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# Controls of Fluid Chemistry on Fracture Growth

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# **FY06 LDRD Final Report**

## **Controls of Fluid Chemistry on Fracture Growth**

### **LDRD Project Tracking Code: 05-ERD-035**

#### **Carol J. Bruton, Principal Investigator**

### **Abstract**

During this two year project (the original proposal requested 3 years funding) we developed and tested a new design for a mini-bending jig for the hydrothermal atomic force microscope (HAFM) and a modified design for the HAFM itself. These new capabilities now permit study of the connection between stress and mineral dissolution and growth, as well as sub-critical crack growth (SCG). We demonstrated the successful design by imaging SCG of glass in situ, in real time in the HAFM, as a function of changing solution pH. We generated a movie of the SCG process. We successfully accomplished our project objectives through year 2.

### **Introduction/Background**

The role of water and its dissolved content in fracturing and rock deformation is poorly understood. However, new models describing the controls of surface chemistry on fracturing, and the relation between fracturing and the breakage of bonds during mineral dissolution suggest new ways to quantify the impact of fluid chemistry on fracturing using the HAFM. We are using the HAFM to image fracture growth as a function of fluid chemistry and pH at subsurface temperatures. The project goal is to modify the HAFM to quantify these effects and advance our understanding of the role of solute-bearing water on rock deformation. This knowledge can then be used to predict and engineer fracture growth in subsurface materials as a function of their environment.

### **Research Activities**

We modified the HAFM to optimize its use for SCG studies. We built a mini-bending jig to apply stress to a sample loaded inside the HAFM. We designed a Kalrez membrane that permits a wider z-range of motion, designed a longer-range piezo tube that extends the x-y scanning range by a factor of nearly 4x, replaced the contact-only (2 detector) optical head with a Top View AFM/LFM (4 detector) optical head that permits Frictional Force measurements. We confirmed the performance of the bending jig using using a 3D FEM to simulate the bending and calculate the stress. We demonstrated the successful completion of project objectives by imaging SCG of glass in situ, in real time in the HAFM, as a function of changing solution pH. We generated a movie of the SCG process.

### **Results/Technical Outcome**

The bending jig that we designed and constructed (Fig. 1) permits us to

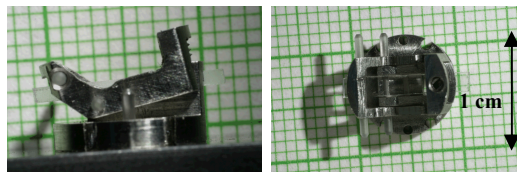


Fig. 1 Mini-bending jig for HAFM

achieve stress intensity factors ranging from  $2.5 \times 10^{-2}$  to  $7.2 \times 10^{-1}$  MPa\*m<sup>1/2</sup>. This covers the stress range pertinent to most materials of geologic significance. It is constructed entirely of non-reactive materials and fits within the flow cell of the modified HAFM. In Fig. 1 we show the actual bending jig, which has undergone extensive testing and evaluation.

Our testing shows that the jig is capable of symmetrically bending the test material (1 mm thick soda-lime float glass) through a radius of curvature ranging from 0.2 to 8 m. The radius of curvature used to calculate the stress intensity factor is determined using a Vertical Scanning Interferometer (VSI).

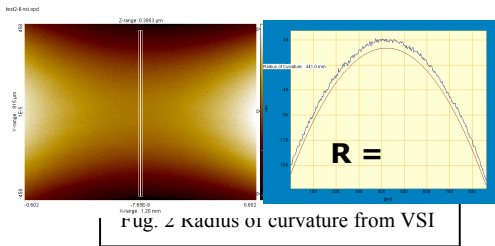
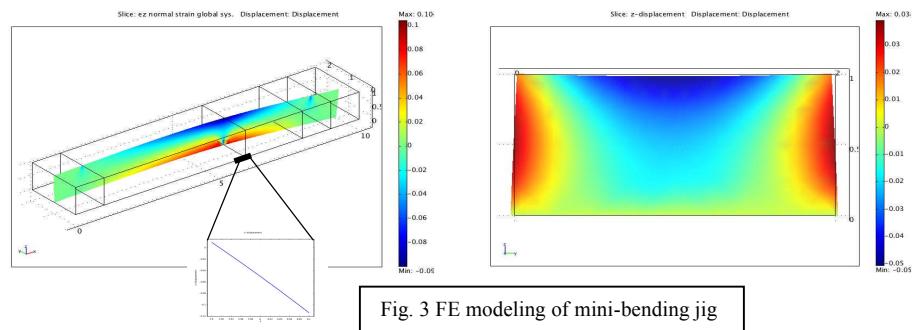


Fig. 2 shows the curvature measured in the mini-jig and Fig. 3 shows the results of finite element analysis confirming the symmetrical nature of the bend. These analyses were performed using the Comsol Multiphysics 3D Finite Element package.

