4. Experimental setup

4.1 UHV system

All experiments were performed in a UHV system designed and set up during the course of the work presented here. It consists of the main chamber, where measurements were performed, and a preparation chamber used for thin film evaporation. Both vacuum chambers were connected by a home made sample transfer system. The base pressure was between $8*10^{-11}$ and $4*10^{-10}$ mbar in the main chamber and between $8*10^{-10}$ and $3*10^{-9}$ mbar in the preparation chamber. Dosage of the surfaces with oxygen, nitrogen, carbon dioxide and argon was accomplished by an evacuated gas inlet system connected to the UHV chamber by a leak valve. A separate leak valve was used to admit water vapor to the chamber. A UHV crystal cleavage mechanism for the preparation of clean CaF_2 surfaces was designed. Figure 4.1 shows a schematic drawing of the vacuum system.

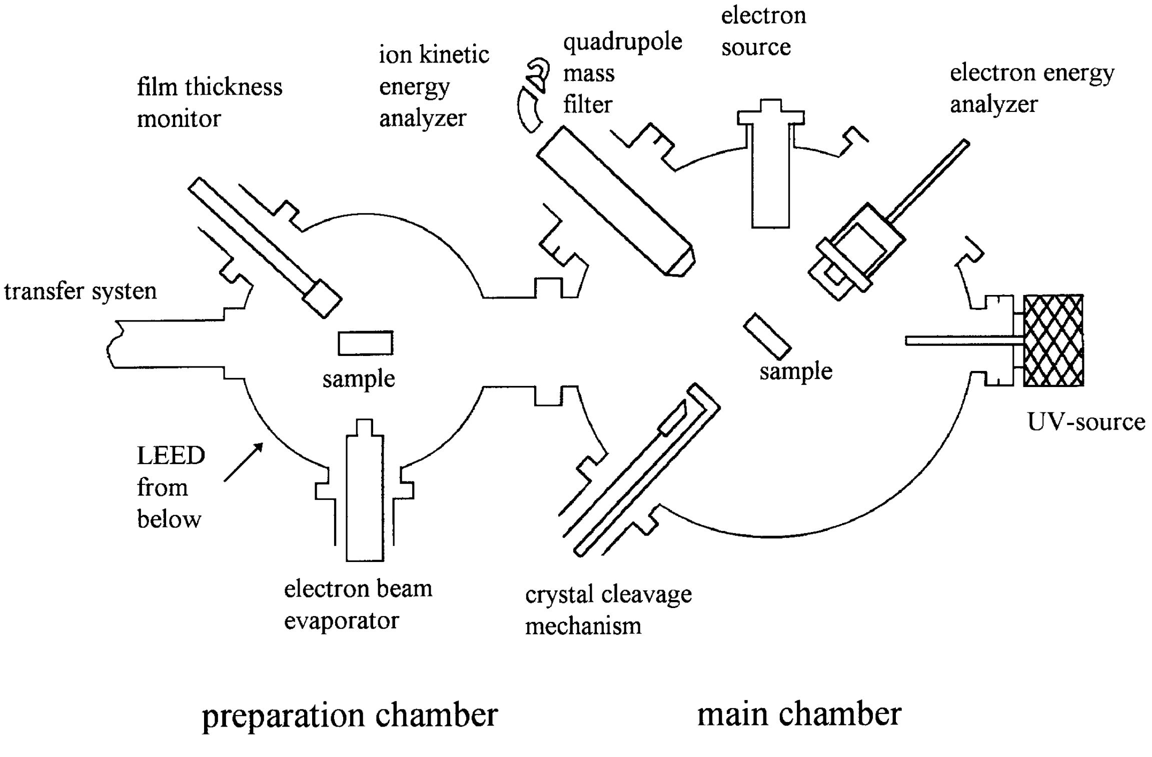

Fig. 4.1 Drawing of the vacuum chamber

Samples were transferred between the chambers on sample holder plates that could be easily adapted to the requirements of different experiments. Mainly three designs were used:
a) A flat plate designed for the investigations of crystals cleaved in air. The crystals of 20 x 20 x 3 mm^3 dimensions were clamped to the plate with tantalum springs. The sample could be heated by means of a resistive heater element to about 670 K and cooled to about 170 K by liquid nitrogen. The temperature was measured with a thermocouple.
b) For crystals to be cleaved in UHV, a device that resembled a pair of tongs was designed. A 6 mm ∅ rod of CaF_2 could be held between the tongs. To produce a fresh surface, the rod was pushed forward in the sample holder and a disk of approximately 1.5 mm thickness was cleaved away. The lengths of the fresh rod was 35 mm, therefore it was possible to perform about 20 measurements without changing the sample. In this sample holder, the sample could be heated to 900 K and cooled to 140 K.
c) For the preparation of thin films, the ends of the silicon substrates of dimensions 5 x 25 x 0.5 mm^3 were clamped to a copper block by tantalum springs. The high temperatures needed for cleaning the sample (1400 K) and during film preparation (1000 K) were achieved by direct current heating. For the photoemission experiments, the sample could be heated by a resistive heater element to about 670 K and cooled to 130 K. The temperature was measured

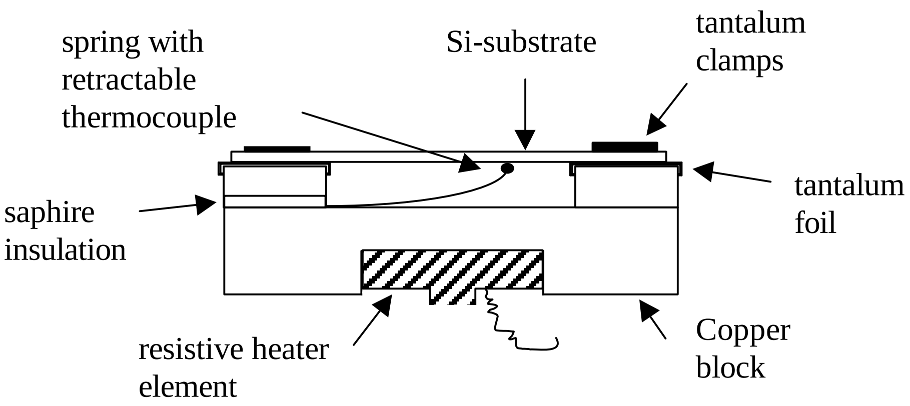

