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Development of an Ultra-High Vacuum X-Ray Oven

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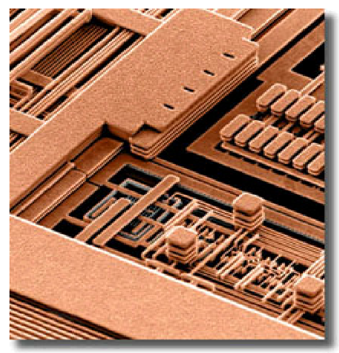
Motivation



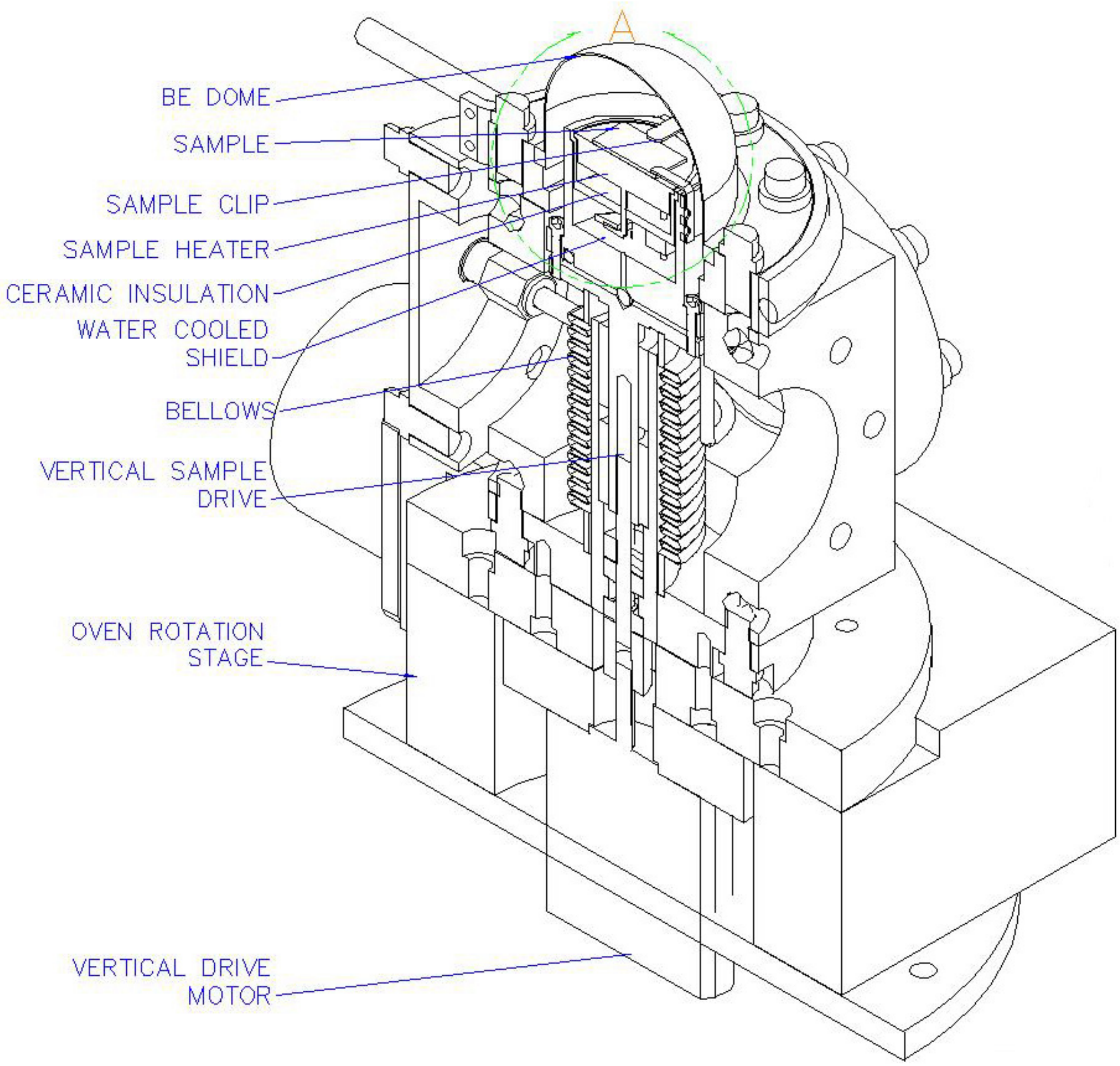
•Thin film devices are used in applications such as MEMS, optical coatings and integrated circuits (shown on right)
•Failure mechanisms and device reliability depend on the mechanical properties of thin films
•Mechanical properties of thin films have been studied by inducing strains in the films by thermal cycling
•The strains originate from a difference in the thermal expansion coefficients between the film and substrate
•X-ray diffraction experiments have been used to measure strain by using lattice plane-spacings as virtual strain gages
•Synchrotron radiation allows measurements on small sample volumes as well as for time dependent phenomena

