Large Diameter Lithium Compensated Silicon Detectors for the NASA Advanced Composition Explorer (ACE) Mission


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LARGE DIAMETER LITHIUM COMPENSATED SILICON DETECTORS FOR THE
NASA ADVANCED COMPOSITION EXPLORER (ACE) MISSION

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ABSTRACT

Fabrication of the 100 mm diameter, 3 mm thick lithium-compensated silicon, Si(Li), detectors for the Cosmic Ray Isotope Spectrometer (CRIS) instrument on board the ACE satellite required development of: new float-zone silicon growing techniques, new Si(Li) fabrication procedures, and new particle beam testing sequences.

These developments are discussed and results are presented that illustrate the advances made in realizing these CRIS Si(Li) detectors, which, when operational in the CRIS detector telescopes, will usher in a new generation of cosmic-ray isotope spectrometers.

I. INTRODUCTION

The Advanced Composition Explorer [1] is a NASA space science mission currently being developed to study the elemental, isotopic, and ionic charge state composition of energetic heavy nuclei accelerated on the Sun, in the heliosphere, and in the Galaxy. The ACE spacecraft, scheduled for an August 1997 launch into a halo orbit about the L1 libration point in the Earth-Sun system, will carry instrumentation designed to measure particles over six decades in energy, ranging from the solar wind at \(-1\) keV/nucleon to galactic cosmic rays near \(1\) GeV/nucleon.

Measurements at the high end of this energy range are made by the Cosmic Ray Isotope Spectrometer (CRIS). This instrument, shown schematically in Fig. 1, employs four stacks of fifteen, 3 mm thick, 100 mm diameter lithium-drifted silicon detectors, Si(Li), to measure the energy loss rate and total energy of stopping heavy nuclei. By combining these data with the particle trajectories obtained from a scintillating optical fiber trajectory (SOFT) detector, which is penetrated by the particles as they enter the instrument, the atomic number and the mass of each detected nucleus can be determined.

The CRIS instrument is designed to study all of the stable and long-lived nuclei up through \(Z = 30\) in the periodic table.

In order to determine the abundances of the rare nuclides in this range the instrument requires both a large geometrical acceptance to achieve an adequate statistical sample and excellent mass resolution to separate rare species from abundant isotopes of the same element. As designed, CRIS achieves a geometrical factor of about \(300\) cm\(^2\) ster. This geometrical factor will, for example, allow CRIS to collect more than \(10^5\) Fe nuclei in its first two years of operation, enough to permit a precise determination of the abundance of even the rarest, stable iron isotope, \(^{56}\)Fe, which is expected to make up only about 0.3% of the total Fe flux.

The CRIS mass resolution objective, \(\sigma_m < 0.25\) amu, is sufficient for measuring adjacent isotopes with relative abundances as large as \(-300:1\). This mass resolution objective imposes stringent requirements on the mapping of detector thicknesses and dead layers, as discussed in Section IV below.
This report focuses on the realization of the Si(Li) detectors for the CRIS instrument.

II. SILICON FOR THE CRIS SI(LI) DETECTOR FABRICATION

Successful fabrication of Si(Li) detectors is critically dependent on the perfection of the silicon crystals employed. While aspects of the CRIS detector fabrication will be discussed further in Section III, we will briefly review here the lithium-ion compensation process, with focus on the crystal quality requirements.

\[ V \cdot \nabla \nabla I = J \]

where \( J \) is the lithium-ion flux and \( V \) is the applied voltage. The particle range in the Si(Li) process is approximately "0.05 μm.

Fig. 2. Approximate lithium-ion distribution after the initial diffusion (1) and after some lithium ions have been drifted into the bulk silicon (2).

