Experimental Capabilities in the Kelber Group

The Kelber Surface Science Laboratory within the UNT Department of Chemistry possesses four multi-technique surface science systems. These systems combine complex reaction/deposition/electrochemistry environments with Photoemission, Auger Spectroscopy, and other techniques for detailed surface analysis. An important feature of all these systems is the ability to transfer a sample between reaction and analysis environments *in situ*, i.e., without sample exposure to ambient: These systems are described below:

1. UHV-EC system with XPS, UPS (System 1). The existing system for combined UHV-EC studies is shown schematically in **Fig. 1.** This system is equipped with a standard 3-electrode cell that can be translated until the meniscus wets the sample surface, permitting electrochemical measurements. The sample can be emersed at controlled potential, rinsed (if desired), and translated into UHV for both x-ray photoelectron spectroscopy and ultraviolet photoelectron spectroscopy (XPS, UPS) The base pressure in the turbopump electrochemistry chamber is ~ 10^{-7} Torr after withdrawal of the electrochemical cell. The electrochemical cell uses a Pt counter electrode, Ag/AgCl reference electrode, and an EG&G PARSTAT 4000A Potentiostat/Galvanostat/ESI Analyzer. The existing, operational UHV chamber (base pressure 2 x 10^{-10} Torr) is turbomolecularly pumped and contains a sample stage with sample cooling-heating capabilities (100 K – 1100 K) by a combination of resistive heating and LN_2 cooling. The chamber

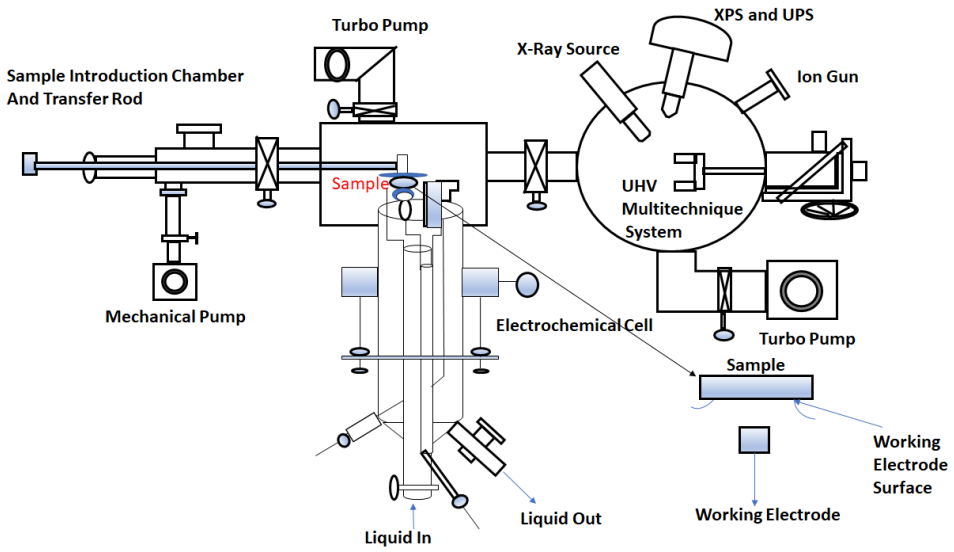

Fig. E1. Schematic of UHV-EC apparatus. The sample surface is wet by the meniscus from the cell. Emersion is at controlled potential.

is equipped for Ar ion sputtering for sample cleaning and has a PHI 5400 140 mm mean radius hemispherical analyzer with a multichannel plate detector for XPS and UPS. The XPS source is a Physical Electronics 04-548 dual anode (MgKα/AlKα) unmonochromatic X-ray source. The UPS source is a SPECS UV 10/35 differentially pumped plasma source.

2. XPS, LEED, plasma and Atomic Layer Deposition System (System 2) This system is shown schematically in **Fig. 2,** and combines capabilities for XPS, LEED, Mass spectrometry, with sample transport between chambers under rigorously controlled conditions. The UHV surface analysis system is equipped with a VSW 100 mm mean radius hemispherical analyzer and PHI 04-548 dual anode x-ray source for XPS, an Omicron 3 grid reverse-view LEED system, a PHI Ar ion sputter source for sample cleaning, and a Stanford Research Systems 300 amu quadrupole mass spectrometer. This system also has an Oxford Applied Instruments thermal cracker for creation of free radicals under UHV conditions. Transport between chambers occurs under rigorously controlled vacuum conditions.

