Low energy positron beam system for the investigation of 2D and porous materials

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Low energy positron beam system for the investigation of 2D and porous materials

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Abstract. An advanced variable energy positron beam (~2 eV to 20 keV) has been designed, tested and utilized for coincidence Doppler broadening (CDB) measurements at the University of Texas at Arlington (UTA). A high efficiency solidified rare gas (Neon) moderator was used for the generation of a slow positron beam. The gamma rays produced as a result of the annihilation of positrons with the sample electrons are measured using a high purity Germanium (HPGe) detector in coincidence with a NaI(Tl) detector. Modifications to the system, currently underway, permits simultaneous measurements utilizing Positron annihilation induced Auger Electron Spectroscopy (PAES) and CDB. The tendency of positrons to become trapped in an image potential well at the surface will allow the new system to be used in measurements of the chemical structure of surfaces, internal or external and interfaces. The system will utilize a time of flight (TOF) technique for electron energy measurements. A 3m flight path from the sample to a micro-channel plate (MCP) in the new system will give it superior energy resolution at higher electron energies as compared to previous TOF systems utilizing shorter flight paths.

1. Introduction

Substantial interest has developed in the field of two-dimensional (2D) materials since the isolation of atomic layer graphene [1]. The rich electronic properties observed in 2D materials, such as the superconducting transitions [2] and semiconducting properties observed in 2D transition metal dichalcogenides (TMDs), provide materials and electronics engineers the prospect of designing and controlling 2D material properties and devices based on them [3,4]. Not only is there interest in 2D materials, but also in the properties specific to the surfaces of materials [5]. In this regard, it is desirable to study these systems with techniques that can probe material properties with atomic layer precision without probe modification of the surface properties and with minimal sample preparation.

Positron beams have been used to study depth dependent properties of overlayers with positron energies typically larger than 100 eV [6]. By reducing the positron beam energy to a few eV it has been shown that positrons can be efficiently deposited into a surface localized state in a process known as Auger mediated positron sticking (AMPS) [7]. Due to this localization, the signals resulting from the annihilation of surface trapped positrons are highly surface specific. Consequently, low energy

positrons are well suited to the investigation of two dimensional materials.

Nanoporous metals are another class of materials which are developed for applications in catalysis, energy storage, bio sensing, etc [8]. An understanding of the chemical and structural evolution of the internal surfaces of these high aspect ratio materials are important in tuning their properties to increase their efficiency in photo catalysis [9] or to understand their stability in service conditions [10]. Conventional methods used in characterizing external surfaces are unsuitable for internal surfaces due to the inability of the electron or optical signals to escape from the internal surfaces. Investigations of internal surfaces have relied on destructive or indirect measurements such as EXAFS [11].

Positrons implanted at a few keV energies into porous materials will predominantly reach the internal surfaces of pores and voids after thermalization and will get trapped in a surface state on the internal surfaces resulting in annihilation with electrons at the top atomic layer. The ability of the annihilation gammas to reach the detector from buried interfaces or from the internal surfaces of pores without the loss of the information that can be derived from the gamma spectra permits annihilation gamma spectroscopies to be used in the characterization of internal surfaces and interfaces [12].

In this paper we describe a new positron beam system that has been developed at the University of Texas at Arlington with the aim of studying 2D materials and internal surfaces of porous metals. The system will allow simultaneous measurement of the Doppler broadening (DB) of the annihilation gamma and Positron annihilation induced Auger electron spectra (PAES). The system is capable of depositing positrons of kinetic energies as low as 2 eV to as high as 20 keV on 2D or porous samples. This will enable us to investigate chemical information (PAES and DB) and open volume defect (DB) distribution on 2D materials when low energy (a few eV) positrons are used. With positrons at a few kilo electron volt energies, structural defects and chemical information of internal surfaces of porous materials may be obtained. DB spectra and PAES spectra collected in coincidence will enable us to correlate the Doppler broadened gamma-ray line shape to positron annihilations with electrons belonging to specific core levels of an element as demonstrated by Kim *et al* and Eshed *et al* [13, 14]. This correlation will be utilized to quantify the Doppler broadened gamma line shape so as to deduce chemical information from the inner surfaces of porous materials through the analysis of the gamma spectrum.

