SURFACE PASSIVATION OF HIGH PURITY GERMANIUM DETECTORS USING AMORPHOUS HYDROGENATED SILICON FILMS

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ABSTRACT

High purity germanium (HPGe) detectors used in Radionuclide Aerosol Sampler/Analyzer (RASA) systems must be operational unattended for long durations. To fulfill the critical requirement, our work strives to improve HPGe detector long term reliability and resolution performance through investigating new surface passivation techniques to passivate and protect the surface area between electrical contacts.

Amorphous hydrogenated silicon (AHSi) films as the passivation alternative to CANBERRA standard silicon oxides for germanium detectors have been investigated. The AHSi films are deposited on germanium surfaces using a radio frequency (RF) magnetron sputtering technique in an environment of argon and hydrogen gas mixture. Sample analysis shows that the hydrogen atoms are well incorporated into the amorphous films and play an essential role in saturating dangling bonds and minimizing surface states on germanium surfaces. Excellent AHSi passivation films have been attained with this technique.

Two HPGe detectors with AHSi films as the passivant have been successfully fabricated. Test results demonstrate that the AHSi films have the following characteristics in passivating HPGe detectors: low leakage current, low noise contribution, good temperature stability of resolution and noise, ability to withstand thermal stress and maintain physical integrity, and ruggedness in atmospheric rapid thermal cycle and aging tests. Amorphous hydrogenated germanium (AHGe) film as a passivant deposited by sputtering techniques will be investigated later in this project.

OBJECTIVES

The objective of this research is to develop passivation techniques that provide superior resolution and reliability in HPGe detectors. The research is driven by the critical need in radionuclide assay systems such as Radionuclide Aerosol Sampler/Analyzer (RASA) systems that HPGe detectors must be in operation unattended for long periods of time. Current commercially available germanium detectors using the SiOx (x~2) passivation technique have demonstrated acceptable performance and reliability for most laboratory applications in radiation detection and measurement. However, for remote deployments such as RASA systems designed for nuclear detonation detection and analysis, detector performance and reliability must be further improved to achieve superior resolution and noise as wells as enhanced durability for long-term operation.

Passivation of germanium surfaces is one of the key factors that affect the performance and reliability of HPGe detectors. The germanium surface between the electrical contacts must be passivated to minimize surface related leakage current. Also, the intercontact surface must generate little dielectric noise. Both surface leakage noise and passivant dielectric noise must remain low to achieve an acceptable signal–to-noise ratio. Unlike native oxides on silicon surfaces which provide excellent passivation, naturally formed germanium oxides on germanium surfaces have unacceptable stoichiometries which may result in charged states. Charged oxide states may cause an inversion layer in P-type germanium or an accumulation layer in N-type germanium (Brown, 1953). Traditionally, a SiOx film deposited by a well-controlled SiO evaporation process in an oxygen-rich environment is used to passivate germanium surfaces (Holland, 1970). The SiO_x process has been a standard technique for commercial detector products at CANBERRA. However, some variabilities in reverse leakage currents as well as dielectric noise associated with the intercontact surfaces have been observed. Surface issues resulting from the evaporated SiO on germanium have been previously investigated (Bardeen et al., 1956; Dinger, 1976). Controlling the surface chemistry and passivating germanium surfaces to achieve a consistently neutral intercontact surface is of paramount importance in the success of HPGe detector manufacturing processes (Martin, et al., 2008; Martin, et al 2009).

In this work, the feasibility and performance of AHSi films as a passivation alternative to CANBERRA standard silicon oxides for germanium detectors were investigated. AHSi films are considered to be effective passivants in the microelectronics and photovoltaic industries (Searle, 1998). The incorporation of hydrogen in amorphous films effectively compensates dangling bonds within the film as well as on the substrate surface. The result on a single crystal germanium surface is to reduce the surface density of states at near mid-band gap. In this research AHSi films were deposited onto germanium surfaces in an argon and hydrogen gas mixture using a sputtered film technique. HPGe detectors fabricated with the AHSi films as the passivant demonstrated excellent resolution performance and reliability.

RESEARCH ACCOMPLISHED

Characterization of sputtered AHSi passivation films

AHSi films were grown using a RF magnetron sputtering technique. The composition and chemistry of the films were characterized by standard analysis techniques to ensure high-quality films were deposited. Germanium test samples were prepared for the growth of AHSi films. The pre-etched samples were transferred into our sputtering chamber at CANBERRA. The vacuum chamber was pumped down to a vacuum level in the range of 10^{-6} Torr and a silicon target was sputtered. An argon / hydrogen gas blend was used as the deposition plasma to create the amorphous films. The film samples were sent to Evans Analytical Group of Sunnyvale, CA to examine the film composition and quality. The laboratory analysis, utilizing state-of-art scientific instruments, investigated the film surface morphology, thickness, atomic concentration, and physical and chemical states.

