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High pressure and high temperature aqueous environment atomic force microscope

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Abstract

We propose a low-cost design for a high-pressure, high-temperature, aqueous environment atomic force microscope. A key point of this design is the use of elastomeric seals as flexure elements for short-range motion while allowing for long stroke coarse motions. Initial experiments with a pressurized hydraulic cylinder at room temperature demonstrate feasibility over at least a 5 micron short-range actuation, and that stick-slip should thus not interfere with the AFM measurement.

In-situ, atomic force microscope

1. Introduction

Atomic force microscopy (AFM) is widely used to study material characteristics, and in-situ operation can provide important data for application-specific materials and coatings. While high temperature [1-6], high pressure [7-9], and aqueous [10-11] operation have all been explored independently, we present an instrument for the in-situ AFM study of samples in a high temperature (350 °C) and pressure (17 MPa) aqueous environment. To attain the required spatial resolution with actuators located outside the extreme environment, we use the elasticity of sealing elements to allow small-range high-precision actuation, while the seals slide for large motion loading and initial positioning of the samples. The low cost system developed uses conventional actuators and fluid cylinders with rod glands having added cooling to withstand the extreme environment.

2. Low Cost Actuation

The main challenges faced in extending atom probe microscopy techniques outside typical laboratory conditions are moving the probe and sample with approximately 1 nm resolution, and recording the probe tip location in the z-axis with better than 1 nm resolution. For tapping mode measurements it is also necessary to excite the tip to oscillate at a specific frequency normally in the 10-100 kHz range. The devices used to accomplish the above, including piezoelectric actuators and laser diodes, cannot be exposed to extreme conditions. Developing novel technologies specifically for each extreme condition is cost and time prohibitive, therefore we present a chamber design that uses existing technologies but places them strategically outside the extreme environment. This separation is enabled by elastic seals, a pressure balanced design, and combined axis motion.

2.1. Elasticity of sealing elements

The AFM probe is mounted on a flexural stage actuated by a rod that extends through the seals and passes to the outside of the cylinder (pressure vessel) where it is driven by piezoelectric actuators. The seals in the rod gland allow sliding motion for coarse motion, and act as flexures for nano motion resolution in the measurement-critical z-axis [12]. To prove this concept, an experiment to find the rod gland spring constant over a 5 µm rod displacement range is described below.

2.1. Pressure balanced design

When the AFM chamber is pressurized to 17 MPa, a 10 mm diameter rod will experience over 1 kN outward force. While this can be handled with appropriately sized actuators, much smaller actuators can be used when the rod is allowed to penetrate two sides of the chamber, resulting in no pressure load. Additionally this provides two points of support for the rod to reduce Abbe error. Long-range coarse actuation using a stepper motor linear stage in series with the piezo enables exploration of multiple parts of a macroscopic sample, or of multiple samples within a single trial.

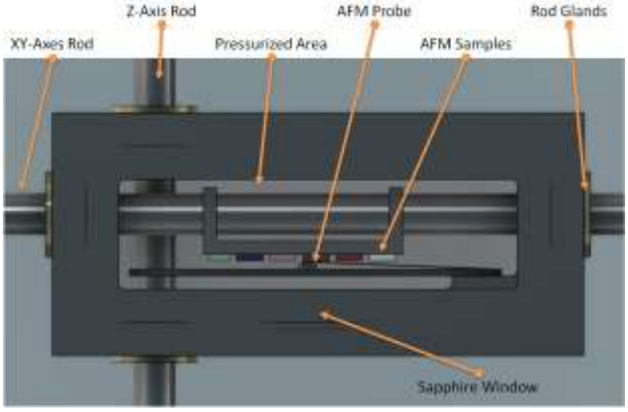

Figure 1. AFM Pressurized Heated Chamber Layout.

2.2. Combined axis rotation and translation

The sliding of the X axis rod provides translation in the x direction along the sample. To avoid the need for another rod, scanning in the y direction is achieved by rotating the X axis rod and counteracting the small radial shift of the scan plane by moving the probe down using the Z axis rod. If the samples are modestly offset from the axis centreline by 5 mm, y axis motion up to ±0.5 mm is achieved with a rotation of ±6 degrees and z axis motion by up to -0.03 mm.