2. Experimental Apparatus

The new positron beam system shown in Figure 1 consists of three main components: 1) A rare gas moderator (RGM), 2) A 3m TOF region, and 3) Sample preparation and analysis chamber.

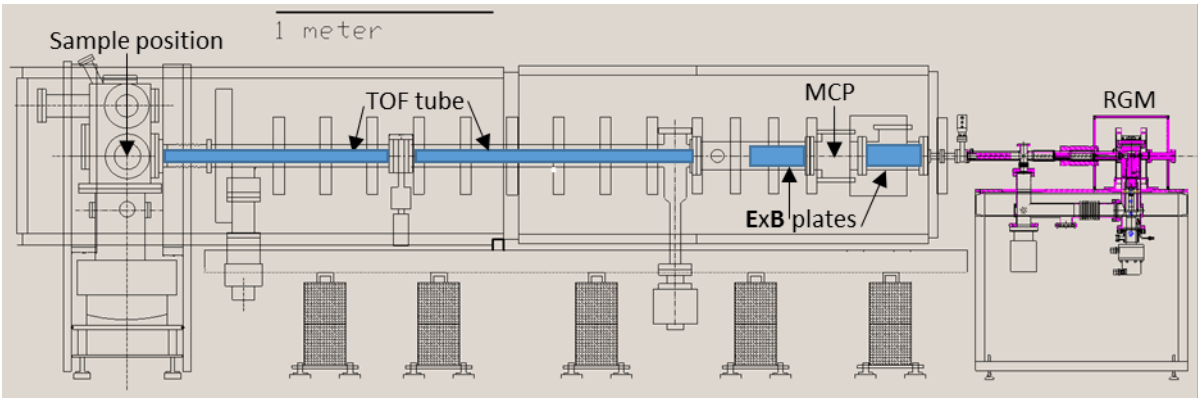

Figure 1. The rare gas moderator (RGM) on the far right is used to produce a slow positron beam. The positrons will be bent around a Microchannel Plate (MCP) electron detector using **E**×**B** deflection plates. A 3 m electric field-free region is used for TOF measurements. The sample chamber at the end houses the sample manipulator and is the site of gamma ray detection.

2.1. Rare Gas Moderator

A solidified rare gas moderator (RGM), produced by condensing Neon gas onto a cold head kept at 6.8 K, is used to generate slow positrons from a 0.5 mCi ^{22}Na radioactive source. A moderator efficiency of ~ 0.2 % has been achieved initially. Further optimization is required in the solid neon growth parameters and in the extraction optics of the slow positron beam to achieve the 1% efficiency reported for solid neon moderator [15]. A typical moderator will degrade to ~ 75% of its initial count rate after 6 days. Control of the positron beam kinetic energy is accomplished by applying a positive bias to the moderator. Once the slow positrons are emitted from the Neon moderator, they are accelerated by the electric field created by the positive bias on the moderator and guided by an axial magnetic field. The positrons are deflected by a magnetic field through an aperture in a tungsten barrier offset from the beam axis. The tungsten barrier serves to filter out fast positrons which are not deflected enough to make it through the offset hole. The positrons which make it through the filter are introduced into the TOF region using a magnetic field of 0.03 T. A beam diameter of ~ 6 mm at the exit of the RGM is decided by the aperture at the exit.

2.2. Time-of-Flight (TOF) Spectrometer

The spectrometer consists of a TOF tube which can be independently biased with respect to the grounded beam tube, a set of **E**×**B** plates, and a MCP electron detector. Positrons entering the TOF region are magnetically guided to the sample using a series of coils which produce an axial magnetic field of ~ 0.006 T. A set of rectangular correction coils are used to counteract the Earth's magnetic field or any other stray magnetic fields along the beam line and to help align the beam on the center of the sample. The **E**×**B** plates will be used to bend the positron beam around the MCP and to bend electrons emitted from the sample into the MCP. The 3 m long flight path of the new spectrometer is expected to lead to a 3-fold improvement in resolution compared to previous TOF systems [16]. Equation 1 shows that the flight time, t, of an electron traveling from the sample to the MCP is proportional to the travel distance, l. What is measured as a flight time is converted to channel number using a time-to-amplitude converter (TAC) and a multichannel analyzer and thus a difference in flight time will correspond to a proportional difference in channel numbers. As the flight path is increased to 3 m from 1 m, the difference in flight times of two electrons of nearby energies increases by three times thereby increasing the separation between them in the TOF spectra to three times their previous separation. This means that the energy per channel is reduced to one third of its previous value. High energy Auger peaks which were irresolvable because of being too close (< 2 ns) in time of arrival will now be resolved as they show up as separate peaks.