Figure 1 shows the test samples prepared for laboratory analysis and a scanning electron microscopy (SEM) surface image of our sputtered AHSi film on a germanium substrate. The surface topography of the initially sputtered film was poor presenting with non-uniformities manifested by defects such as bubbles and circular holes. The dark circles appeared to be areas that were beginning to bubble but had not yet flaked off. The circular holes were the areas where the film was missing. It is extremely important to obtain a dense and homogenous film for passivation on germanium surfaces, so that the surfaces become impervious to ambient atmosphere. The partial coating of germanium surfaces could deleteriously compromise germanium detector performance and reliability. The presence of circular holes penetrating the sputtered films is possibly due to several reasons such as poor surface preparation, compressive stress, or embedment of deposition gases. To eliminate these adverse effects, several sputtering tests to optimize the growth conditions were carried out. In RF sputtering techniques, gas pressure, RF power, sample to target distance, and sputtering time are the four critical parameters for achieving high quality sputtered films. With optimized conditions, excellent passivation films have been successfully grown. The bubble-like defects found on the germanium test samples are not present on germanium surfaces in our newly fabricated germanium detectors with AHSi films as the passivants.

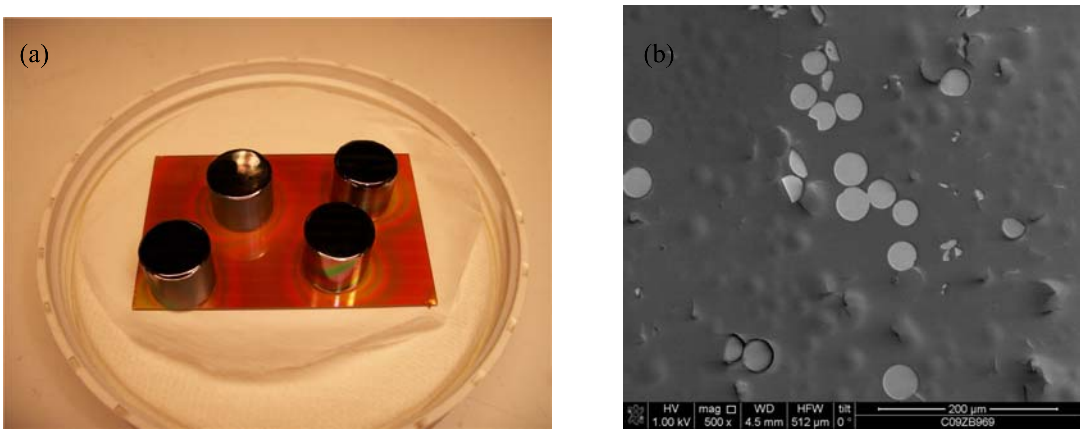


Figure 1. (a) Sputtered AHSi film test samples deposited on germanium substrates (b) Scanning electron micrographs of sputtered AHSi film grown on a HPGe substrate. The film shows the bubble-like defects on germanium surfaces.

The thickness and the elemental composition of the AHSi film were determined by Rutherford Backscattering Spectrometry (RBS) analysis, and the surface chemistry was examined by X-ray photoelectron spectroscopy (XPS). We are mostly interested in how much hydrogen was actually incorporated into the film during the deposition. Table 1 summarizes the results from the RBS analysis showing that the atomic concentration of hydrogen in AHSi films is about 21%. XPS results also suggest that the sputtered film on the germanium substrate is composed primarily of elemental Si with lower levels of SiO_2, indicating that the silicon atoms are bonded/clustered with the hydrogen atoms. The success of high percentage incorporation of hydrogen into the films is instrumental to our study in compensating the dangling bonds on germanium surfaces, suggestive of a high quality AHSi passivant for germanium surfaces.

Table 1. RBS analysis of the sputtered AHSi films

Depth (Å)	**Atomic concentration (%)**			**Density**
	Si	**H**	**Ge**	**(at/cc)**
6500	78.8	21.2	-	6.70×10^{22}
Bulk	-	-	100	4.42×10^{22}

