Fermi National Accelerator Laboratory

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Adhesive Tests for Use on 3-D Silicon Vertex Detectors

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Introduction:

An adhesive must be chosen for the production of modules for a three dimensional Silicon Vertex Detector[1] with both disks and barrels. The adhesive will be used to assemble the silicon wafers into a module.

1. The Chosen Adhesives

1.1 The Requirements

The requirements of the chosen adhesives are that the adhesive must not be a conductor, must not destroy the silicon, must be a physically tough adhesive, must be quick polymerizing, and be radiation hard to a dose of $0.4 MGy/r^2$ [2].

1.2 The Chosen Few

The selected adhesives were based on the above requirements and chosen from three different families of adhesives:

Locktite 324: a urethane methacrylate ester.
NOA 81: a UV curing prepolymer acrylic.
Masterbond UV15-7: a UV curing epoxy.

All of these adhesives are dissolvable in a chlorine based solvent. The solvents used were trichloroethylene and methylene chloride.

2. The Construction of a Joint for Mechanical Tests

2.1 The Silicon

The samples were made from 0.025" thick polished silicon cut in 0.375" by 0.625" rectangular shapes.

2.2 The Need for Pyrex Glass Supports

Without some sort of additional support the joints were very weak and easily destroyed in handling. Pyrex prisms were made to fit in the corner of the joint to increase strength. A material like Pyrex is needed because it has a coefficient of thermal expansion similar to that of silicon.

2.3 Preparation of the Joint

The silicon was cleaned with an alcohol and a chlorine based cleaner such as trichloroethylene or methylene chloride to remove any grease and remove any adhesive that was left from a previous sample.

2.4 Construction of the Joint

After cleaning the silicon, one end of one piece was held perpendicular to the middle of the other piece in a "T" like configuration. For the adhesives using a primer, the glass prism was lightly coated with the activating primer, dipped in the adhesive and quickly applied to the joint area within approximately 2 minutes. For the UV curing adhesives, the prism was dipped in the adhesive, then applied to the joint and received a 50 second dose from a UV lamp (Ultra Cure 100). The samples then were left to sit overnight to fully polymerize before testing.

3. Measuring the Movement of the Joint

3.1 Equipment Used

The apparatus used to measure the movement of the joint can be seen in Figure 1.

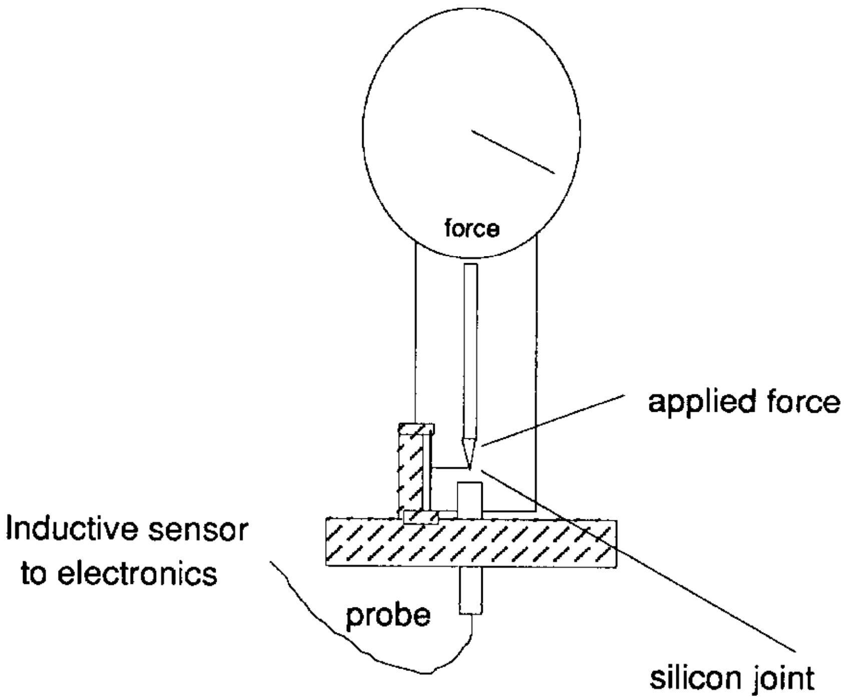


Figure 1

The joint was firmly clamped in place in the fixture. An inductive sensor was then placed through a hole in the bottom of the fixture directly below the silicon. A piece of metal approximately 1" square by 0.032" thick was then placed on the top of the silicon. A metal is needed because an inductive type sensor must have a target that is conductive. The anvil was then lowered onto the metal that was resting on the topside of the silicon joint. The weight applied to the joint for creep tests was 2 ounces. The sensor then read out a voltage that is a linear function of the average distance from the sensor to the conducting surface. This allowed us to monitor movement of the joint while various forces were applied over long periods of time.

3.2 Effects of Temperature on the Equipment

Temperature played a large role in the readings of the proximity sensor. Over the period of a day, the temperature in the room often fluctuated 20°F. The proximity sensor is placed through a hole drilled in the bottom of the fixture and clamped. Because of this configuration, the distance changes from the sensor to the metal plate on the silicon were partially due to the linear expansion of the apparatus and electronic drift.

4. Stress Strain of the Silicon

The silicon itself bends due to the forces applied to it. This can be seen from the equation :

$$D = wL^3/3EI$$

Where

w is the force applied to the joint (force used = 0.125 pounds).

Length is the approximate distance from the joint to the location of the applied force (0.4375").

E is the elasticity of the silicon ($E = 30x10^6$ psi).

I is (Width * Thickness^3)/12 where

Width is the width of the silicon piece used(0.375").

Thickness is the thickness of the silicon used (0.025").

The calculated deflection, D, is 0.000238" for 0.025" thick silicon.

5. Stress Strain of the Silicon Joint

The deflection of the joint was measured for each of the samples with different forces applied to the joint. If this is done quickly the effects of thermal expansion should not be noticed. A graph of deflection (in mils) vs. force applied (in ounces) is shown in Figure 2.