Experimental Requirements

•Large temperature range to induce the largest possible strains
•Oxygen free environment to prevent oxidation of metal films
•Single crystal diffraction geometries to measure strains in specific crystallographic orientations
•Precise sample positioning and stability during thermal cycles
•Sample displacements (normal to the film plane) away from the center of rotation (COR) of the diffractometer cause systematic errors in strain measurements
•Thermal expansion of the heater assembly during thermal cycling will cause sample displacements

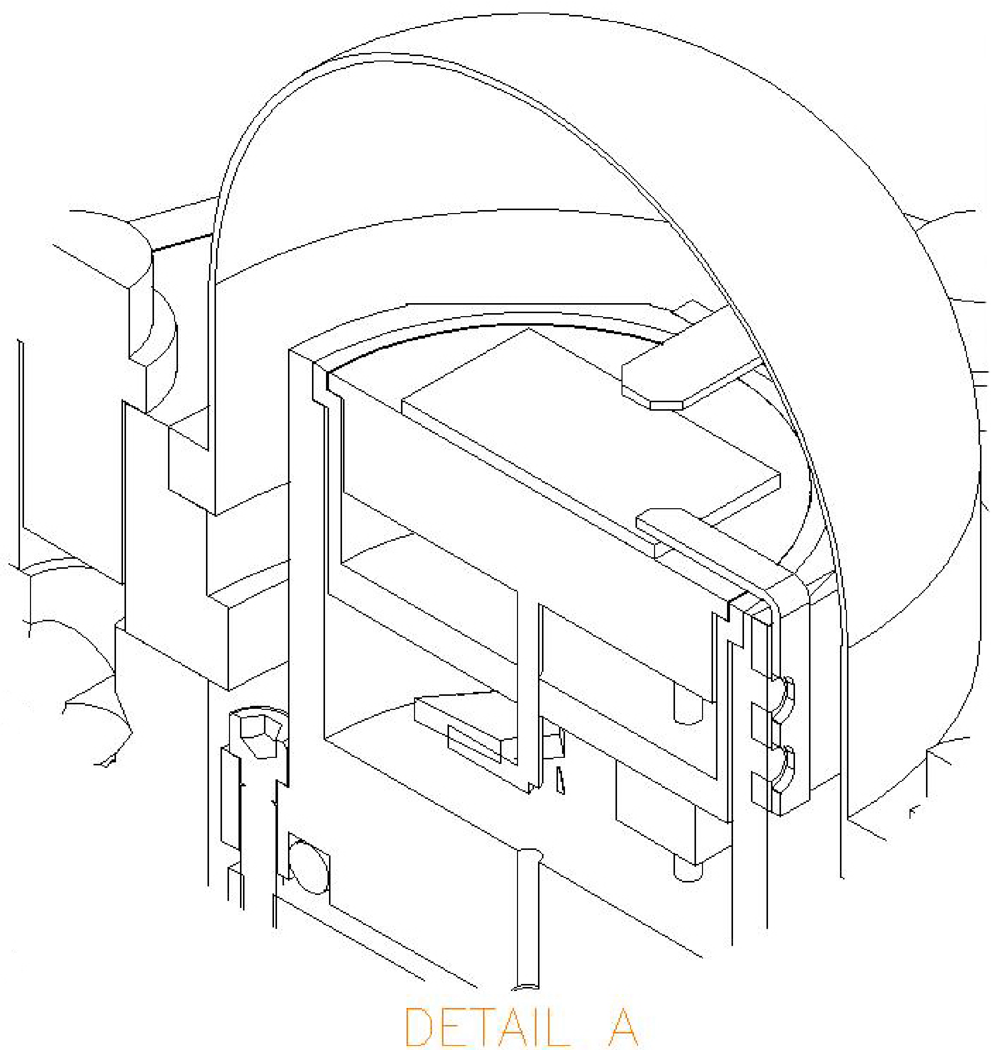
Chamber Assembly

•The vacuum oven is mounted on a Huber 410 rotation stage (shown here in cross-section)
•The chamber body is made of a stainless steel cube with Conflat flange ports
•The chamber is capped by a beryllium dome, which allows complete access for reflection geometries
•The heater assembly (shown in detail in the next panel) is mounted to the vertical sample drive
•The vertical sample drive is separated from the sample environment by a bellows