Fig. 4.2 Design of the sample holder plate used for thin film preparation. Heating to achieve the high temperatures needed for film preparation is accomplished by current flow through the substrate. To avoid contamination during heating, the thermocouple can be retracted from the substrate with a wobble stick. During measurements, the sample can be heated with a resistive heater element. For cooling, a cold finger connected to a liquid nitrogen reservoir via a flexible copper cord can be attached to the sample holder plate.

with a thermocouple that was pressed against the Si substrate from the back side by means of a spring. To avoid damage of the thermocouple and contamination of the substrate during cleaning and film evaporation, the thermocouple could be retracted from the substrate by manipulation with a wobble stick. A schematic drawing of the sample holder plate used for thin film preparation is given in Fig. 4.2.

In the main chamber, the plates were held in a frame providing electrical contacts for heating and temperature measurement. A flexible copper wire connected to a liquid nitrogen reservoir could be attached to the sample holder plates with a wobble stick. The sample could be positioned by means of a stepper motor driven manipulator. The following techniques were available for experiments in the main chamber:

4.2 Ultraviolet photoelectron spectroscopy (UPS)

For UPS measurements, the sample was irradiated with 21.2 eV photons from a commercial HeI UV gas discharge lamp. The flux of photons from the source was determined to be about $2x10^{11}$ photons/s by measuring the photocurrent with a gold film, and correcting for the photoelectric yield for gold [GKu79]. The spot diameter on the sample was estimated to be about 1.5 mm. For some experiments a continuous irradiation of the sample was not desired. Therefore, a shutter was installed preventing the UV light from impinging on the sample surface between measurements. The pressure in the main chamber rose to about $5*10^{-9}$ mbar during lamp operation. Since the pressure rise is only due to the pure He gas, the sample surfaces was not polluted. The fixed angle between UV source and electron energy analyzer was 55°. Angular dependent photoemission experiments were performed by turning the sample holder, however, in this way only the polar angle υ could be changed. For thin film samples, the wavevector $k_{\|}$ was changed along the Γ-M line of the surface Brillouin zone. The azimuthal angle remained undetermined for the rod shaped CaF_2 crystals.