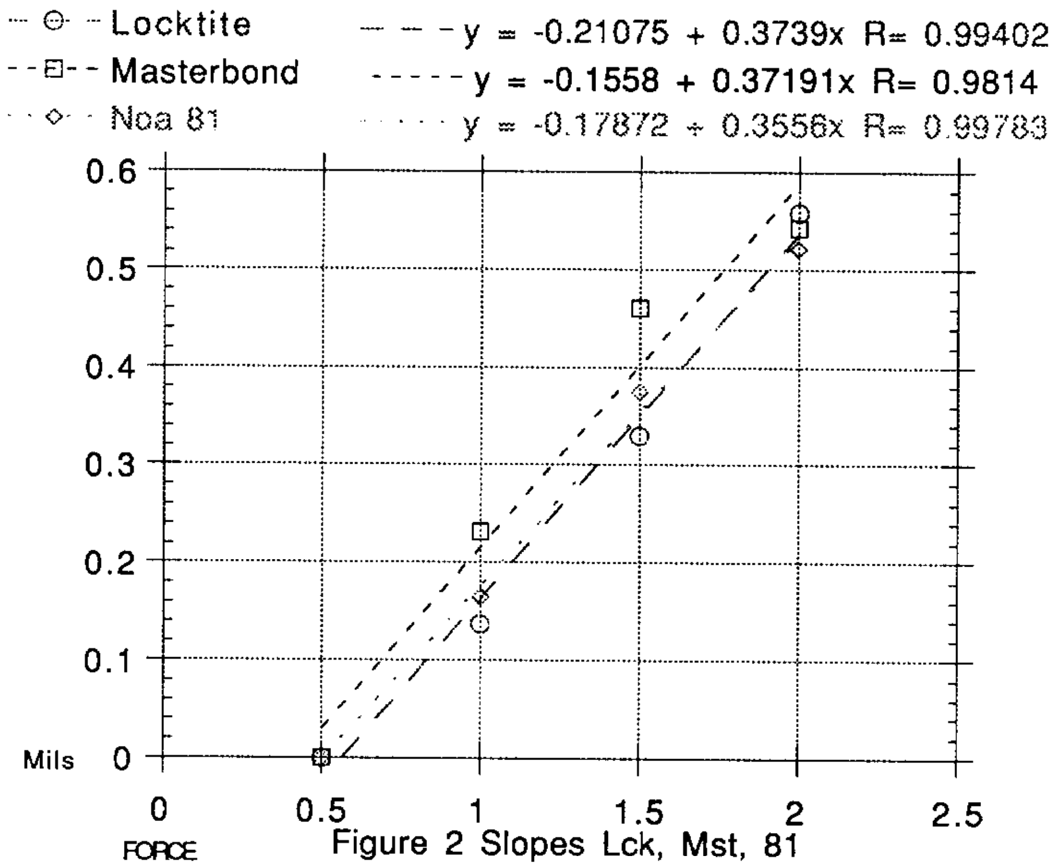


Figure 2 Slopes Lck, Mst, 81

6. Effects of Temperature on the Joints

6.1 Equipment Used and Procedure

Force was applied to the joints in both an oven and freezer. A bar of known mass was applied as a force on the joint. The bar then was set on the joint at 45° so that the bar set in a cradle made by the silicon joint as shown in Figure 3.

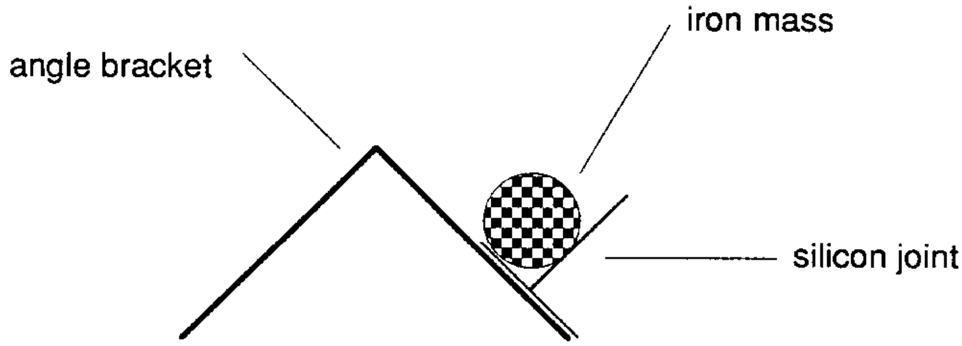


Figure 3

The sample was then set in a temperature isolated environment until the joint had failed or had survived the time limit set.

