Design of a multi-axis cryogenic sample manipulator for soft X-ray and VUV spectroscopy

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Abstract. We have designed and constructed several manipulators for cryogenic samples and soft X-ray and VUV spectra. These manipulators are compatible with ultrahigh vacuum and up to six axis motions -- three translational and three angular motions. Three translational and the polar angular motions are implemented with commercial stages. The azimuthal (in the beam direction) and tilting motions are driven with separate gear trains and connected to stepping motors on the top flange (100 CF). The azimuthal angular range is about ±180°, and the tilting range is from 75° to -25°; the resolution is better than 0.1°. The sample position is designed to be situated at the center of the polar and azimuthal rotation axes. The tilting axis is designed with an offset to decrease the spatial interference with the analyzer for photoemission spectra. The sample is attached to the sample holder and transferred to the cryogenic stage via a load-lock system. The sample holder is cooled with a continuous-flow cryostat (Janis ST-400) via flexible copper braids. With liquids helium and nitrogen for the cryostat, the lowest temperature of the sample holder attains 9.15 K and 82.4 K, respectively. During tests, the rate of consumption of liquid helium is less than 0.8 L/h.

1. Introduction

Angle-resolved photoemission spectra (ARPES) provide a powerful tool to probe the electronic structure of solids. A systematic mapping of the band structure of solids and surfaces provides insight into the electronic interactions in those solids. This property is especially important for strongly correlated and nano-structured systems in which intricate electronic interactions and geometrical constraints produce novel quantum effects including competing order parameters, quantum critical behavior, high-temperature superconductivity, topological insulators, heavy-fermion and complex oxides [1]. For modern high-resolution ARPES, energy analyzers with sub-meV energy resolution and 30° collecting angle are commercially available. A sample manipulator plays an important role in the accuracy and efficiency of data collection, but no commercial product satisfies the requirements of various ARPES systems at the same time. To meet these requirements for most research topics, a new sample manipulator has been developed.

2. Design and construction

The design of a sample manipulator for a low-temperature ARPES experiment must satisfy several requirements. First, motion with six degrees of freedom including three translations and three rotations is displayed in Figure 1; three translations serve to move the sample to the measurement position, and three rotations are used to map the entire electronic structure of solids at the Brillouin zone. Second, a helium continuous-flow cooling system is preferable to a closed-cycle refrigerator system because the
former causes less vibration. Thirdly, to meet the requirements of a low transition temperature of high-
temperature superconductors and heavy-fermion systems, a working temperature below 10 K is
essential. Finally, because a residual magnetic field around a sample can affect the accuracy of spectra
at small photon energies, only non-magnetic materials should be used to construct a manipulator.

In the key idea of our design, a continuous-flow cryostat system (Janis ST-400) is an ideal candidate
because of its compact size. Three translations and a polar motion are implemented with a commercial
stage. Azimuthal and tilting motions are driven with stepping motors individually and integrated with
the same cryostat system. The main parts comprise a supporting bracket, a sample holder box and two
rotation mechanisms, as shown in Figure. 1. The sample holder box contains an azimuthal rotation
axis and a tilting axis. Inside the supporting bracket, two ceramic bearings serve to support the
azimuthal axis. A pair of spur gears drives the azimuthal motion. Outside the supporting bracket, a
worm-wheel gear drives the tilting motion. To prevent crosstalk between both axes, flexible cooper
braids connect the cold finger and the sample holder. Titanium alloy and ceramic bearings are adopted
as the materials near the sample mount because of their small thermal conductivity and non-magnetic
property. To connect between the cryostat cold head and the rotatable sample mount, flexible copper
braids provide an effective path for heat transfer. To avoid the thermal radiation from the supporting
bracket, the sample holder box and the supporting bracket are cooled with the pipe of returning chilled
gas of the cryostat system to decrease thermal radiation. As the length of the sample manipulator
exceeds 1 m, thermal contraction occurs to an extent about 2 mm between the cryostat and the
supporting pipe, which induces a large deformation in the transverse direction because of the
temperature difference between the supporting pipe and the rotary shaft. A flexible joint mechanism is
applied in the connecting rods between the gear and the stepping motor to absorb this thermal
contraction.

