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Cryogenic PEEM at the Advanced Light Source

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ABSTRACT

X-ray PEEM at liquid helium temperatures at a 3rd generation synchrotron is discussed. Detailed instrument design and performance is presented along with examples of the scientific opportunities afforded through routine low temperature performance with negligible tradeoffs of imaging performance or general ease of use.

1. Introduction

X-ray Photo Emission Electron Microscopy (X-PEEM) with soft X-ray synchrotron radiation has been utilized for the last few decades in many disciplines, the largest category of which can broadly be described as materials science of thin films, layered materials, and surfaces that exhibit inhomogeneity on nm to micron length scales. This last distinction is paramount to the choice of a technique capable of distinguishing local spectroscopic information that varies due to constituent material inhomogeneities, intrinsic domains, and/or patterned samples and would be averaged together if sampled with a non-spatially resolving technique. Many of these material systems exhibit couplings between materials and layers as well as complex phase diagrams exhibiting hysteretic effects when traversing phase changes. Sample temperature is thus of great interest to the community of X-PEEM users as a control parameter in experiments to study the rich physics of these phase changes. Access to cryogenic temperatures in particular has been a long time desire and has proved to be in great demand at the PEEM3 instrument at the Advanced Light Source (ALS) where for the last few years the user community has been able to perform routine measurements at temperatures as low as 20 K with minimal disruption to other instrument capabilities and performance.

PEEM, as a microscope, is challenged by vibrations. As a spectromicroscope requiring multiple time scale data acquisition from seconds to hours, it is also particularly challenged by drift on the same disparate time scales. These considerations are the primary difficulties facing cryogenic PEEM realization but are hurdles that have been solved by many other microscopy techniques, for example [1–3]. An additional complexity in some PEEMs, including the instrument described here, is that the microscope is designed with the sample at high voltage meaning that one must cool the sample while maintaining up to a 20 kV electrical isolation between the sample and the cryostat. Described in detail below is the successful design strategy employed to mitigate these challenges through cooling the smallest possible mass, indeed not cooling the sample manipulator at all, and employing thermal and vibrational isolation of this small mass from the rest of the instrument and the cryostat. In this way drift is minimized, cool down times are measured in minutes, sample cycle times are greatly reduced from cryogenic to non-cryogenic samples, and in general a cryogenic sample environment is introduced with negligible interactions and tradeoffs with the rest of the complex electro-mechanical instrument.

2. Mechanical design

The PEEM3 microscope located at beamline 11.0.1 of the ALS at Lawrence Berkeley National Laboratory is the current embodiment of an iterative, in-house engineered and fabricated PEEM program running concurrently for the last 15 years with a vibrant scientific program. The manipulator, which aligns the region of interest of the sample under test in front of the objective lens of the imaging column, is a home built 5 axis device capable of ±5 mm of travel in three orthogonal linear directions and ±2 degrees of tilt about the plane of the sample to optimally align the surface normal to the optical axis of the microscope. The manipulator is capable of sub 100 nm steps for centering of the region of interest and vibrational stability better than the currently available resolution limit of the electron optics (~30 nm). The mechanism is motorized and encoded and is integrated into a fully automated transfer system that moves sample pucks between three parking docks and the microscope through a computer interface. One of the strengths of

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the instrument is the fairly large sample pucks that provide for almost a cubic inch of space above and beyond the baseplate needed for interfacing with the microscope manipulator. This space is used for a variety of exotic and yet to be defined custom sample holders as well as for standard heatable (20–400 °C) holders. Holders capable of holding custom chips with lithographically designed circuits and HF connections, integrated electromagnets with in plane and out of plane configurations, and holders with on-board external laser driven amplifier circuits for time resolved measurements have been successfully used, to name a few. Since mid 2008, and routinely with an improved system installed in 2010, custom holders have been provided to the user community capable of both liquid nitrogen and helium cooling of samples with temperature regulation as well as compatibility with our electromagnet holder for the application of magnetic fields while cold.

Fig. 1 shows a computer aided design (CAD) model of the PEEM3 manipulator and a schematic showing the main components of the design. The cryostat is attached to the external vacuum tank of the instrument and is a potential source of vibrations to the sample manipulator even with no cryogen flowing, as the microscope is designed with the main optical table effectively isolated from its surrounding vacuum chamber. The cold head of the cryostat is attached to the moving manipulator sample dock first through a flexible copper braid for compliance of the sample motion and to mitigate vibration transmission. A sapphire rod at the end of the braid allows for reasonable thermal conductivity while maintaining electrical isolation of the 20 kV sample dock from the grounded cryostat. The top of the sapphire is then attached to a stack of thin gold coated copper ribbons that are part of a clamping mechanism which makes the thermal connection to the special cold sample holders.

