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# Development of SQUID Microscope for Localization and Imaging of Material Defects (NDE)

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**Abstract.** Dramatic progress was made in FY1997, the first full year of implementing a new technique for detecting and imaging material defects in nuclear weapon components. Design, fabrication, and initial tests of a "SQUID Microscope" has been completed utilizing the extraordinary sensitivity of High-Critical-Temperature (HTC) Superconducting QUantum Interference Device (SQUID) technology. SQUIDs, the most sensitive magnetic field detectors known, are used to sense magnetic anomalies caused by the perturbation of an induction field by defects in the material under examination. Time variation of the amplitude ( $A$ ) and angle ( $\theta$ ) of an induction field with unique spatial distribution allows examination of material defects as a function of depth and orientation within the sample. Variation of the frequency of amplitude variation,  $\omega(A)$ , enables depth selection in a given sample. Scanning the sample in physical,  $A$ , and  $\theta$  space enables detection and localization of defects to high precision. A few examples of the material defects anticipated for study include cracks, stress fractures, corrosion, separation between layers, and material inclusions. Design and fabrication of a prototype SQUID Microscope has been completed during FY97. Extensive testing of the physical, thermal, precision mechanical, and vacuum performance of the SQUID microscope were performed. First preliminary tests of the integrated system have been performed and initial results were obtained in the first week of September 1997, more than 3 months ahead of schedule.

## Objectives

Develop a superconducting quantum interference device (SQUID) microscope to detect, localize, and image material defects in stockpile components (e.g. cracks, weld defects, corrosion, corrosion pits, stress fractures, layer separations, etc.) by mapping extremely weak magnetic field anomalies associated with these defects.

[SQUID BACKGROUND:] The extraordinary sensitivity of SQUIDs enables their use to sense a large variety of electromagnetic field effects. SQUIDs have shown their value in many applications. A very few examples where SQUIDs have made and are making revolutionary contributions are: 1) imaging of human brain function in study and diagnosis of brain disorders; 2) peripheral and medial nerve studies; 3) detection and localization of buried structures, gas pipes, electrical lines, etc.; and 4) inspection of storage drums to look for active corrosion.

## Milestones

As a consequence of hiring a very talented and energetic postdoc (M. Espy) and recent commercial availability of HTC SQUIDs, we have been able focus more attention on the task end-goal of a HTC SQUID microscope. The decision was made to skip nearer-term proof-of-principle milestones using LTC SQUID technology and build a SQUID Microscope prototype. The risk associated with this decision, as we show below, has clearly paid off by attaining FY98 milestones 3 to 6 months ahead of anticipated schedule.

**FY97 Milestones:** 1) Simulated measurements of material defects using LTC SQUID system - *Skipped*. 2) Construct HTC SQUID sensor system to measure laboratory induced material defects - **COMPLETED**. 3) Develop linear, broad band flux-lock loop and amplifier design (Kansas City) - **COMPLETED, Patent application submitted**. 4) Scope requirements for 3-D sample scanning robotics (Savannah River) - *Begun FY97*

**FY-98 Milestones:** 1) Design & Build SQUID microscope Test SQUID microscope on laboratory induced material defects to demonstrate localization and imaging of material defects - **Prototype Completed in FY97**. 2) Construct 2-D motion stage & Data acquisition software for system - **Prototype Completed in FY97**. 3) Measurement of material defects in system with geometry specific to ESP program requirements - *Begun in FY97*.

## Deliverables

- 1) Low-resolution, LTC SQUID Sensor System - Milestone/deliverable skipped (see above)
- 2) HTC SQUID Microscope Prototype - Initial system completed, delivered, and in use.
- 3) HTC SQUID Microscope Measurements - First preliminary measurements performed, September 1997.
- 4) Broadband (DC-10MHz) Flux-lock loop & amplifier design, Patent application filed (KC Plant)

## Accomplishments

This report represents the accomplishments of the SQUID Sensor Group at Los Alamos National Laboratory and collaborators at the Kansas City Plant/Allied Signal (K. Ganther, L. Snapp) and the Savannah River Plant/Westinghouse (R. Fogle)

**Dewar Design.** The primary goals of the dewar design were to 1) keep the SQUID sensor at an operating temperature below 79K, 2) keep the sample of interest at room temperature, and 3) have the separation between the SQUID and the sample less than 1mm (SQUID-sample separation directly impacts resolution). Materials for all dewar parts were required to be non-magnetic.

