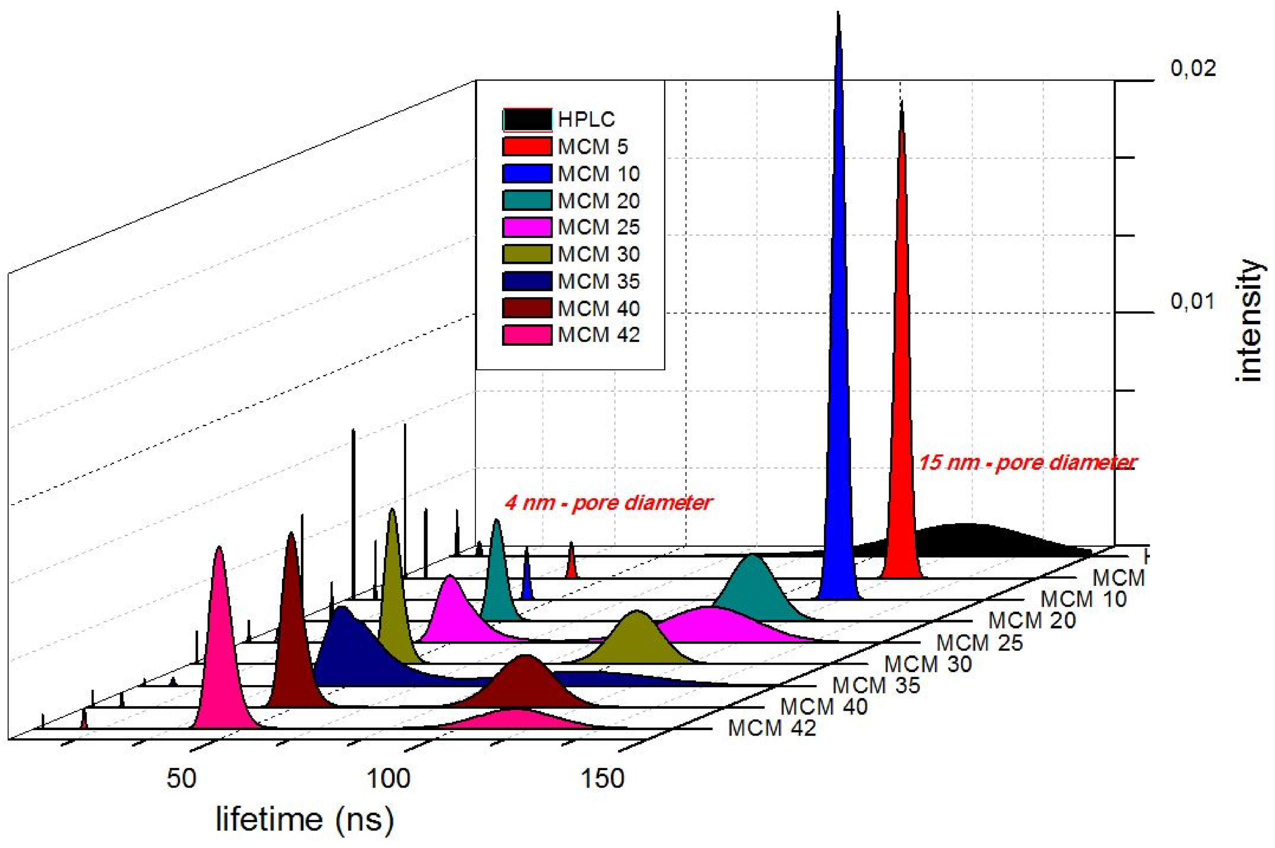


http://www.chemistry.wustl.edu/ gelb/cpgvis.html.