Photoelectrons were recorded with a double pass hemispherical energy analyzer according to the design proposed by Jost [Jos79]. The instrument was operated at a constant pass energy of 10 eV, and spectra were taken by applying an accelerating or retarding field to the analyzer. The energy resolution of the system of ±80 meV was measured on a silver (100) sample (see Fig. 4.3). For this spectrometer, the kinetic energy of electrons emitted from the Fermi edge of silver was found to be 16.4 eV.

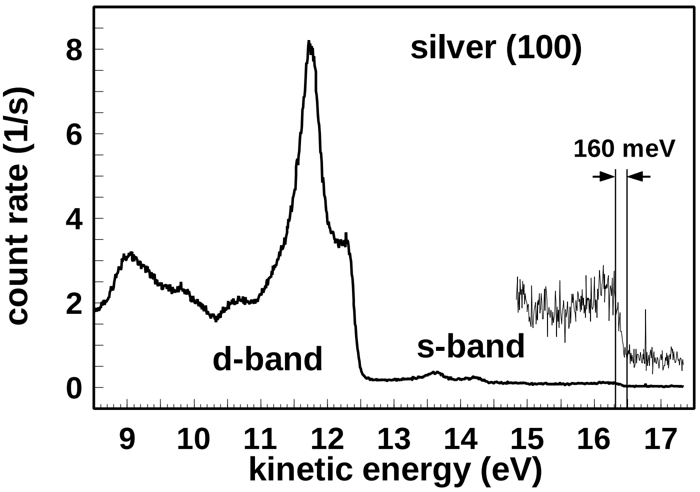

Fig. 4.3 Photoemission spectrum taken from a silver (100) surface. The two small structures at 13.6 and 14.5 eV kinetic energy are due to excitation of d-band electrons with HeI satellite lines at 23.09, 23.75, and 24.05 eV photon energy.

4.3 Optical spectroscopy

The setup for the measurement of optical absorption spectra was designed and setup by N. Bouchaala [Bou97] during for his diploma thesis. It consists of a Xenon high-pressure lamp, a grating monochromator, a light chopper, an optical fiber and two diodes, the first recording a reference signal before the beam enters the vacuum chamber, and the second the intensity transmitted through the crystal. Spectra were taken in the wavelength range from 900 to 200 nm (corresponding to an energy range from 1.37 to 6.2 eV), the resolution was 8 nm. The signal from the photodiodes was detected with lock-in amplifiers to improve the signal-to-noise ratio and remove artifacts from background light. The setup is schematically depicted in Fig. 4.4.

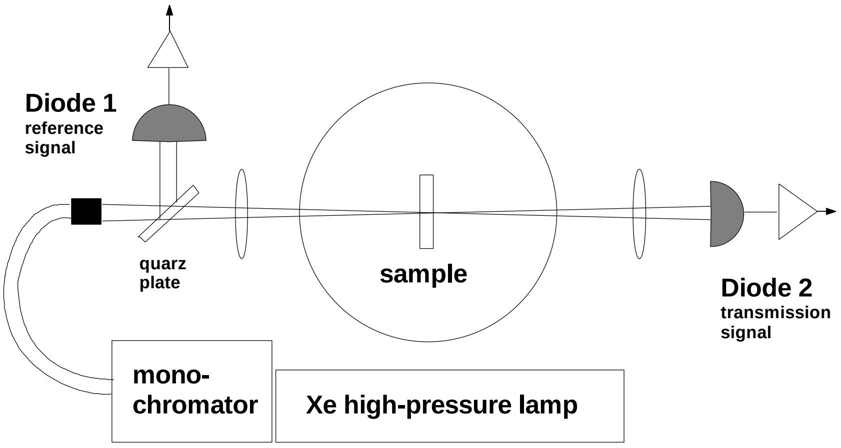

Fig. 4.4 Schematic drawing of the setup used for optical transmission spectroscopy.