These goals were achieved in the design shown in figure 1. The inner and outer dewar cylinders are fabricated from G-10 (fiberglass). The inner dewar holds the liquid nitrogen (LN) used to keep the SQUID below 79K. The SQUID is in thermal contact with the LN reservoir via a sapphire cold-finger and physically located in the vacuum space between the inner and outer dewar walls. During operation, the SQUID, on the cold finger, and the sample, at room temperature, are positioned very close to the window ( $\sim 0.001''$ ), to minimize the SQUID-sample distance. In tests requiring induced eddy-currents in the sample, an induction coil is mounted on the dewar baseplate. The induction coil is designed in a novel "double-D" configuration that produces an induction field null at which the SQUID is located, preventing the SQUID output from being dominated by the primary induction field.

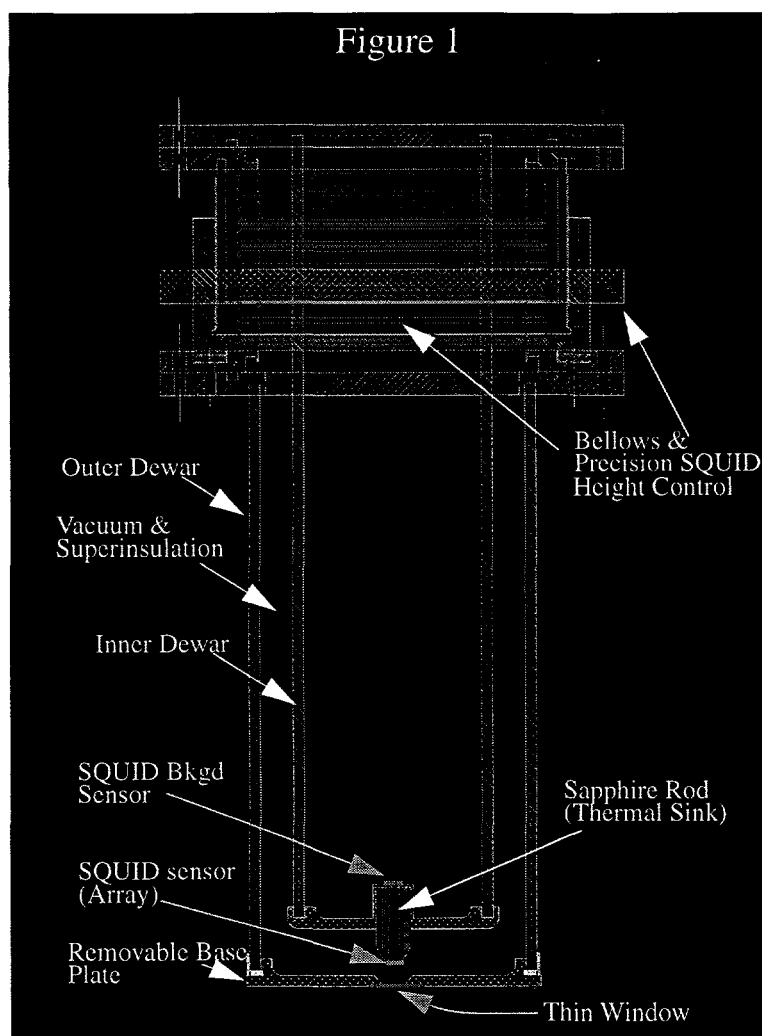
Two different thin window materials have been extensively tested, Kapton and quartz. Both materials are very strong and are poor thermal conductors. Kapton has the advantage of being less expensive and less likely to break, however because it is more flexible than quartz, it bows into the vacuum space further increasing the SQUID-sample separation. Although a Kapton window has been used successfully for the initial microscope data, presented below, the final window material will most likely be quartz.

**Present Dewar Status.** Fabrication and assembly of the first microscope dewar is complete and is being used for acquisition of preliminary data. During fabrication, we discovered diffusion leaks in the G-10 cylinders used in the dewar fabrication. A polymer paint was used to attempt to correct this problem, however the paint has subsequently been observed to outgas severely, requiring continual use of a vacuum pump to maintain the thermal isolation. Although this has not prevented acquisition of initial microscope data, it will ultimately be necessary to eliminate the electromagnetic and mechanical noise caused by the vacuum pump. New G-10 tubes, rated for vacuum use and tested at LANL, have been purchased and machined. They will be replaced within the next few weeks.