$$\# \propto t = \sqrt{\frac{m}{2E}}\, l \qquad (1)$$

2.3. Sample Preparation and Analysis Chamber

The sample chamber, capable of achieving a vacuum of 1.33x10^{-9} Pa, is equipped with a sputtering system to clean the sample, a LEED system for surface structure determination, and an electron Auger Electron Spectrometer (EAES) for additional characterization of surfaces. A permanent magnet producing a magnetic field of 0.07 T at the sample position is used to parallelize the electrons leaving the sample. The magnet also reduces the positron beam to ~4 mm on the sample allowing analysis of small samples. The sample is mounted on an x-y-z stage manipulator which helps in moving the sample from the beam axis to the sputtering position and to the analysis position of LEED and EAS. The sample is electrically isolated from the manipulator and is biased through the copper rod of a HV feed through. The system is equipped with a high resolution HPGe detector and a fast NaI(Tl) detector for coincidence doppler broadening (CDB) measurements.

3. System Testing

Having two oppositely placed gamma detectors in coincidence mode (Figure 2) reduces the background by an order of magnitude as shown in figure 3 which is comparable to the improvement obtained with similar coincidence systems [17]. In coincidence mode the output of the NaI(Tl) detector is used to gate the amplified signals from the HPGe detector at the multichannel analyzer (MCA). The histogram of the gamma counts generated by the MCA is converted to energy spectra by calibrating the detector system using four known gamma peaks of ^{133}Ba. The energy resolution of the HPGe detector was found to be 0.98 keV at the 356 keV peak of ^{133}Ba.

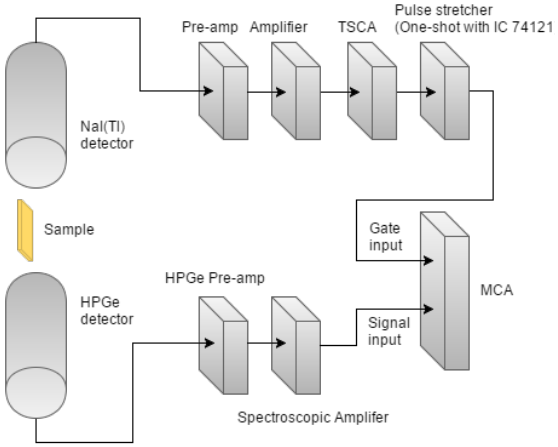

Figure 2. The NaI signal is amplified and the window corresponding to 511 keV gamma rays is selected using a timing single channel analyzer (TSCA). This signal is used as a gate for the HPGe detector.

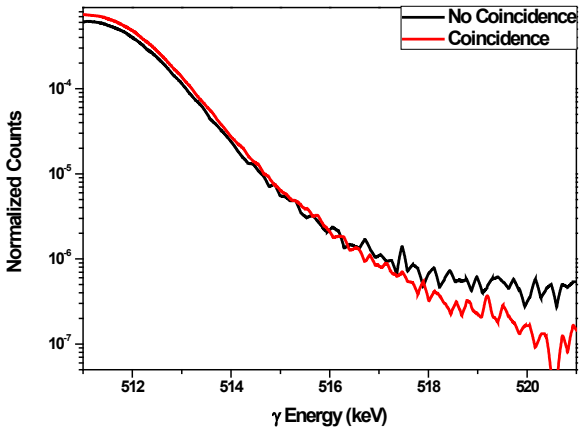

Figure 3. Collecting gamma ray spectra using coincidence mode reduces the background counts by an order of magnitude. This allows us to determine the high momentum region of the 511 keV line shape more accurately. Both spectra were normalized to their integrated intensities from 501-521 keV. Counting rates during this experiment were ~ 10 counts per second.