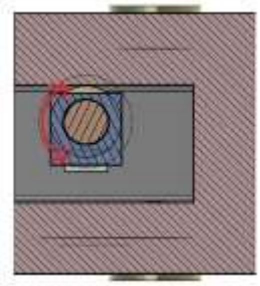

Figure 2. Demonstration of motion along sample y axis as result of rotation of the X axis rod.

3. Laser System

To measure AFM probe motion inside the chamber, we cannot use the standard optical lever approach, since the sapphire window that forms the pressure barrier limits both the maximum angle of incident light and the minimum distance to optical components. Instead we use an already proven interferometric approach. The proof of concept prototype uses a simple polarized interferometer, while the final design will use Laser Doppler Velocimetry (LDV) for improved accuracy and noise immunity [13, 14]. The developed concept of photothermal AFM probe actuation [14] can be used in this system to overcome the difficulty of the pressurized chamber in driving the AFM probe at high frequencies.

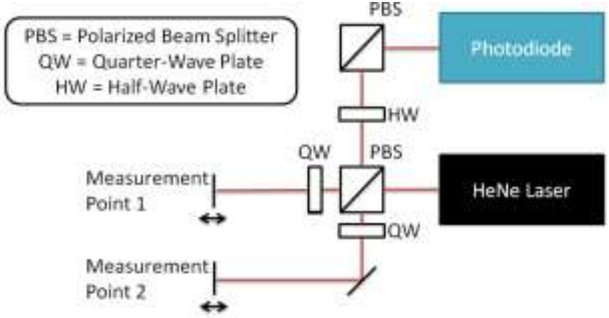

Figure 3. This interferometer setup was used to verify seal elasticity.

4. Preliminary Results

We use a double rod end hydraulic cylinder, with internal piston seals removed, as a simple test of the above actuation concept. Using a heated steel bolt as a low-range high-force actuator, we apply a force to one end of the rod, and measure displacement of the other end using the interferometer of Fig. 3. We then find that the force on the actuator follows Hooke's law with a spring constant that is a function of internal pressure in the hydraulic cylinder. Assuming the seal acts as a spring we can calculate effective seal stiffness as follows:

$$k_{eff} = \frac{k_B}{\frac{k_B}{k_A}+1}\left(\frac{v_u}{v_l}-1\right)$$

Where k_B is the stiffness of the bolt and k_A is the stiffness of the actuator assembly, v_u is expansion velocity with no load, and v_l is expansion velocity when coupled to the pressurized hydraulic cylinder. The reasonable agreement of the calculated stiffness with linear deformation of rubber elements, along with the observed return of the rod to its original position as the bolt cools down, indicate that these seals act as flexural elements within at least five microns displacement range.

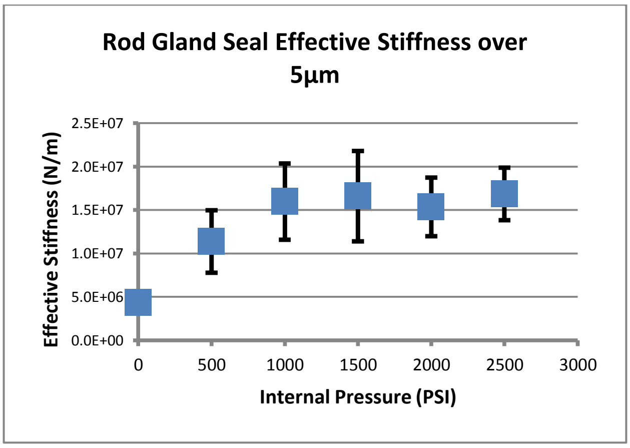

Figure 4. Results of the seal stiffness measurement in the hydraulic cylinder over a displacement range of 5 microns.

No signs of stick-slip above the range of 100 nm are observed. This suggests that the z-axis may need a factor of 10 motion reduction using a flexure cantilever in order to achieve 10 nm resolution. A seal stiffness plateau is reached between 1500 and 2000 PSI, with the highest stiffness in the range of 10^7 N/m.

5. Conclusion

Sufficient actuation for AFM scanning purposes can be achieved in a compact manner using two perpendicular rods crossing inside a heated pressure boundary. Initial results support the use of elastomeric rod glands despite the high scanning resolution since the seals act as flexures for low displacements. The effective stiffness of the seals is shown to depend on the internal pressure of the chamber.

Acknowledgements

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