**Dewar Testing.** Using the present dewar, we have investigated how close the SQUID can approach the thin window while maintaining the desired operating temperature ( $< 79K$ ), the relative merits of different thin-window materials, and the optimum materials to thermally couple the SQUID to the cold-finger.

Our investigations have determined that a few layers of goldized mylar are an optimum balance between emissivity and conduction to allow the SQUID to approach within  $250\mu m$  of the window with no appreciable increase in temperature ( $\sim 76K$ ) compared to the "resting" position  $6.35mm$  back. The temperature remains below  $79K$  for SQUID-window separations between  $250\mu m$  and  $25\mu m$ . SQUID-window separations below  $25\mu m$  cause the temperature to rapidly rise above the desired operating temperature of  $79K$ . These observations are independent of window material and exceed our design goal of SQUID-sample separation of  $1mm$ .

The window deflection under vacuum was also studied to determine optimal window materials. The deflection, window thickness, and the SQUID-window separation are all factors that limit the minimal SQUID-sample distance, and thus the ultimate spatial resolution of the microscope. We found for our window aperture of  $1.16cm$  diameter,  $125\mu m$  thick Kapton bows in  $750\mu m$  under vacuum load, whereas  $300\mu m$  thick quartz deflects  $< 25\mu m$ . Therefore, a thicker and stronger material such as quartz ultimately reduces the SQUID-sample distance. Kapton will remain useful in tests where minimal SQUID to sample separation is not critical as it is much less expensive and fragile. The preliminary results shown here are taken with a Kapton window.



The thermally conductive adhesive used to couple the SQUID to the sapphire cold-finger was also investigated. The material must have a high thermal conductivity as well as be easily removable at room temperature yet adhesive at cryogenic temperatures. Vacuum greases have traditionally been used for this purpose. Specific vacuum grease adhesive and thickness was optimized by affixing a precision thermistor to the tip of the cold finger with various materials and configurations. The same test apparatus was used to determine the optimal amount of super-insulation to use and the temperature at various distances from the window.

**First Results of SQUID Microscope .** First data with the new SQUID microscope were acquired over the past week (September 1997), 3 to 6 months ahead of program schedule. It should be noted that neither the microscope nor the sample motion system were in optimal configuration. The motion control system for the first data was not implemented, therefore "scanning" was limited to 1-D by hand. Realistic position accuracy and reproducibility is a few millimeters. Also, a Kapton window was used, limiting the SQUID to the sample separation to ~20mm, consequently drastically lim-

iting the spatial resolution to that scale. However, we were still able to take very promising data. Figure 2 shows the results of a low-resolution scan of a 1.5mm thick aluminum plate with a 6.35mm slot cut out of it (diamonds), and a 1.5mm thick aluminum plate with no slot (squares). We also acquired data for the slotted plate hidden under a similar plate with no slot (triangles). The difference between the slotted and solid plates is striking, and there is clear evidence that the slot can be seen even with a plate covering it.

System improvements were made by better centering of the induction coil and automated motion control for precise scan spacing (though sample-to-sample reproducibility was not implemented). The SQUID-sample distance was also reduced somewhat by better sample positioning, although this is difficult to quantify in our present set-up to better than ~10mm. Figure 3 presents data taken with a 1.5mm thick aluminum plate with a 375 $\mu$ m wide crack (diamonds), as well as data from a similar plate with no crack (squares), a plate with no crack covering the plate with the crack (triangles), and two plates with no cracks (circles). These first results are extremely encouraging as there was no difficulty in seeing the induced material defects. Preliminary examination of a stress fracture in a Ta-W hemicylinder indicates even smaller feature sizes can also be observed.