Figure 2 illustrates the basic assumptions involved in this compensation process.[2] Si(Li) detector fabrication begins by diffusing lithium into a p-type silicon wafer to form an n+p junction with the resulting lithium profile approximated by the dotted line in the figure. By heating the wafer to greater than 100 °C and applying a reverse bias across the n+p junction, lithium ions are driven out of the n+ lithium region into the p-type bulk region, compensating or pairing with the acceptors present. After some time, the lithium profile assumes the shape represented by the solid line in the figure, at which point the following differential equation describes the growth rate of the lithium-ion compensated region:

\[ \frac{dW}{dt} = \mu_L N_A E - \frac{V}{W} N_L W R_L \]

where \( \mu_L \) is the lithium-ion mobility, \( N_A \) is the net acceptor concentration in the p-type wafer, and \( V \) is the voltage applied across the compensated region. \( W \) is the concentration of lithium-ion precipitation sites and \( R_L \) is the average rate of lithium-ion loss due to these sites.[3] From this simple model and Eq. (1) two points can be readily made regarding the silicon crystal quality:

1. The lithium-ion concentration profile needs to contain sufficient lithium to fully compensate the p-type wafer. However, the lithium concentration cannot be arbitrarily increased by a longer, higher temperature diffusion schedule as this can lead to the introduction of unwanted impurities (Cu, Au), which can seriously degrade the crystal quality. Therefore there is a lower bound on the crystal purity that can be used in the Si(Li) process - about 100 ohm-cm, and an upper resistivity bound also becomes apparent. If the normalized lithium-ion loss term, \( N_L W R_L / N_A \), becomes significant in comparison with the ideal lithium-ion flux, \( \mu_L V / W \), the compensation process will slow or stop. Silicon crystals often have imperfections that can act as lithium-ion "loss" sites. These sites, as can be seen from Eq. (2), become more dominant in higher resistivity (low \( N_A \)) silicon. There is, therefore, an upper bound on the crystal purity that can be used in this Si(Li) process - about 10,000 ohm-cm.

Within these two bounds, experience in fabricating Si(Li) detectors at LBNL, over many years, indicates that p-type floating-zone (FZ) crystals with 1-2 K ohm-cm resistivity normally yield usable Si(Li) detectors, providing that there is not an excessive concentration of lithium-ion loss sites.

This silicon requirement for the CRIS Si(Li) detectors presented a major problem for TOPSIL, who have for over twenty years supplied LBNL with Si(Li) detector grade silicon. The difficulty is due to a segmentation that has evolved in recent years in the production of high-purity FZ silicon. TOPSIL, like many other silicon crystal vendors, now depend on one or two polysilicon producers for their polysilicon feedstock. The polysilicon producers have striven to improve the purity of their material, with the result that polysilicon directly suitable for the production of Si(Li) detectors at LBNL, over many years, indicates that p-type floating-zone (FZ) crystals with 1-2 K ohm-cm resistivity normally yield usable Si(Li) detectors, providing that there is not an excessive concentration of lithium-ion loss sites.

<table>
<thead>
<tr>
<th>Impurity</th>
<th>Concentration (Maximum)</th>
<th>Resistivity (Minimum)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Boron</td>
<td>0.02 ppba</td>
<td>12,000 ohm-cm: P</td>
</tr>
<tr>
<td>Phosphorus</td>
<td>0.02 ppba</td>
<td>5,000 ohm-cm: N</td>
</tr>
<tr>
<td>Carbon</td>
<td>0.10 ppma</td>
<td>-</td>
</tr>
</tbody>
</table>

A FZ crystal produced from a polysilicon rod with the specifications given in the table will have a resistivity of about 5 K ohm-cm and the crystal will be n-type.

TOPSIL has now developed a system to dope crystals with boron during the actual FZ pulling process so that the final crystals are p-type with a resistivity of 1-3 K ohm-cm.
This process requires knowing the donor concentration in the polysilicon and to date the axial resistivity variations have been of the order of 20%, which may be reduced in the future by better characterization of the starting polysilicon material. Wafers from two different FZ crystals grown with this doping technique were then used to fabricate the CRIS Si(Li) detectors.

III. FABRICATION OF THE CRIS SI(LI) DETECTORS

An abbreviated LBNL process sequence for fabricating Si(Li) detectors is shown in Fig. 3. We have discussed this process sequence in detail earlier [5] and will only highlight a few aspects here where this sequence was modified to facilitate the fabrication of the 100 mm diameter CRIS detectors.