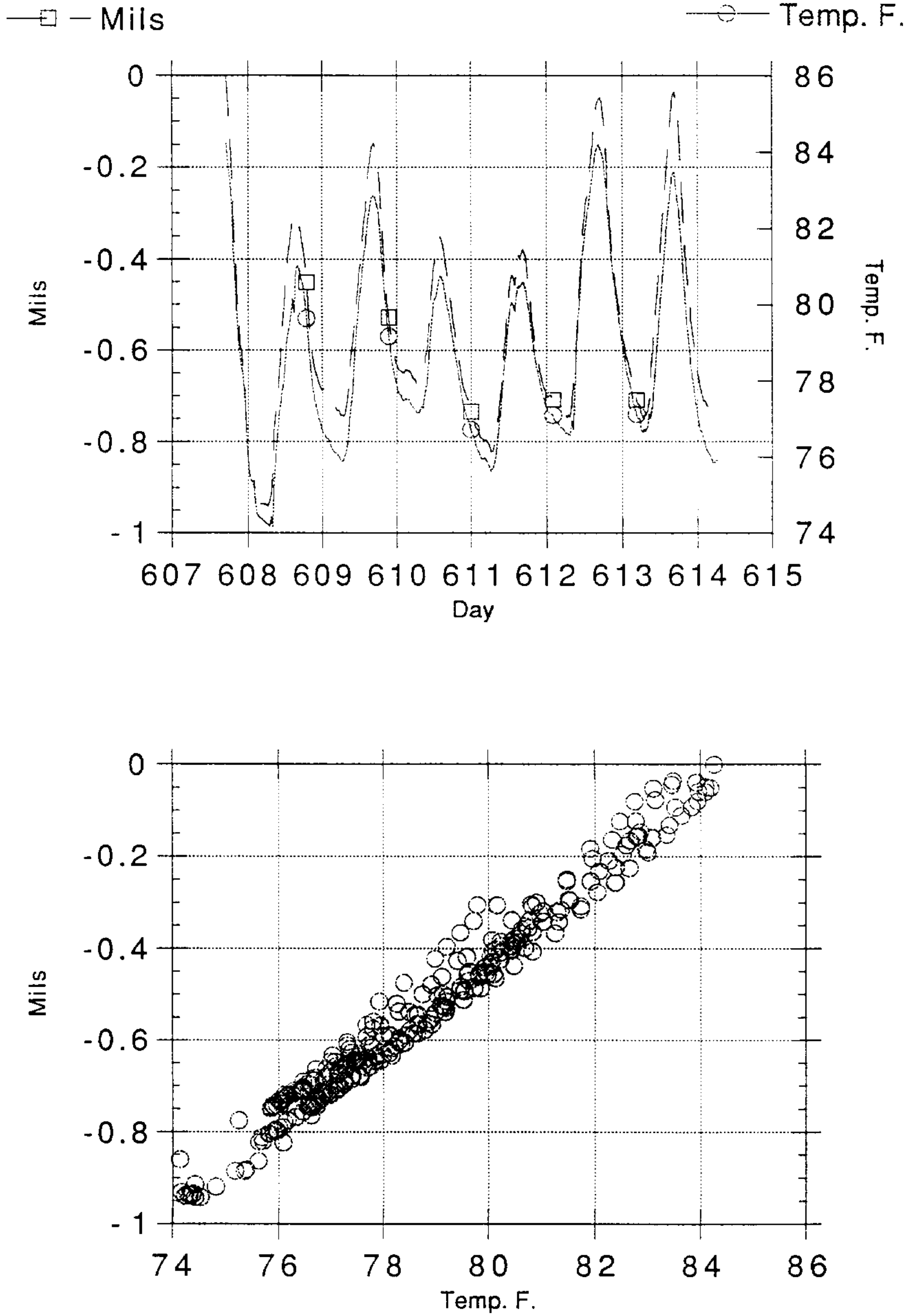
6.2 Results of Temperature Tests

Samples were made and placed in an oven using the apparatus in Figure 3. Samples of each kind of adhesive were constructed. The first set of samples were placed in the oven at 160°F. for 3 hours with an iron mass of 65.0 grams placed on the joint. All of the samples survived. The samples were then placed back in the oven with an 85.0 gram iron mass placed on them. After 30 minutes, the only one that did not break was the Locktite sample. This sample remained in the oven for an additional 24 hours at 200°F. and did not break. The second set of samples was placed in a freezer with an 85.0 gram mass; all samples survived a 24 hour test.

7. Creep Tests

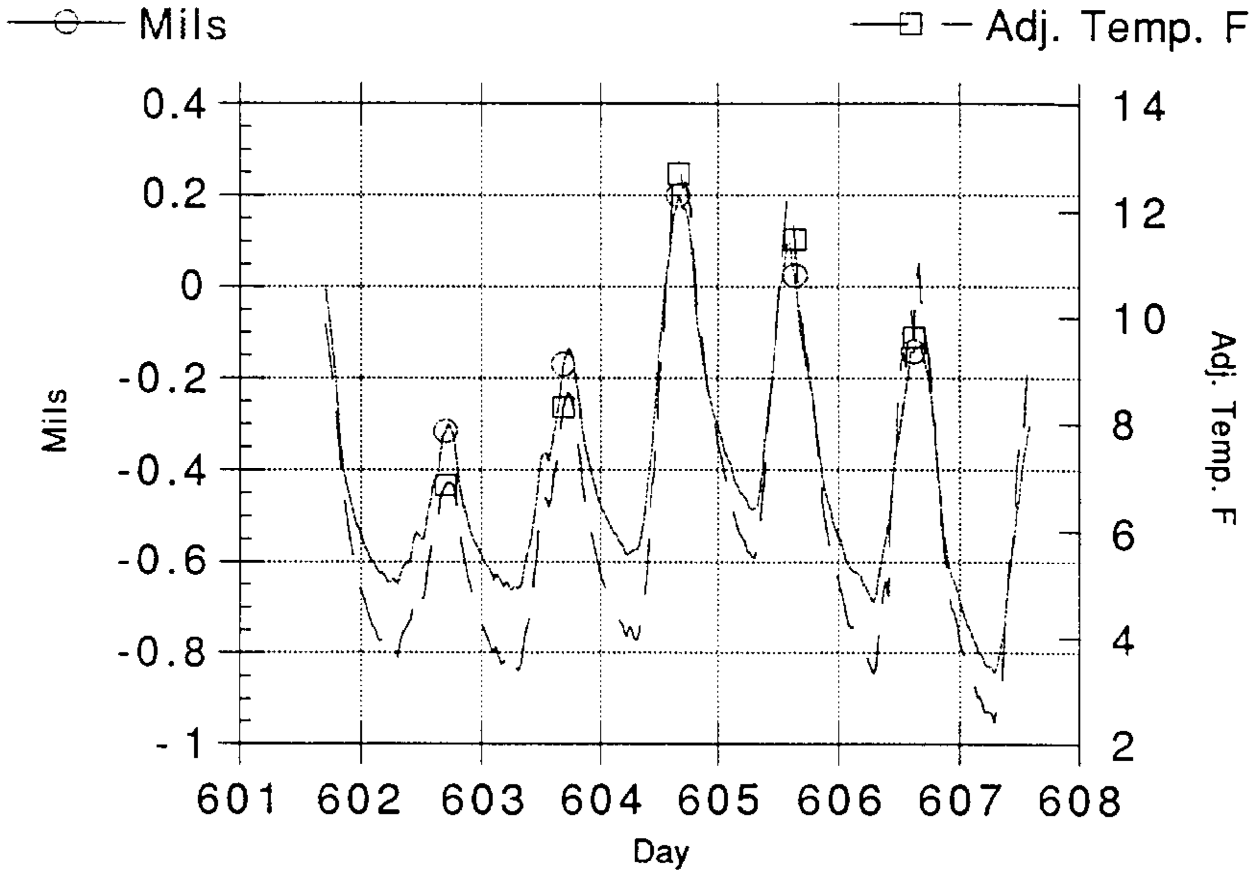
The samples were placed in the apparatus as shown in Figure 1. The readout system[3] used inductive sensors and had the capability to monitor the joint movement for several days. Results are seen in Figures 4 through 6. Since the movement follows the temperature changes, the movement does not clearly show joint creep. Also by comparing the plots to each other, one can see that each joint moved approximately 0.8 mils for a 10° change in temperature. This can be attributed to the thermal expansion of the apparatus and the dependence of temperature on the electronics.