In order to accommodate the mini-bending jig, and to improve the performance of the HAFM, we have made extensive modifications to it. We redesigned the HAFM to use a longer piezo tube that extends the x-y scan range from 30 to 120 μ and the z-range from 1.2 to 2.3 μ. We needed to extend the HAFM body to accommodate the new, longer piezo tube.



We devised a new Kalrez membrane shape and used a new composition material for the membrane that has a higher T limit, lower durometer (it's softer), and yet has a lower gas permeability. The new shape uses a pre-formed dimple that lowers stiffness and drag on the piezo. We redesigned the HAFM to use the new style

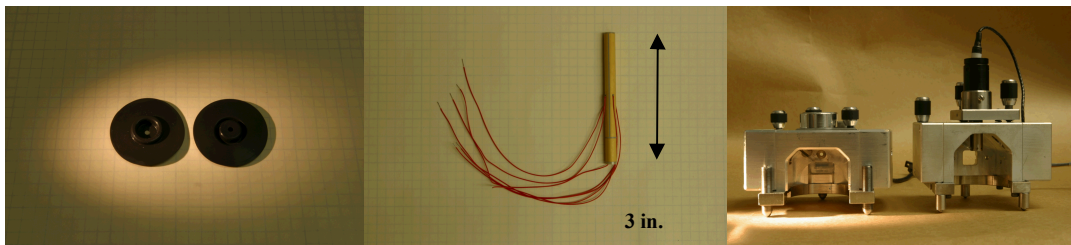


Fig. 4 New membrane, new piezo and new optical head for HAFM

Top View© optical head, that provides the ability to make lateral force measurements (frictional force microscopy) and also allows realtime movie making using the optical CCD camera. The base plate for the optical head was redesigned to use a kinematic mount consisting of 80-pitch screws, allowing fine positioning/focusing of the laser on the tip. The key elements of these improvements are shown in Fig. 4. In Fig. 5 we show the modified flow cell cover that permits insertion of the mini-bending jig within the HAFM, as well as the completely assembled HAFM.

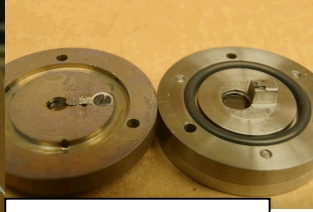


Fig5. New flow cell cover and assembled HAFM

After calibrating the new HAFM, we made our first real SCG measurements and realtime movies of the process in the HAFM under controlled T.P, and fluid chemistry conditions, including switching fluid composition during an experiment. In this experiment we initiated a small (20 $\mu$ ) crack using a Vickers indenter,

stressed the sample and then imaged the crack in a pH 5.5 (equilibrated with the atmosphere) aqueous fluid for several hours. We then switched the incoming fluid to a pH 9 dilute buffer solution and watched the crack grow to over 120  $\mu$  length. Fig. 6 shows the crack having grown to the right edge of the field of view, radiating directly away from the right-most corner of the pyramidal indentation. The starting crack length was only 20 microns, while the total scan field of view is 120  $\mu$ . A surficial polishing scratch is also visible, running roughly top to bottom, but the surface scratch has had no effect on growth of the crack.

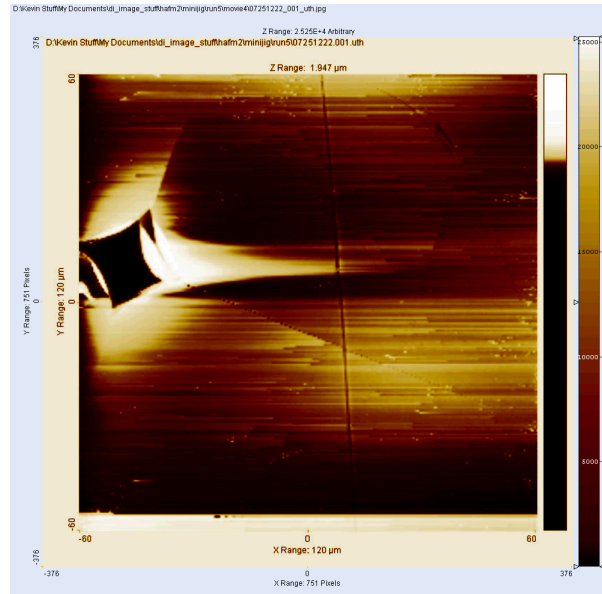


Fig. 6 Frame from SCG movie

### Exit Plan

We did not receive the requested 3<sup>rd</sup> year of funding, so our exit plan was not able to be implemented.

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### **Auspices Statement**

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