Application and Examples

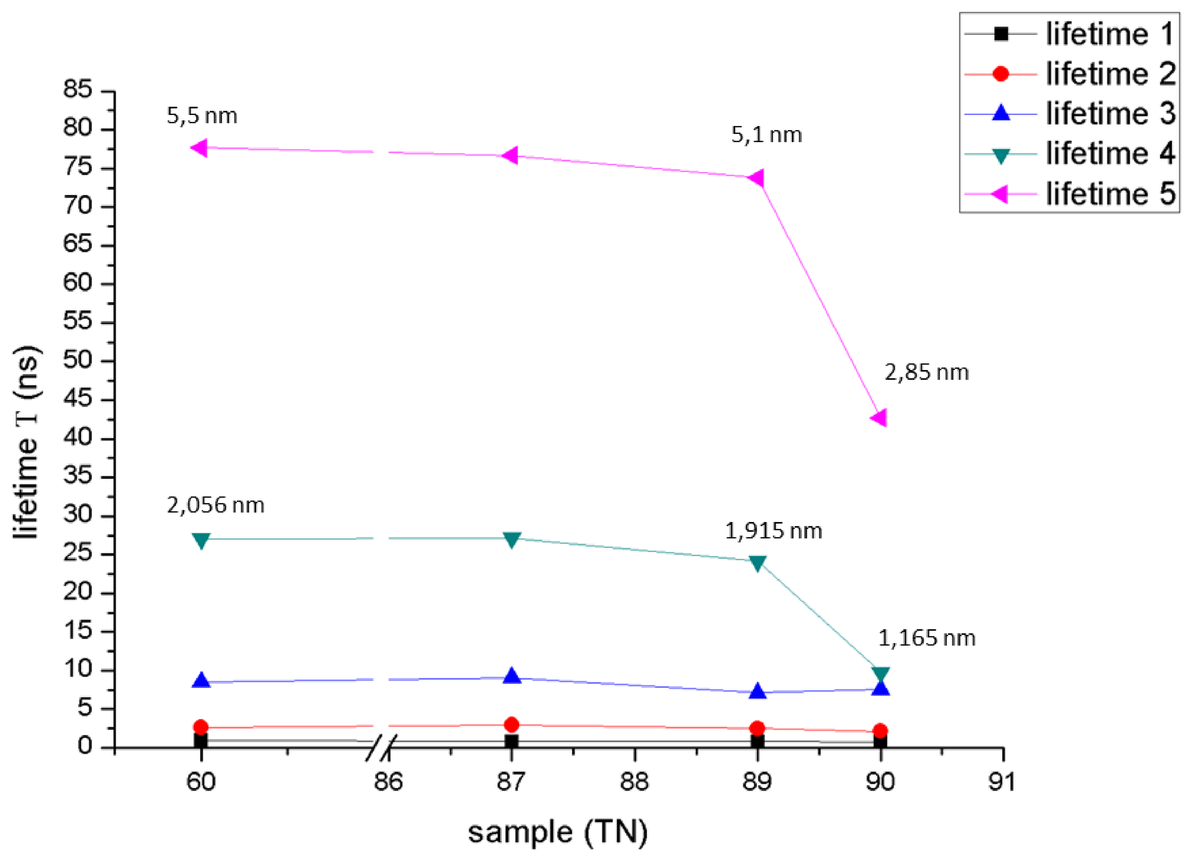
To show pore size distributions we are using the analysis software MELT. In the first example on the left side the distribution of two pore sizes from nano capsules is shown. While the smaller pores have a straight distribution within 0.1nm and a clear peak, the pore distribution for the larger pores is very flat and wide.





The second example shows an transformation of a porous material. 4nm pores were build into the wall of 15nm pores. The intensity (equal to the number of pores at a discrete lifetime) of the 4nm pores is increasing with the amount of solution.

It is also possible to measure loaded pore systems with drugs, salts, liquids or other materials. In this last example 5,5nm and 2nm pores were filled with salt. Loaded pores have a lower positron lifetime because of the smaller open volume. In this case a 5,5nm pore is loaded by 50% with salt in average. The same for the smaller 2nm pores. The intensity of the lifetime is an indicator for the number of filled pores.



Poster is available at http://positron.physik.uni-halle.de
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