Fig. 4 NOA81



Mils
Temp. F.
0
-0.2
-0.4
-0.6
-0.8
- 1
86
84
82
80
78
76
74
607 608 609 610 611 612 613 614 615
Day
Mils
Temp. F.
0
-0.2
-0.4
-0.6
-0.8
- 1
74 76 78 80 82 84 86
Temp. F.

Fig. 5 Masterbond



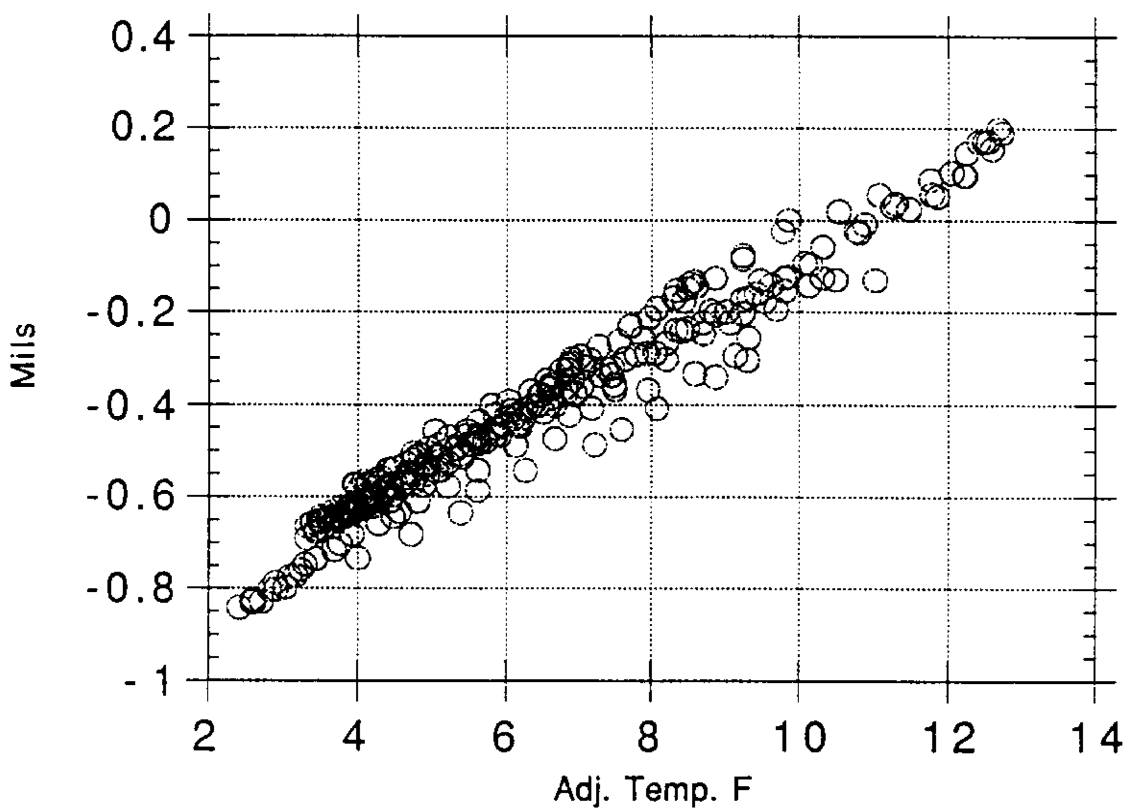
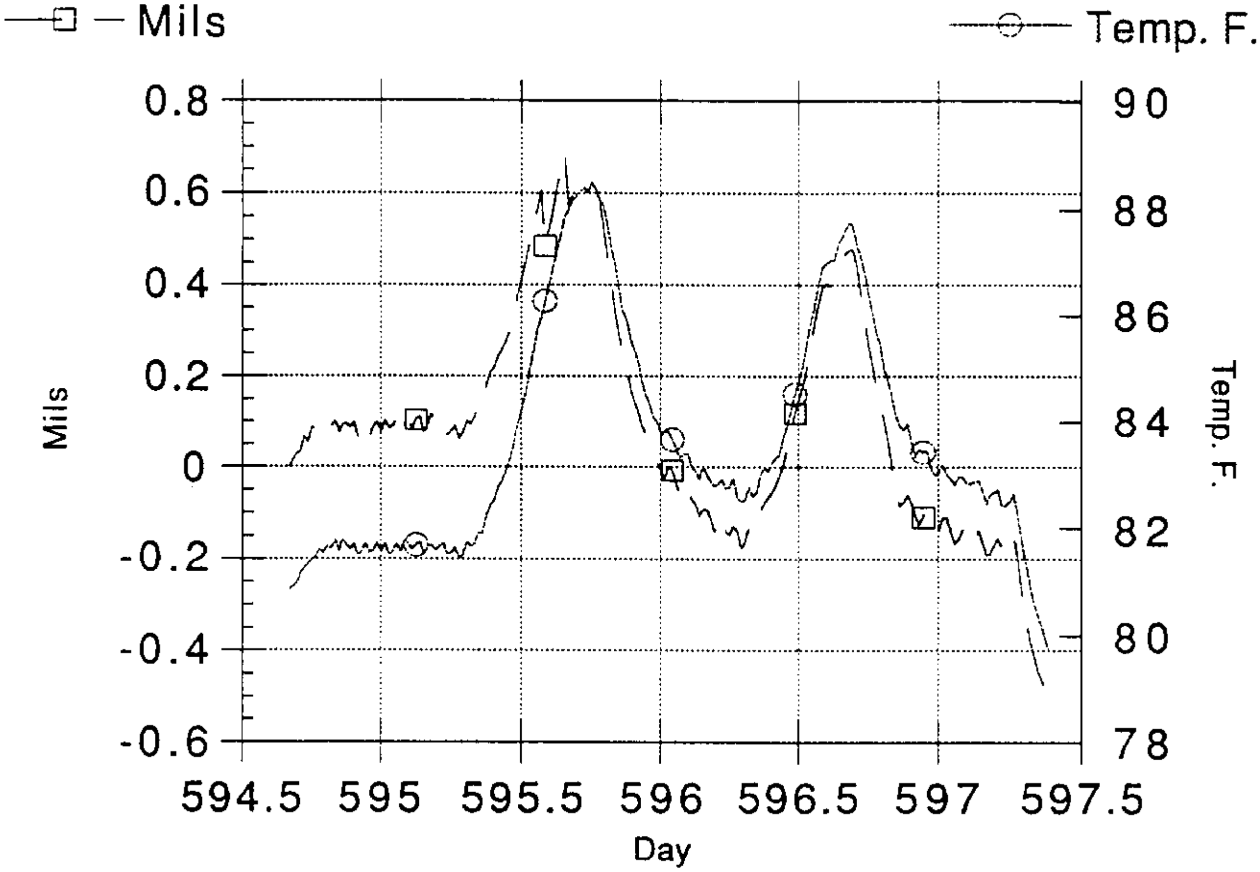
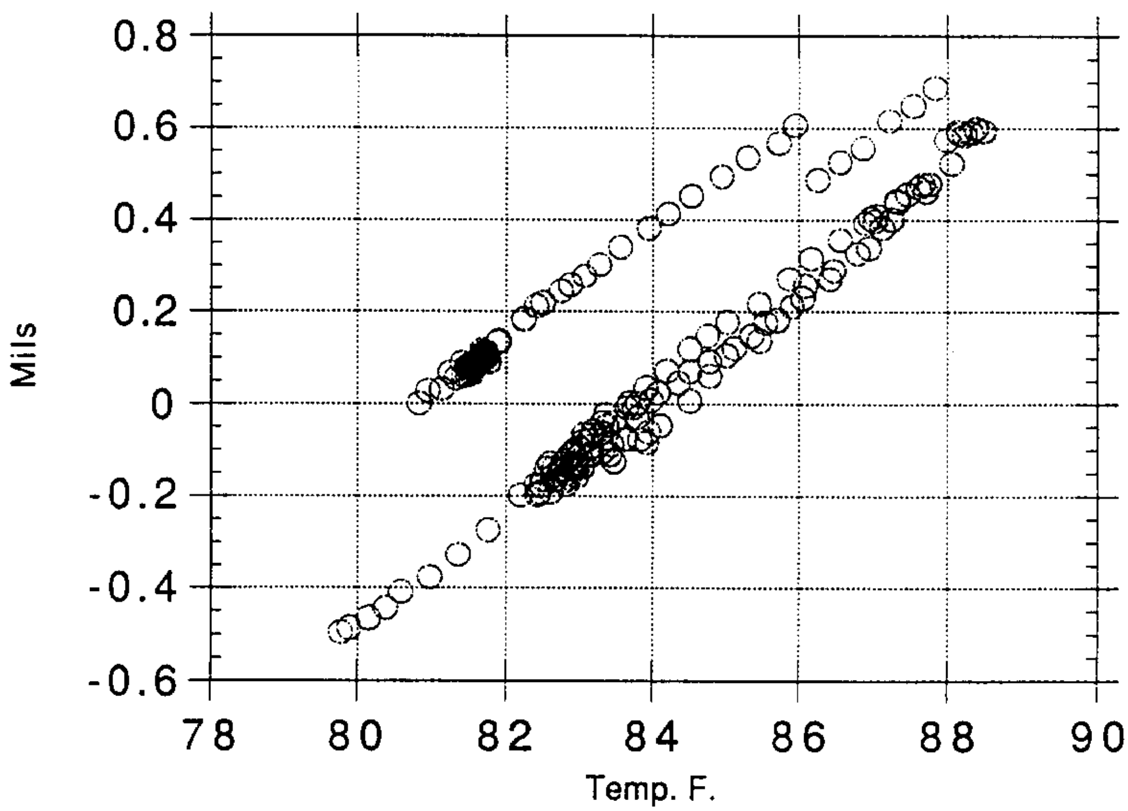