The system is capable of changing the kinetic energy of the positrons at the sample from 2 eV to 20 keV by appropriately biasing the sample with respect to the moderator. Figure 4 shows the positron kinetic energy measured at the entrance of the TOF tube by using it as a retarding field analyzer. Here the moderator was biased to 1 V resulting in a positron energy distribution with a FWHM of ~1.5 eV and peaking at 2.75 eV. Tests confirmed that the annihilation gammas from positrons reflected from a positively biased sample did not effect the results as shown in Figure 5. The reflected positrons retrace their path through the system and annihilate near the RGM as evidenced by the icrease in the gamma-ray counts near the RGM exit aperture with increase in positive bias on the sample.

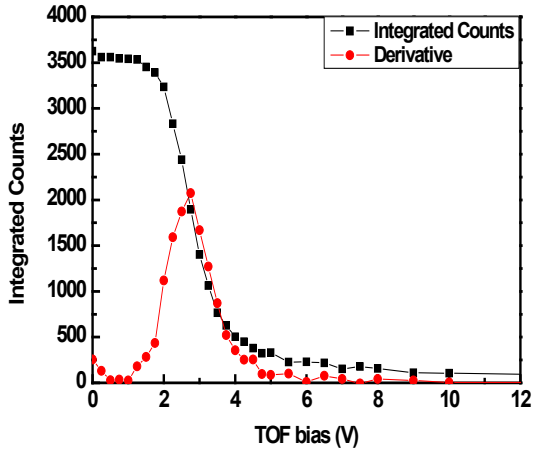

Figure 4. Positron kinetic energy distribution with a moderator bias of 1 V. Here we have plotted the integrated counts under the 511 keV peak as detected by the HPGe detector at the sample as a function of positive bias on the TOF tube. The derivative of the integrated counts give us the distribution of the positron energies, also shown in the figure. The energy is peaked at ~2.75 eV with a FWHM of ~1.5 eV.

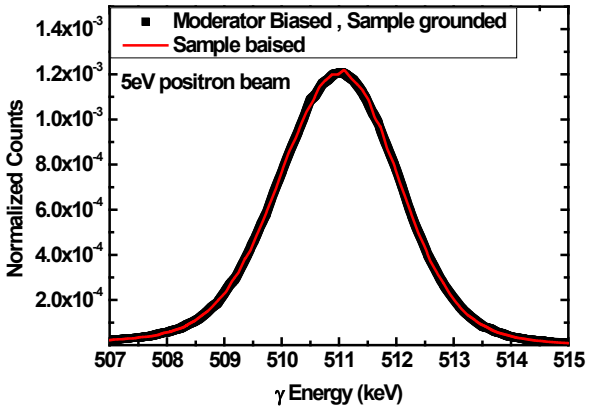

Figure 5. 511 keV gamma peak produced by biasing the system in two different ways to produce a 5 eV positron energy at the sample. The first method biases the moderator at +4 V with the sample grounded. The beam energy was determined to be 5eV at the TOF tube. In the second method, the moderator is biased to +32 V to get a 33 eV positrons at the TOF tube. The sample is then positively biased to 28 V to decrease the incident positron energy to 5 eV. Both methods are shown to produce equivalent gamma spectra.

4. Conclusions and Future Work

A positron beam system capable of simultaneously carrying out CDB and PAES measurements is under construction at UT, Arlington. The CDB spectroscopy system has been tested and utilized to investigate multilayer (6-8 layers) graphene grown on polycrystalline Cu [18]. Modifications to the system are currently underway to install a MCP electron detector and timing electronics. Electrons emitted from the sample travel through a 3 m flight path before being detected. The 3 m electric field-free region will give the new system superior energy resolution as compared to previous systems with shorter flight paths and allow for the investigation of internal and external surfaces. Additional improvements over the previous system include the addition of a HPGe detector for CDB experiments, as well as plans to digitize the data collection processes. Digitization will allow for time-correlated measurements of annihilation gammas and electrons collected at the channel plate.

Acknowledgments

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