![Figure 1. Drawing of main parts of the two-axis sample manipulator; polar motion is linked to the
upper rotational stage; the copper braid is not shown here.](image)
In the design of the copper braid joint with a copper block, we adopted a keyhole joint scheme to decrease the thermal resistance. This scheme has a regular cylindrical hole with an additional slot cut through the circular center. After inserting the copper braid then pressing tight the joint, a keyhole shape results that is beneficial for its plastic deformation and wrapping the copper braid after pressing and with no gap. In a preliminary test similar to figure 2, the keyhole joint attained 7.6 K less than a regular cylindrical joint. A similar configuration of this pressing method is found in reference [2].

3. Test Results

To measure the thermal resistance from the cold head to the sample holder, we compared the flexible copper braid/block assembly to a solid copper plate. The measurement was performed in a high-vacuum system; the temperature sensor was a silicon diode (DT-470), which was calibrated by Lakeshore with accuracy 0.25 K; the cold head was cooled with a cryocooler (Mitsubishi). In the copper braid/block assembly, the diameter of copper braids was about 3 mm; the length was about 7.5 cm. The assembly consisted of two braids in parallel with two blocks at both ends with keyhole joints. A copper plate without joint and having the same cross section and length as of the copper braid/block assembly served for comparison. Figure 2 shows the temperature distribution of the copper braid/block and the copper plate. The cold head of a closed-cycle helium refrigerator at 6.55 K served as a cold end, designated as T0. Tj1 and Tj2 denote the interfaces of both joints between the copper braid and the copper block. T1 is the interface between the cold head and the first copper block.

Comparison with the copper plate without joint indicates that the temperature gradient for each joint is about 1.35 K by the key hole and pressing method, (10.44-7.72)/2.

![Figure 2. Temperature distribution of the copper braid/block assembly and copper plate; for the position of the heat path see the text.](image-url)

We examined the cooling performance of the cryogenic sample manipulator with liquids helium and nitrogen. With liquid nitrogen the temperature of the sample holder attained 84.4 K after 30 min and 83.8 K after 60 min. With liquid helium, the temperature of the sample holder was 10.3 K after 30
min and 9.7 K after 80 min. In these tests, the temperature of the radiation shield is an important factor for the minimum temperature of the sample holder and the rate of consumption of helium; for our manipulator, that rate is about 0.8 L/h at 12.5 K. The manipulator was installed at beamline BL21B1 in National Synchrotron Radiation Research Center, Hsinchu, Taiwan. A few layers of graphene (FLG) on a SiC substrate were used to test the performance of the angular motion. The band dispersion of FLG and graphite are expected to be linear near the Fermi level and have a point contact (Dirac point) between valence and conduction bands [3,4]. For a hexagonal surface Brillouin zone, the Dirac point on the Fermi surface should contribute a six-fold symmetry property. These properties allow us to examine the accuracy of the angular motion and the efficiency of sample manipulator. The constant-energy mapping of FLG/SiC was measured near the H or K point of Brillouin zone by varying incident photon energy and rotating the tilt angle with 0.5° step. The FLG/SiC samples were growth by thermal decomposition method and all spectra were taken at 54 eV of photon energy by Scienta R4000 energy analyzer in UHV environment. Figure 3 displays the constant-energy mapping of FLG/SiC at the Fermi surface and 1.0 eV of binding energy near the K point at the Brillouin zone. The Dirac point with six-fold symmetry was located clearly at the H point of Brillouin zone on the Fermi surface. The accuracy 0.1° of the angular motions allowed us to determine the Dirac point precisely. The total result for the constant-energy mapping was completed within 2 h because of the flexible and precise angular motions.

![Figure 3](image)

**Figure 3.** Constant-energy mapping of a few layers of graphene on a SiC substrate. The spectra were recorded at photon energy 54 eV and 82.4 K.

4. Summary
We have designed a manipulator with a large angular range for the measurement of soft X-ray and UVU spectra. The ranges of azimuthal and tilting motions are about ±180° and 75°/ -25° respectively. The total angular resolution is better than 0.1°. The temperature of the sample holder can attain 12.5 K with rate 0.8 L/h of consumption of liquid helium.

5. References
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