Fig. 6 Locktite





8. Radiation Effects on the Joint

Six samples of silicon joints, two of each adhesive, were sent to Argonne National Laboratory and were put in a beam of neutrons of flux $10^{14}/cm^2$. The placement of the test joints are seen in Figure 7.

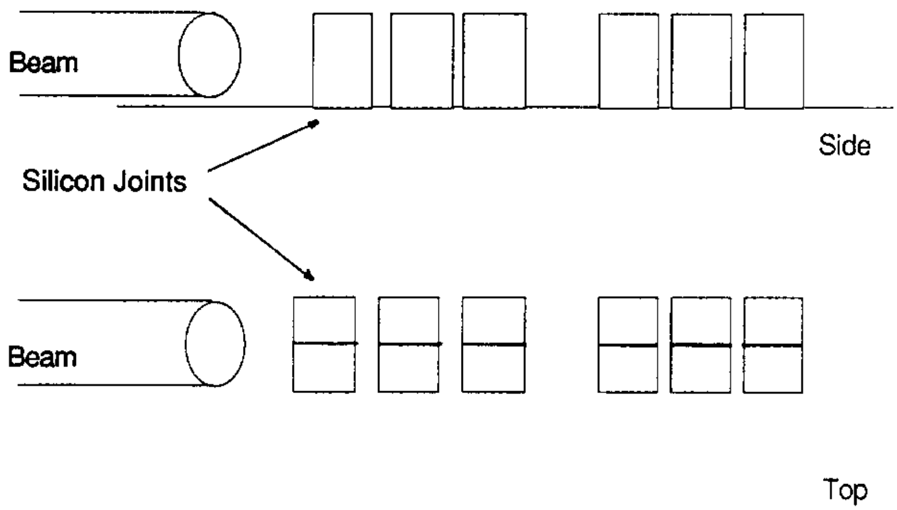
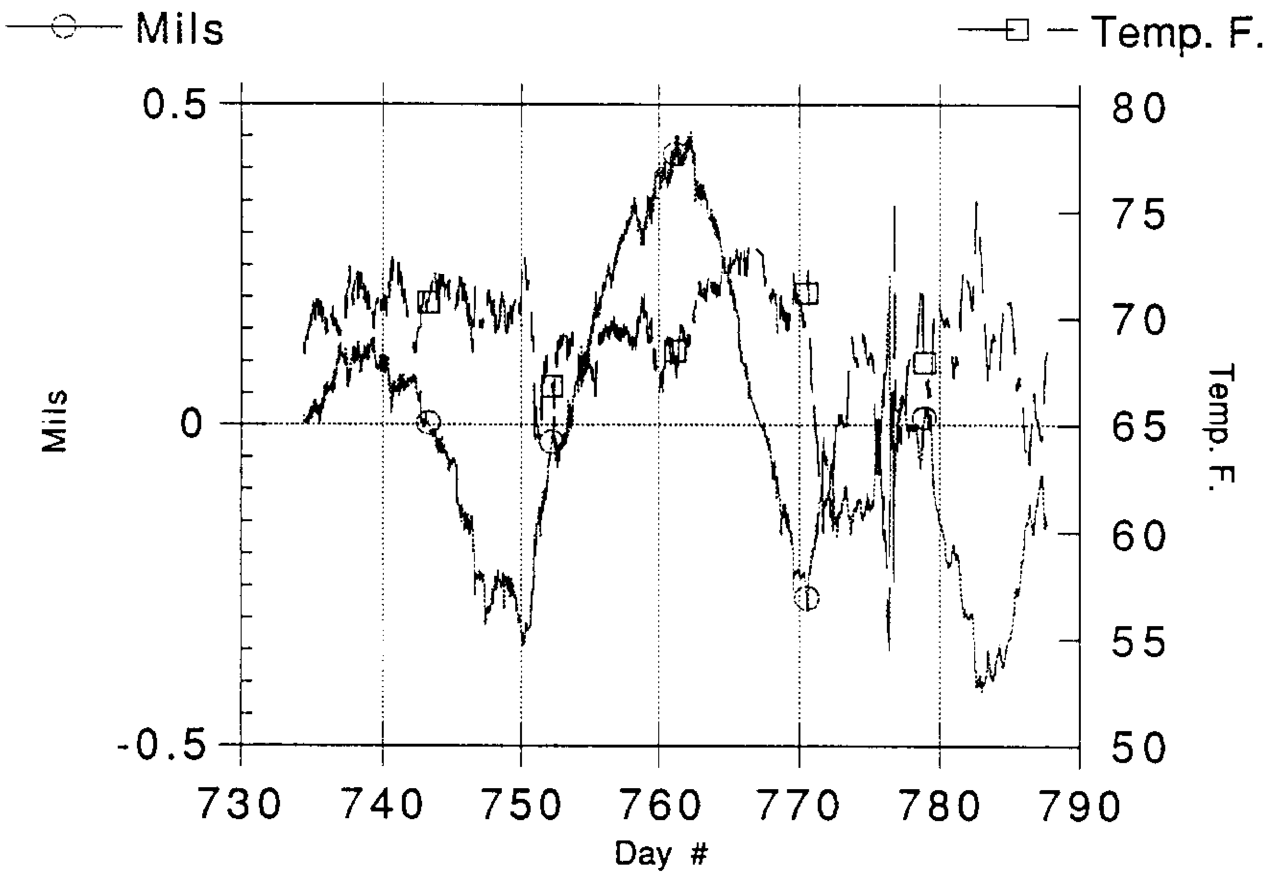


Figure 7

The amount of radiation a sample received is proportional to the distance to the end of the beamline. None of the samples showed yellowing or other visible signs of radiation damage. The samples were then tested for creep with the results seen in Figures 8 through 10. The NOA81 sample shows a movement of 0.8 mils for about a 20° change in temperature. From the data, the Masterbond joint exhibits more creep than the other samples. The adhesive bond was later found to be broken in the apparatus. The Locktite sample shows a movement of 0.5 mils for a 10° change in temperature. These results are reasonably consistent with the earlier data before the samples received radiation. Creep or damage caused by radiation was not clearly seen in the NOA81 and Locktite samples. The movement can be attributed to the dependence of temperature on the fixtures and electronic drift.