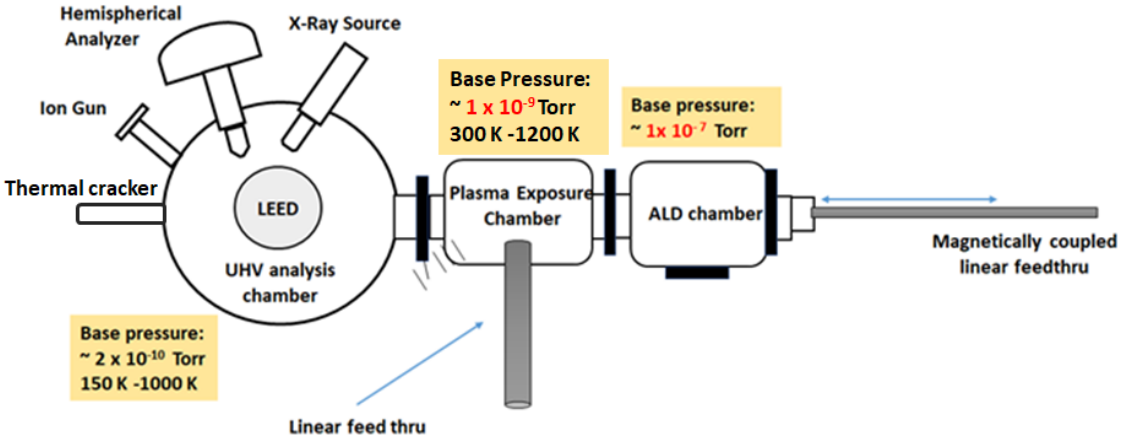

Fig. E2. Schematic of system with capabilities for LEED, XPS, mass spectrometry, DC Magnetron sputtering and (proposed) enhanced electrochemical cell with photoelectrocatalysis capabilities

Magnetron sputter deposition and AES analysis (System 3) The multichamber system for magnetron sputter deposition is shown schematically in **Fig. E4** and is fully operational. The sample stage allows sample cooling/heating between 200 K and 1500 K with LN_2 cooling and resistive heating. Sputter deposition is from commercially available DC magnetron (Meivac, " Mini-Mak") sources with a source-sample stage distance of 3 inches. AES and low energy

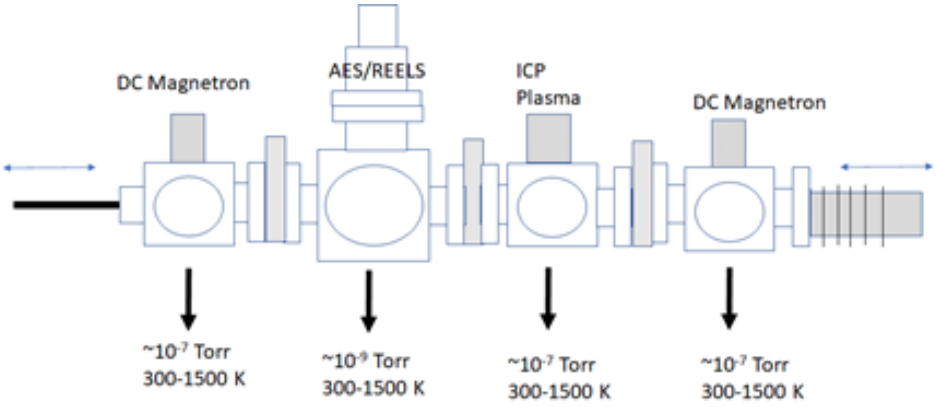

Fig. E3. Schematic of magnetron sputter deposition cluster system, with turbo-pumped

reflective electron energy loss (REELS) analysis is carried out by a Staib ESA100 single pass

cylindrical mirror analyzer with co-axial electron gun. Each of the three chambers shown is pumped by a turbomolecular pump.

MBE/XPS/AES-REELS/LEED/STM (System 4) This system, shown schematically in **Fig. E4** contains a turbomolecularly pumped chamber (base pressure; 1 x 10^{-9} Torr) with a four source electron beam evaporator (Mantis) for molecular beam epitaxy (MBE), and an ion-TSP-pumped chamber (base pressure 5 x 10^{-11} Torr) equipped with a reverse-view LEED I(V) system (Omicron); and a double-pass cylindrical mirror analyzer with co-axial electron gun (Staib DESA100 for Auger Electron Spectroscopy and Reflective Electron Energy Loss Spectroscopy (AES-REELS), as well as a dual anode (Mg/Al) X-ray source (Physical Electronics 04-548) for XPS acquisition. This chamber also has an ion sputter gun for sample cleaning and an Omicron ambient temperature STM.

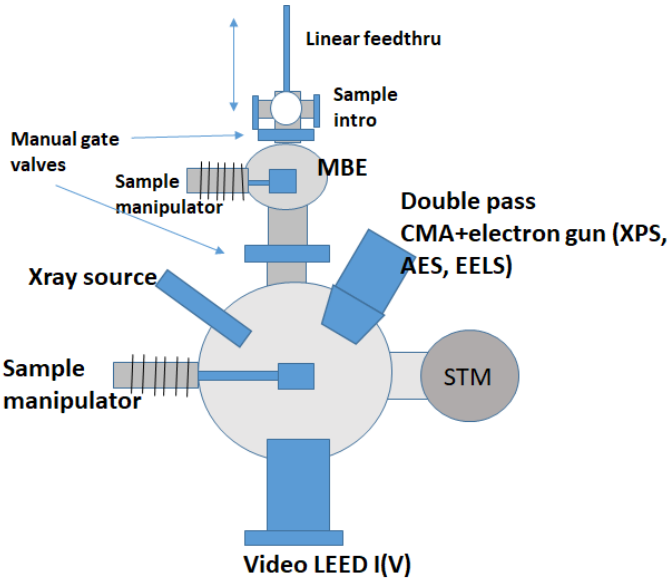

Fig. E4 Schematic of MBE/XPS/AES-REELS/LEED/STM system

Other Equipment in the Kelber Group In addition to the above systems, the Kelber group possesses:

- An Electrochemical Atomic Force Microscope (VEECO Nanoscope E)—this will be used to assess *operando* changes to surface morphologies and possible surface instabilities

- A Tube Furnace (Lindberg)—this can be used, as necessary, to make thin carbide films on metal substrates by heating in a flow of CH_4

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