As alluded to in the introduction, the cryogenic sample holders are designed with a small (<50 g) copper mass that is mechanically attached, yet through the use of plastic standoffs thermally isolated from, the baseplate of the sample holder necessary for compatibility with the system. This copper mass contains a cavity for the sample under test, a Si diode for temperature measurement, an integrated heater for intermediate temperature control, and an attached bulk piece of silicon (referred to as a nose) which extends from the copper to interface with the cold clamping mechanism (see Fig. 2). During introduction of the sample into the manipulator, the normal insertion of the sample baseplate places the silicon nose of the cold holder between the copper ribbons of the clamp. The normal locking mechanism further inserts the silicon nose, inducing a clamping force as the leaf spring backed ribbon stack is displaced. The cryostat now has good thermal conductivity to the copper mass of the cryogenic sample holder, but remains electrically isolated and vibrationally weakly coupled. Furthermore, standard microscope holders without the protruding nose do not make contact to the cold clamping mechanism and are thus largely isolated from potential vibrations through the cooling mechanism from the outside environment.

Although the cooled copper part of the sample holder has poor thermal conductivity to the baseplate, it is still finite. The sample baseplate, and thence the entire 5-axis manipulator, will slowly cool. This slow cooling can induce extremely long time scale drift and settle times. The large manipulator has struts of approximately 20 cm length. If these were allowed to vary in temperature by even a few tens of degrees, motions of 100s of microns would be expected at the sample, and indeed were witnessed in an earlier version of the set up. To mitigate this, there is another flexible copper braid employed to help thermally anchor the movable end of the sample manipulator to room temperature (see Fig. 1). As long as this thermal path's conductance is significantly larger than the standoffs on the sample holder and the tortuous path through the manipulator struts, the stage and strut mechanisms of the microscope manipulator will remain at a stable temperature and cooling of the sample baseplate and dock are minimized. Drift is dominated by the thermal expansion and contraction of the materials of the sample holder and dock only and is typically much less than a field of view (~30 μm) between room temperature and cryogenic temperatures.

3. Performance

With the relatively small mass being cooled, cool down times are measured in minutes. Typical times are less than 30 min to reach within 3 degrees Kelvin of the lowest temperature possible. 25 K is routinely maintained at flow rates of 1.5 l of liquid helium per hour, with slightly lower temperatures reached at the cost of more cryogen. When liquid Nitrogen is used as a cryogen, temperatures of approximately 100 K can be reached with cool down times extending to approximately 1 h. Sample holders can be transferred into and out of the manipulator regardless of the temperature of the sample holder or cooling mechanism. This enables swapping of one cold sample for another, or to a room temperature sample, without the need to wait for the entire system to come back to room temperature, greatly increasing the throughput and efficient use of the system.

The effects on imaging performance due to the interface to the cooling mechanism itself as well as cryogen flow induced perturbations are quantified in Fig. 3. Our standard test sample is used to measure the loss of contrast of the instrument as a function of the spatial size of features. The sample consists of a lithographically produced pattern of converging Ni lines arranged in a circular wheel referred to as a "star" pattern. The PEEM image in the top of the figure, acquired by imaging at an X-ray energy corresponding to the Ni L3 absorption resonance to maximize contrast, shows the central portion of the pattern with the smallest feature sizes. Visible in the left part of the image is the central zone of the pattern where breakdown of the lithography has occurred at approximately 20 nm feature sizes, while the solid dark arc visible on the right corresponds to line widths of 100 nm size. Line profiles of the image intensity were taken at various feature sizes and are shown offset vertically in the lower left panel. Contrast is defined as the difference divided by the sum of the peak to valley intensities obtained from these line profiles and are plotted in the lower right figure for three distinct microscope conditions. For each condition, the contrast of the features of 150 nm size can be observed in the line profiles to have flat tops and bottoms, indicating maximum contrast of the instrument has been reached. The contrast at this feature size was normalized to a value of 1 for each set of measurements at the three microscope conditions. Plotted as partial modulation transfer functions are: (1) a room temperature or "standard" holder as a reference, (2) a cryogenic sample holder at room temperature, and (3) the same holder at liquid nitrogen temperatures, with cryogen flowing.

It is clear that down to 100 nm feature sizes there is little difference in imaging performance between the three conditions. At 75 nm feature size, the cold holder at room temperature shows similar contrast to the standard holder, but when cold there is a measurable loss of contrast. At 50 nm length scales the cold holder at room temperature shows the effects of the mechanical coupling to the cryostat. The contrast for cold temperature performance is roughly 40% of the standard value at this size scale. At the smallest measureable feature size of 30 nm for this test pattern, a contrast of roughly 25% of maximum remains for the standard, room temperature, instrument, while the cryogenic performance has dropped to a barely discernible 3%, or roughly a 90% contrast loss between the two conditions. Imaging performance is measurably reduced during cryogenic operation at the smallest length scales, but good performance down to 50 nm scale size is possible for all