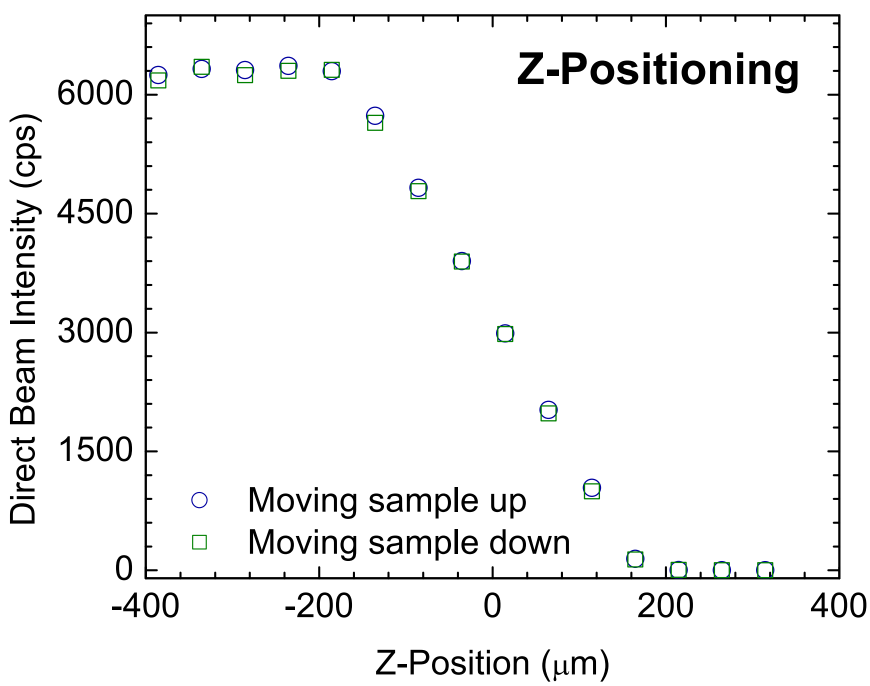
Heater Assembly

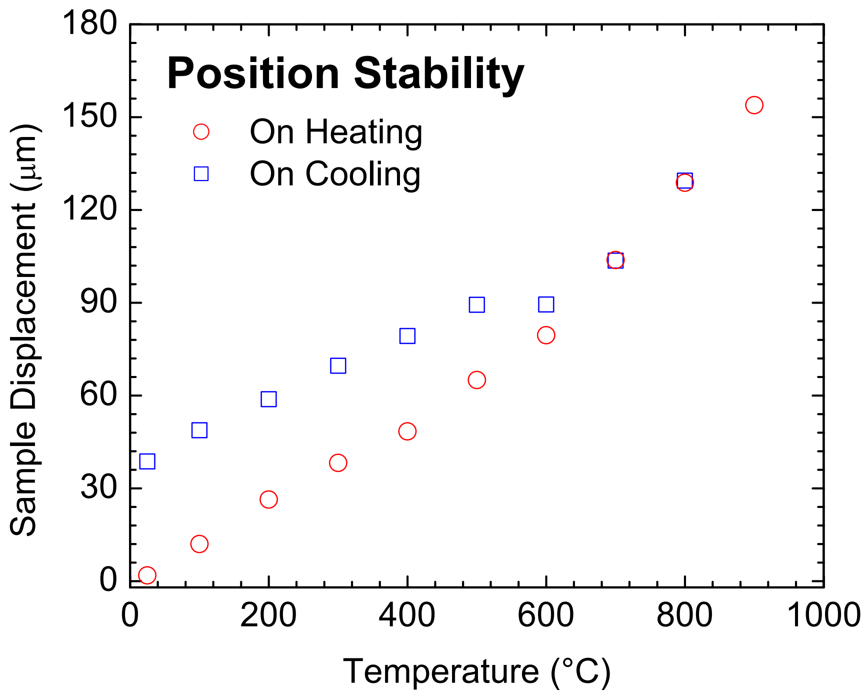
•Engineered to minimize sample displacements during thermal cycles
•The water cooled copper shield determines a reference plane
•A ceramic (Macor®) insulator provides a thermal barrier between the heater and water cooled shield
•Heater (custom button heater by HeatWave Labs) and insulator are inset into the shield such that all vertical thermal expansion occurs in thin lips at the surface of the heater and insulator (0.125 cm and 0.250 cm, respectively)
•The sample clips also hold the heater down
•The sample clips have a low profile design
•Four thermocouples are used to monitor the temperature characteristics of the assembly