Fig. 8 ArgNOA81-1



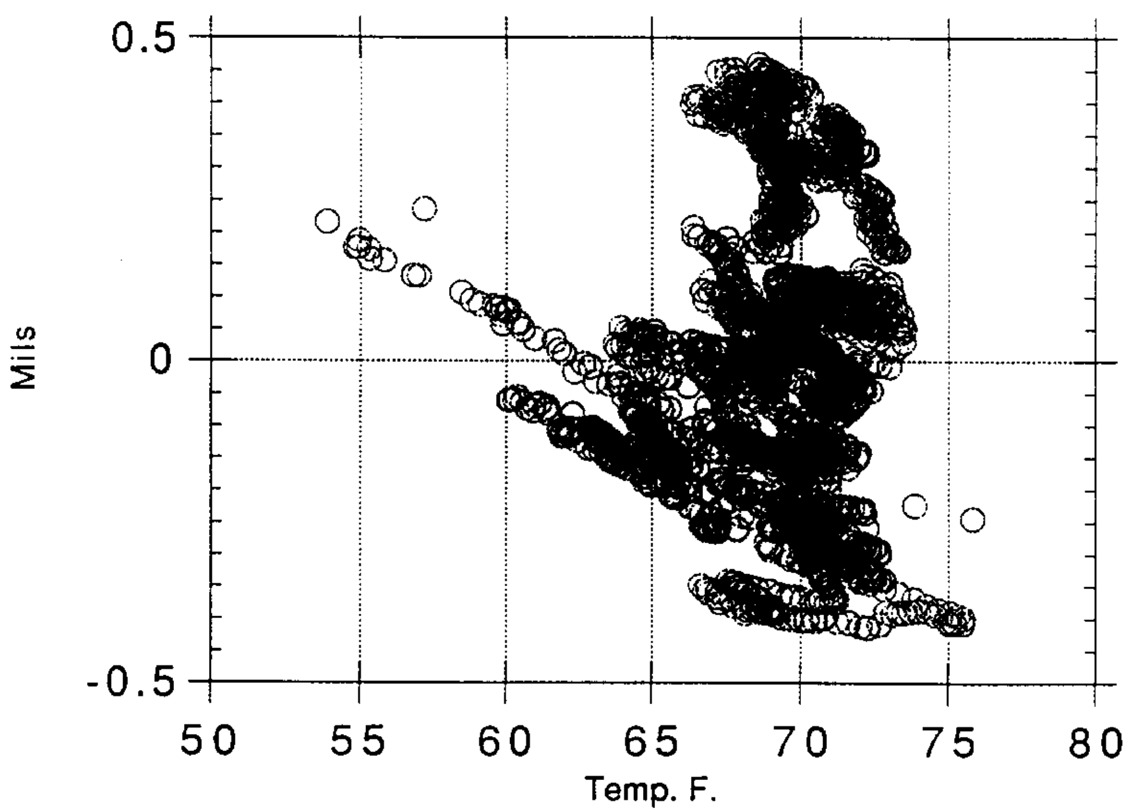
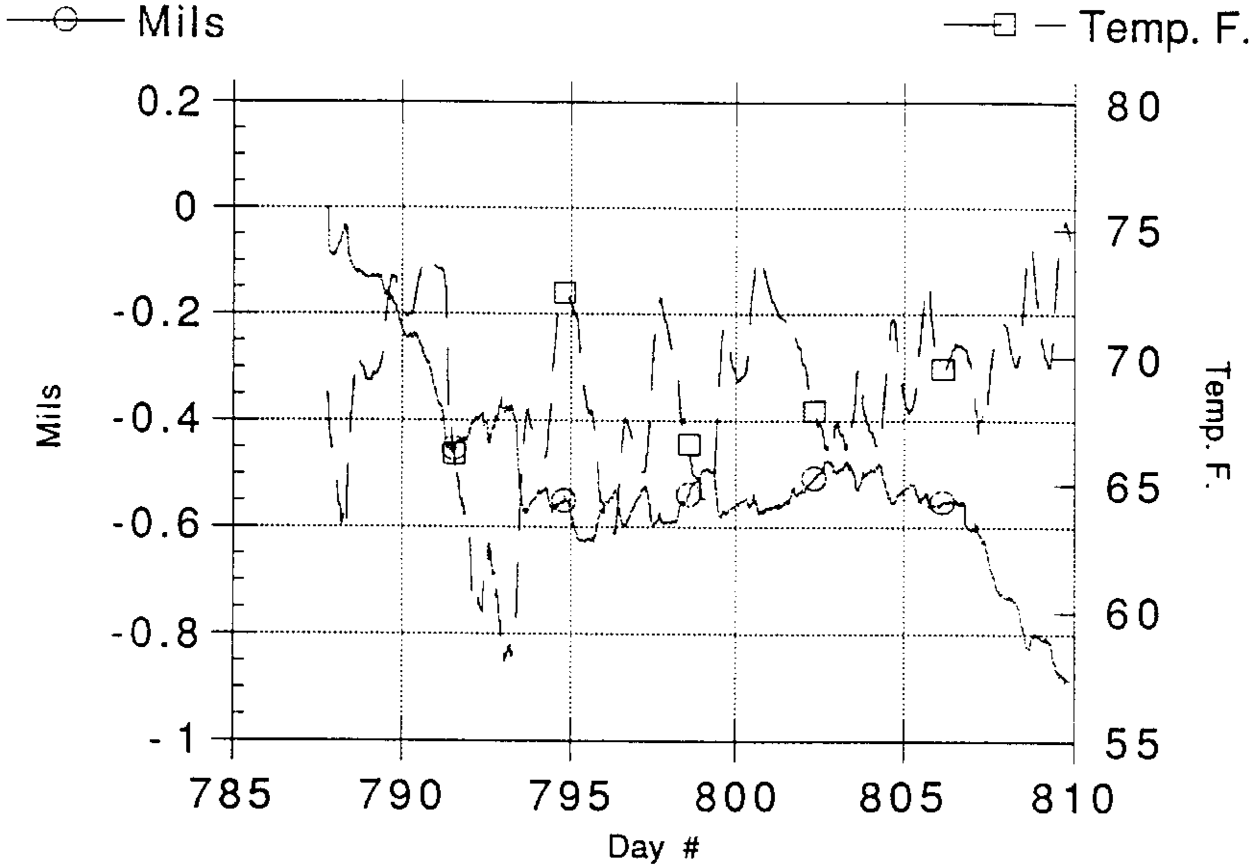