1. Diffuse in lithium N⁺ contact 375 °C, 15 minutes
2. Ultrasonically cut grooves into wafer
3. Chemically etch grooves on top and "well" into bottom side
4. Drift lithium ions into the bulk of the detector at 110 °C, 500 V
5. Initial detector performance check
6. Mechanical thinning of lithium contact
7. Final detector testing

Fig. 3. A simplified LBNL Si(Li) fabrication sequence for single groove detectors. Double groove detectors, which constitute most of the CRIS Si(Li) detectors, have a second groove cut after Step 5.

All of the fabrication steps, except 2 and 6 in Fig. 3, were performed in LBNL's new Instrument Support Laboratory Class 10,000 cleanroom, which has 22 ± 1 °C room air temperature control and 50 ± 5% relative humidity control. The machining steps, cutting the grooves in the wafer (Step 2) and thinning down of the lithium n⁺ contact (Step 6), required the design of special tooling. For groove cutting, we developed a fixture for holding the wafers so that the maximum eccentricity in cutting the initial groove was less than 0.05 mm. We also modified a lapping machine so that we could precisely control the thinning of the lithium contact to better than 0.01 mm. As discussed further in Section IV, the lithium contact thickness is important to the CRIS Si(Li) detector performance in cosmic ray identification. As a quick check of this contact thickness during thinning, we measured the lithium contact sheet resistance and required that this resistance be greater than 20 ohms, which we had earlier determined corresponded to a lithium contact thickness of less than 20 μm, as measured using ²⁴¹Am alphas.

The lithium drifting of these detectors was greatly assisted by our use of the computer controlled drift stations, which we have also reported in an earlier paper.[6] These units, while monitoring the drift voltage, temperature, and leakage current also calculate the theoretical drift depth. If the lithium-ion loss term in Eq. (2) can be ignored, then Eq. (2) has a simple solution:

\[ W = (2 \mu L V t)^{1/2} \]

where \( t \) is the total drift time. Our lithium-ion drift computer program uses Eq. (3), with corrections for the temperature dependence of the lithium-ion mobility, to estimate the actual compensated depth during the drifting process. Our nominal drift conditions for these detectors were 500 V, 110 °C. The settings on the computer program were chosen to slowly (over two days) ramp up first the temperature and then the drift voltage so that at no point during reaching the nominal drifting conditions did the drift leakage current become unstable. Typically during the drift the leakage currents were less than 5 mA, were stable, and the drift proceeded without any difficulty. We successfully drifted some 167 wafers out of the 168 wafers obtained from TOPSIL, although not all of these finally passed the CRIS mechanical and electrical specifications. Figure 4 shows a bare silicon wafer, the CRIS detectors mounted in their test packages, and an assembled test telescope. The packages shown are only for facilitating laboratory testing and are not the actual CRIS flight instrument packages.

IV. PERFORMANCE OF THE CRIS SI(LI) DETECTORS

The delivered CRIS detectors had electrical and mechanical characteristics summarized in the table below.

<table>
<thead>
<tr>
<th>Detector Style</th>
<th>QTY</th>
<th>Average Leakage Current (μA) 400 V 22 °C</th>
<th>Average Noise (keV) at 3.2 μsec peaking time</th>
<th>Average Total Thickness (μm), gauge measured</th>
</tr>
</thead>
<tbody>
<tr>
<td>Double Groove</td>
<td>74</td>
<td>18.4 ± 2.8 (Total) 5.9 ± 1.7 (Center)</td>
<td>140 ± 52</td>
<td>2990 ± 30</td>
</tr>
<tr>
<td>Single Groove</td>
<td>29</td>
<td>14.9 ± 3.5</td>
<td>180 ± 69</td>
<td>2990 ± 30</td>
</tr>
</tbody>
</table>

The CRIS Si(Li) detectors are put through an extensive set of tests in order to establish their long-term stability and...
suitability for flight, and to obtain detailed calibrations which can be used in the interpretation of the flight data.