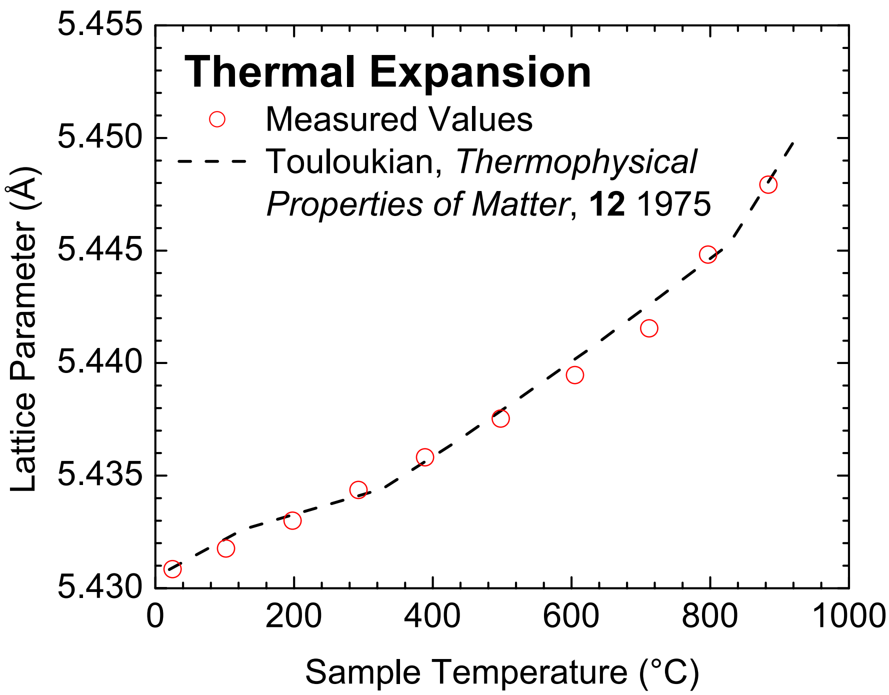


Sample Positioning and Stability

•A single crystal (001) Si wafer was used to measure the sample positioning and stability characteristics of the oven
•The COR was found by monitoring the direct beam while scanning the sample with the vertical drive motor
•The Z-position corresponding to half the maximum intensity was taken to be the COR
•The Z-position scans are highly reproducible, even when scanning in a reverse direction
•Sample displacements as a function of temperature were determined from Z-position scans taken during a temperature cycle