Fig. 9 ArgMasterbond-1



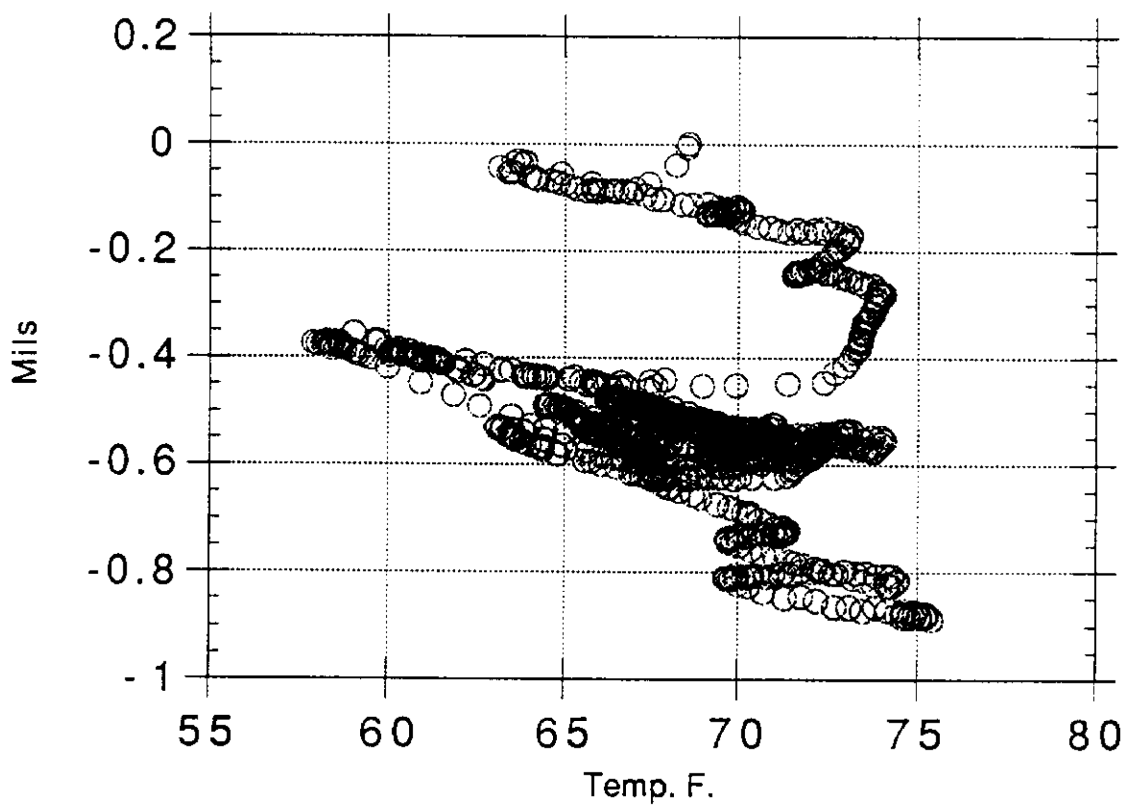
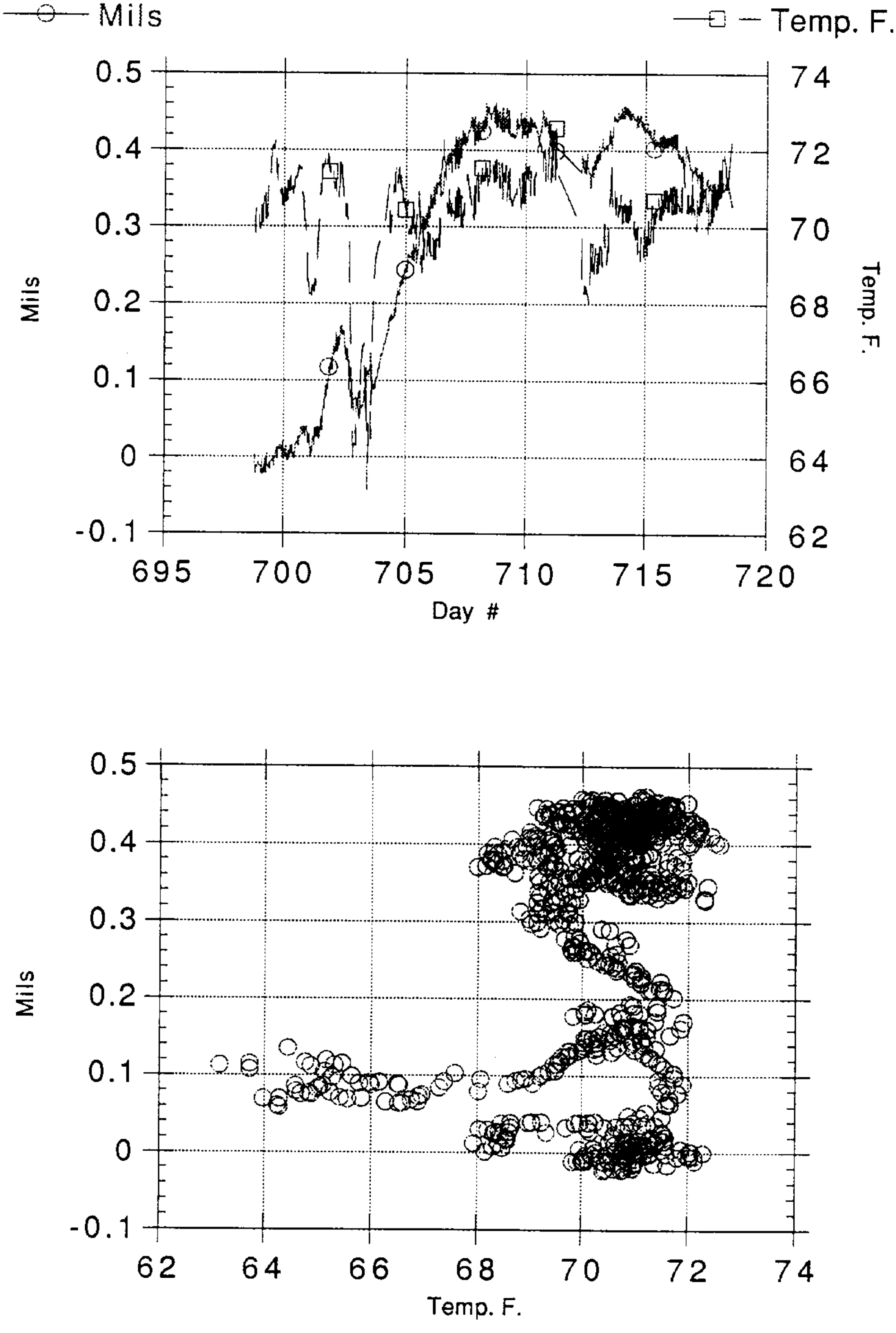


Fig. 10 ArgLocktite-1



9. Glue Tests on a Micron Detector

Locktite 324 was used in glue tests at Oklahoma University on a DC-coupled multistrip Micron detector. Using Locktite, a piece of 0.5 inch Pyrex glass tubing and an SVX chip (bare silicon) were glued to the diode side of the detector. During curing and cooling to -22° C, the detector was monitored by observing the analog signal displayed on an oscilloscope. Minor discrepancies were observed in the first few hours of cooldown and curing. After a full cure and cooldown, the operation of the detector returned to normal. The glues were then dissolved and the pieces removed using methylene chloride. The detector was placed in a bath for 12 hours while the pieces were being removed. The detector's operation after this was normal. Other pieces of silicon and glass were glued and unglued to the ohmic side of the detector using Fermilab's facilities. The ohmic side of the detector used was inoperative, so inspection was done under a microscope. No damage was visible.

10. Conclusions

For a UV-curing adhesive, NOA-81 is suggested because of its long term strength. However, if it is impossible to use a UV-curing adhesive because of the difficulties in getting the source close to the joint during construction of a module, then it is possible to use Locktite 324 which is a primer activating adhesive. The Locktite 324 also seemed to bond better and more reliably with more adhesion to the silicon than any of the other adhesives.

11. Acknowledgements

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12. References

[1] H. Castro *et al., Letter of Intent for the BCD, A Bottom Collider Detector at the Fermilab Tevatron,* (October 7, 1988).

[2] D. E. Groom, *Radiation Levels in SSC Detectors,* (1989).

[3] H. Mulderink, N. Michaels, and Hans Jostlein, *Mechanical and Thermal Behavior of a Prototype Support Structure for a Large Silicon Vertex Detector (BCD)*, Fermilab TM-1616 (August 23, 1990).