The detector stability is checked in a thermal-vacuum life test carried out over a period of 30 days. During this time the detectors are kept continuously under bias in a vacuum $1 \times 10^{4}$ Torr while a record is kept of their noise and leakage current. Over the course of this test the detector performance is checked at room temperature, $+35^\circ\text{C}$ and $-30^\circ\text{C}$. We are presently evaluating these tests to determine the current and noise stability of these detectors and to select a detector set that we can expect to perform well over a period of two or more years in space.

In order to achieve $\sigma_M < 0.25$ amu for iron-group nuclei, the thickness of silicon penetrated by each particle must be known to an accuracy of a few microns. The presence of a high density of lithium at one surface of the Si(Li) detectors results in a "dead layer", which if not known precisely, can reduce the spectrometer's resolution.

To map the thickness of the silicon wafer and of the dead layer we make two sets of measurements. First, a non-contact capacitance gauge is used to determine the thickness of the silicon wafer at 24 discrete points distributed over its surface. These measurements have an absolute accuracy of better than 1 $\mu$m. Second, a map of the full detector area is obtained by using a beam of energetic $^{36}\text{Ar}$ ions from the cyclotron at Michigan State University. These particles penetrate the detector that is being mapped, producing an energy loss signal, $\Delta E$, and stop in a second Si(Li) detector where they deposit their residual energy, $E'$. In addition to $\Delta E$ and $E'$, we also measure each particle's point of incidence on the detector surface. The residual energy, $E'$, varies from point to point with any thickness variation of the $\Delta E$ detector.

Figure 5 is a plot of the $E'$ energy signal versus the capacitance gauge thickness measurements for all of the points at which the latter measurements are available on 72 $\Delta E$ detectors. A linear fit to these data gives us a conversion from the $E'$ energy to absolute thickness. Using this relation we can then use the accelerator beam data to extend the thickness map over the entire detector area. Figure 6 shows such a thickness map for a typical CRIS Si(Li) detector.

Fig. 5. A plot of $E'$ versus capacitance gauge thickness measurements

Fig. 6. An $^{36}\text{Ar}$ beam derived thickness map of a CRIS Si(Li) detector. The dimensions on the contours are in $\mu$m.
In Fig. 7 we show mass histograms obtained using the accelerator data for the ΔE detector shown in Fig. 6. The width of the upper histogram, obtained assuming a constant thickness for the ΔE detector, contains a major contribution from the thickness variations of the detector. When we use a detector thickness map (Fig. 6) to derive the ΔE thickness penetrated by each particle we obtain the lower histogram in Fig. 7. The width here is dominated by fluctuations in the energy-loss process, which imposes an intrinsic limit on the resolution attainable with this technique. In the flight instrument we are able to further improve on this resolution through the use of multiple detectors to measure ΔE.

![Mass histogram for 36Ar](image)

**Fig. 7.** An 36Ar mass histogram produced using ΔE and E' measurements from a pair of CRIS Si(Li) detectors, both before correcting for thickness variations (upper panel) and after (lower panel).

The sum of the two detector signals, ΔE + E', can vary from point to point because of the variation in the detector’s dead layer thickness. In order to calibrate this relationship, we also collected 36Ar data with various aluminum foil absorbers placed immediately in front of the detectors. Analysis of these data and its application to mapping the dead layer on each CRIS detector is still in progress.

**V. CONCLUSIONS**

This is an interim report on our progress to date in realizing the 100 mm diameter, 3 mm thick Si(Li) detectors for the CRIS instrument on the Advanced Composition Explorer Mission. We have highlighted the critical dependence of the Si(Li) detector fabrication on the silicon crystal parameters and have outlined the approach taken by TOPSIL in addressing these requirements. Preliminary data on the 36Ar beam thickness mapping of the CRIS Si(Li) detectors and on the mass resolution suggest that the CRIS instrument will achieve the mass resolution objective of σM < 0.25 amu required for the identification of Fe isotopes, which provide a significant signature on cosmic ray evolution.

**ACKNOWLEDGMENTS**

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**REFERENCES**