•The position stability is consistent on heating and can be calibrated but the heater assembly design must be further refined to maintain a consistent trend on cooling
•Using the sample displacement calibration for heating, the lattice parameter of Si (measured from (400) peaks) as a function of temperature was found to be in good agreement with literature values

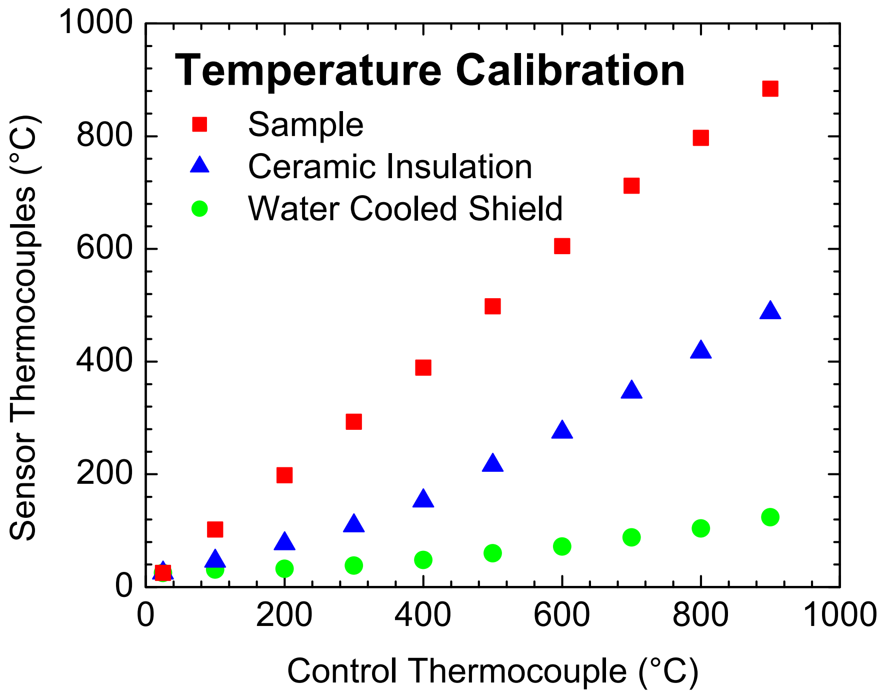



Position Stability
On Heating
On Cooling
Sample Displacement (μm)
Temperature (°C)
0
30
60
90
120
150
180
0
200
400
600
800
1000


Thermal Expansion
Measured Values
Touloukian, *Thermophysical Properties of Matter*, **12** 1975
Lattice Parameter (Å)
Sample Temperature (°C)
5.430
5.435
5.440
5.445
5.450
5.455
0
200
400
600
800
1000