**Kansas City Plant Collaboration:** Amplification and control electronics currently available for SQUID sensors limit the potential near-surface depth resolution due to the limited frequency of the feedback loop (flux-lock loop). The limited frequency response of the flux-lock loop also determines the lower

Figure 2

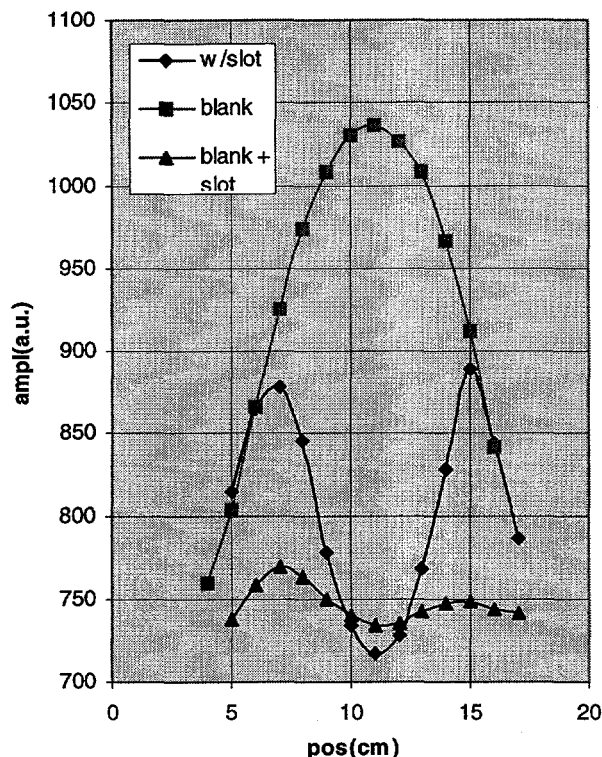
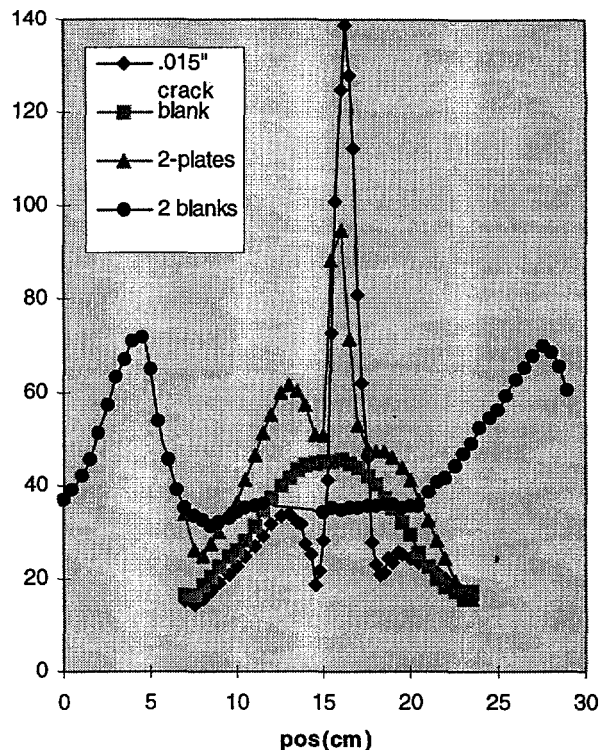


Figure 3



limit one can attain for system electronic delay. This delay inherently limits the ability to cancel unwanted background signals such as the signal resulting from the primary induction field. As a consequence of these limitations, Allied Signal engineers K. Ganther and L. Snapp have designed a novel linear amplifier and matched flux-lock loop circuit for control of SQUID sensors with at least 10MHz feedback loop frequency. The design has been modeled with realistic SQUID parameters using SPICE and similar simulations codes and results are extremely promising. Fabrication of an initial test circuit has begun and completion is anticipated in early calendar 1998. The design generated sufficient interest to prompt the filing of a patent application for high-frequency feedback and amplification circuit to control SQUIDS and other sensors that operate in a null-detector mode. Implementation of this design would substantially improve the SQUID microscope resolution as well as potentially improve performance requiring strong induction field strength, such as for evaluation of material defects in low-conductivity samples.

**Savannah River Collaboration.** Future improvements to the SQUID microscope include the implementation of a fully automated 2-D nonmagnetic scanning stage. Two primary sources of magnetic noise from such a system are stepper motor noise and that associated with magnetic materials in the x-y stage itself. The magnetic noise of the stepper motors is inevitable by the nature of the device and can be minimized by distancing the motors from the stage. Also special "low EMI" indexers have been purchased commercially and tested successfully with the SQUIDS. Collaborators at Savannah River Plant assisted in locating a source for special nonmagnetic stages and components. A nonmagnetic system was custom built by a commercial vendor and used for the 1-D scan presented in Figure 3. During FY97 collaborators from the Savannah River plant also evaluated program needs for 3-D motion of non-planar samples under a SQUID microscope. It is anticipated that the design of a precision 3-D motion system will commence in FY98.