The quantity that is determined with this technique is the optical density. It is defined as OD = -log(I_t / I_0), where I_0 is the intensity in front of the sample and I_t the transmitted intensity. Light is not only absorbed by the defects, but also scattered. The total loss consisting of both these mechanisms is called extinction. The optical density of the irradiation induced defects is determined by measuring the extinction before and after irradiation. In a scan performed before irradiation the reference intensities R_1 and R_2 at the two diodes are recorded. After irradiation, the respective intensities I_1 and I_2 are measured in the same way. The optical density of the irradiation induced defects is given by:

$$OD(\hbar w) = -\log\left(\frac{I_2(\hbar w)}{I_1(\hbar w)} \frac{R_1(\hbar w)}{R_2(\hbar w)} \right)$$

By this referencing procedure, the spectral intensity distribution of the lamp, and the extinction from UHV viewports, lenses and the bare crystal are compensated.

4.4 Electron stimulated desorption (ESD)

Exposure of the sample surfaces to electrons of 0.6 to 3.0 keV primary energy was done with a commercial electron source. Beam currents were typically between 2 and 15 µA, the electron spot size on the sample was 4x4 mm^2, thus the resulting current densities ranged from 12 to 90 µA/cm^2. A pulse counting quadrupole mass spectrometer with a parallel plate energy analyzer was used to measure the ion kinetic energy distribution. The calculated energy resolution of the instrument was 0.7 eV, a measurement gave a somewhat lower resolution of approximately 1 eV (see Fig. 4.5).

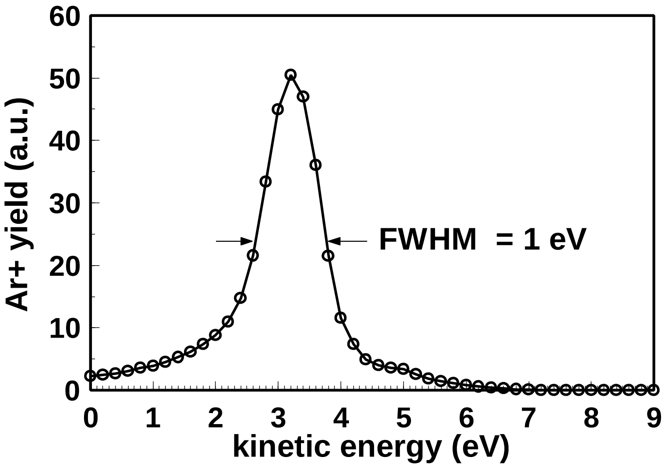

Fig. 4.5 Determination of the energy resolution of the quadrupole mass filter. Sample was a Si (111) substrate as used for the preparation of thin CaF_2 films. The sample holder was at a potential of +5 V during the measurement. Primary energy was 1.5 keV, the current density was 60 µA/cm^2. The experiment was performed at room temperature (RT = 300 K).

4.5 Preparation of thin epitaxial CaF_2 films on Si (111)

The preparation chamber was equipped with an electron beam evaporator, a film thickness monitor and a LEED (Low Energy Electron Diffraction) optics. It also contained a sample magazine for storage of three sample holder plates. The high temperatures during film production were checked with an infrared detector.

The heteroepitaxy of CaF_2 on Si(111) was extensively investigated in literature [RHK87], [DRH95], [HLO95]. To achieve well prepared films, it was found essential to heat the Si substrate to about 950 K during evaporation of CaF_2 [OBr90a].

The Si (111) substrates of 5x25 mm dimensions were cut from a commercial wafer (7.5 Ωm, n-type Ph-dot.) of 0.5 mm thickness. The substrate was clamped to the sample plate and transferred into UHV. After bakeout of the preparation chamber, the sample was cleaned by overnight heating at T = 900 K. The SiO_2 layer was removed by flashing the substrate to 1400 K for 10 to 20 s. Then the substrate was slowly cooled down from 1150 to 1050 K to obtain a Si(111) 7x7 reconstruction. The quality of the surface after this procedure was checked with LEED. For the evaporation of the CaF_2 film, the substrate was held at a temperature of 950 K. The pressure in the chamber was typically $5x10^{-9}$ mbar during film preparation. For the first experiments, CaF_2 powder (Merck suprapur) was evaporated from an electron beam heated molybdenum crucible at a rate of 3.5 to 4.0 nm per 100 s. During the later experiments, it was found more convenient to use a crystal that fitted snuggly into the crucible as evaporation material, reducing strongly the time needed for outgasing. It was also found that similar evaporation rates as for powder could be obtained with much less power. The sample was transferred to the main chamber and the quality of the CaF_2 film was controlled with UPS.