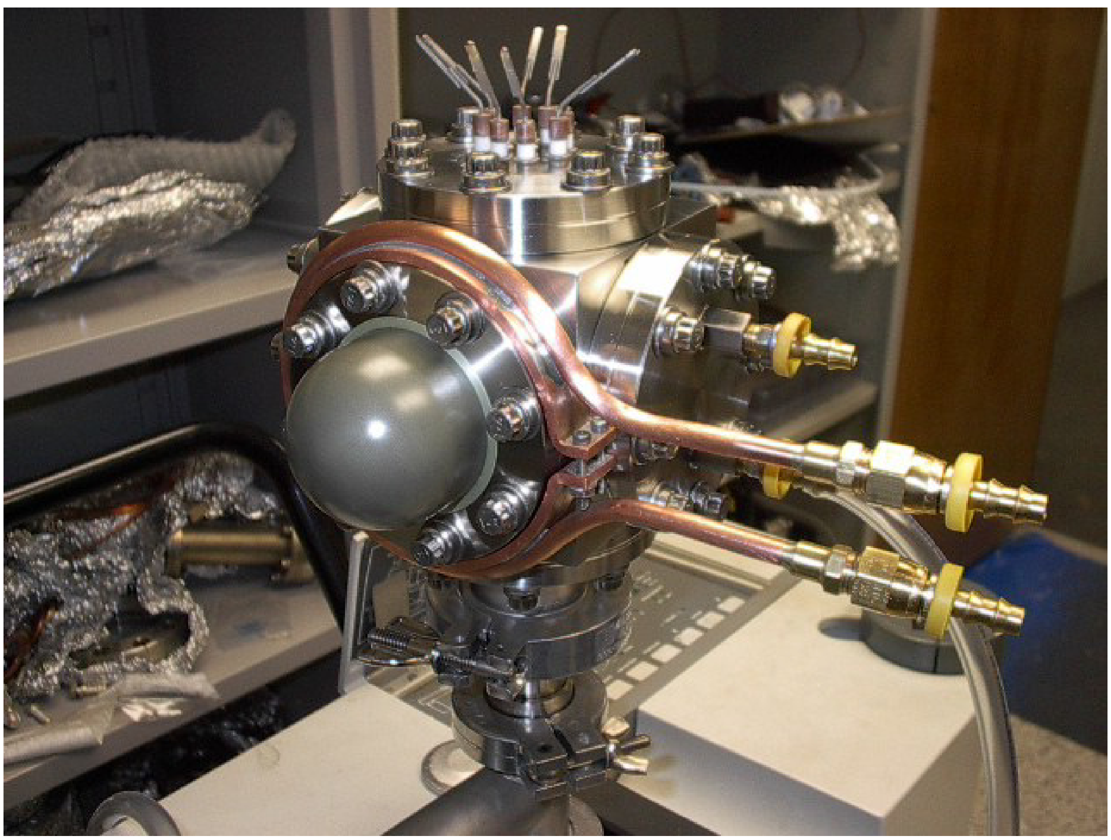
Temperature Characteristics

•The temperature of the heater, sample, ceramic insulation, water cooled shield, beryllium dome and cooling water water were monitored during a thermal cycle
•Power was provided to the heater based on the temperature measured by the heater (Control) thermocouple
•The maximum temperature of the beryllium dome was 65°C
•The cooling water remained at 16°C ±1°C
•The temperature of the water cooled shield was greater than expected and resulted in larger than expected sample displacements (discussed below)
•The cooling water system will be optimized to reduce the maximum temperature of the shield



Vacuum Oven Attributes

•Temperatures up to 900°C
•Designed for Ultra-High Vacuum (UHV)
•Single crystal diffraction geometries
•The sample orientation makes full use of the Phi, Chi and Omega diffractometer rotations
•No obstacles to the incident or diffracted beams above the sample
•The heater assembly is designed to reduce sample displacements caused by thermal expansion
•Precise sample positioning is possible with a motorized vertical sample drive
•The sample normal can be aligned to the Phi axis with screw adjustments that work like a two-axis goniometer



Summary

•A new oven has been developed to make synchrotron x-ray diffraction measurements on plate samples in a UHV environment
•Initial commissioning experiments were performed to characterize the thermal profiles of the heater assembly and the sample position as a function of temperature
•The maximum sample displacements from thermal cycling can be reduced by optimizing the water cooling system
•The sample position as a function of temperature can be calibrated on heating but adjustments to the heater assembly are required to maintain the calibration on cooling
•Good agreement was found between measured data and literature values for the temperature dependent lattice parameter of Si after performing the Z-position calibration