Introduction: In the past few years, there has been an increasing amount of interest in the properties of adsorbates on stepped surfaces for a variety of reasons, including enhanced reactivity of steps and nucleation of overlayer growth. However, very few studies concerning the detailed structure or geometry of adsorbates at stepped surfaces have been reported, in part because of the relatively low coverages involved, the problems of isolating adsorbates at step sites, and the lack of long range order which makes the use of diffraction-based techniques less appropriate.

Methods and Materials: In order to examine the geometry of an atomic adsorbate at step sites on a metal surface, we have begun an investigation of S atoms adsorbed at room temperature on the Ni(117) surface, using the technique of back reflection x-ray standing waves (BRXSW). The advantage of BRXSW for studying such systems is that the technique does not require long range order in the adsorbate layer, but only relies on the substrate to produce the standing wave field. In addition, with a new end station featuring a large hemispherical energy analyzer with multidetection capabilities, we have the sensitivity to probe submonolayer coverages of most adsorbates.

Results: This work is being carried out at the X24A beamline using a new end station and detector as described above. The measurements we have made consist of back reflection measurements with the photon beam positioned normal to the (100) terraces of the Ni(117) surface, which involves tilting the crystal so that the beam is at an angle of ~11.5° with respect to the surface normal. In this geometry, standing waves from the (200) back-reflection are produced at a photon energy of ~3526 eV. For our BRXSW measurements we record the reflectivity as well as the Ni 2p and S 1s core levels as the photon energy is scanned over the rocking curve around the Bragg peak. We have carried out preliminary measurements for S/Ni(117) at saturation coverage (where the S molecules are predominantly in terrace sites) as well as for S at lower coverages, where the relative proportion of step sites is higher. The sulfur overlayers were prepared by first dosing the Ni stepped surface with H2S at room temperature, followed by an anneal to drive off dissociated hydrogen.

Acknowledgments: For D. Heskett and J. Warner acknowledgment is made to the Donors of The Petroleum Research Fund, administered by the American Chemical Society, for partial support of this research.