Detector Fabrication using AHSi Passivation Films

Standard electrode germanium (SEGe) detectors P84648A and P3488B were fabricated using the sputtered AHSi technique to evaluate the passivation performance of the films. The detectors had been evaluated in the past with different passivation techniques which resulted in good resolution and low noise performance. Previously the core hole was implanted with boron ions to form a thin p^+ contact, and an outside wraparound thick Li layer was produced to form a rugged n^+ contact. After both detectors received a nitric/hydrofluoric acid solution etch of the groove surface to obtain a clean germanium surface, they were immediately transferred into a vacuum process chamber for AHSi film depositions. Figure 2-a shows the SEGe detector P3488B where the intercontact surface was passivated with an AHSi film. The film presented a dense and uniform coating on the germanium intercontact surface. No visible porous or fissure defects were found in the film with microscopy inspections, which makes this detector surface passivation nearly impervious to ambient atmospheric conditions. Figure 2-b shows the I-V and C-V measurements at liquid argon temperature for detector P3488B. The leakage currents are generally low but with a gradual increase as the reverse bias increases from 3000 V to 5000 V. The depletion voltage is around 3400 V, and the capacitance after depletion is about 34 pF. These results suggest that the AHSi passivation technique results in low leakage current necessary for high resolution germanium detectors. The AHSi passivant withstands high electric field between the two electrical contacts, and induces low or no density of charged surface states which would result in surface leakage current.

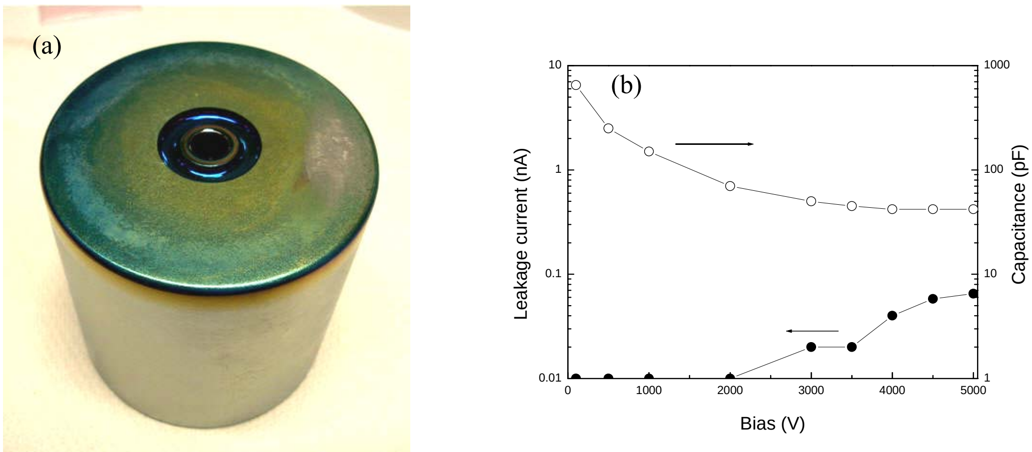


Figure 2. (a) SEGe detector P3488B for which the intercontact surface was passivated with the sputtered AHSi film. (b) C-V and I-V characteristics for the SEGe detector P3488B at liquid argon temperature (87 K).

Detector Resolution and Noise

To measure the detector resolution and performance, the detector P84648A was mounted into a CANBERRA 7500SL dipstick cryostat. The cryostat cold finger was inserted into a liquid nitrogen Dewar for detector performance tests and then placed into a liquid argon Dewar for re-evaluation. Table 2 shows test results of detector P84648A as a function of reverse bias including detector resolution, noise, peak-to-Compton ratio, and detector efficiency. Detector resolution is represented by full width at half maximum (FWHM) and full width at tenth maximum (FWTM) of ^{60}Co (1.33 MeV) and ^{57}Co (122 keV) and the electronic noise by the FWHM of an injected pulser signal. Increasing the reverse bias appears to slightly worsen the detector resolution. At 3.5 kV bias, the FWHM of ^{60}Co at 1.33 MeV and ^{57}Co at 122 keV are 1.68 keV and 0.87 keV, respectively. While at 5.0 kV bias, the FWHM of ^{60}Co at 1.33 MeV and ^{57}Co at 122 keV are 1.73 keV and 0.95 keV, respectively. The deterioration of detector noise is related to the increase of the parallel noise due to the effect of increased detector leakage current. The detector relative efficiency is 21%, which is in agreement with the previous result where the wet chemical oxide (WCO) passivation was employed (Martin et al., 2009). The detector maintained constant resolution and efficiency while being biased over a period of 12 hours. Finally, the cryostat was placed into a liquid argon Dewar to warm the detector by 10 K. Subsequent measurements showed that the detector's resolution and relative efficiency were consistent with the LN_2 measurements, suggesting good temperature stability of the detector performance.

Detector P3488B was mounted into a CANBERRA Cryo-Pulse® 5 (CP-5) electrically cooled system. The CP-5 cryocooler allows controlled variation of the detector temperature over a wider range than permitted by a liquid cryogen cooled cryostat. Detector test results at a temperature of 93 K are listed in Table 3. The resolution is approximately 2 keV at 1.33 MeV for ^{60}Co, and 1.25 keV at 122 keV for ^{57}Co. The injected pulser signal resolution is 1.18 keV. The detector relative efficiency is approximately 100% consistent with the active volume the detector, and the peak-to-Compton (P/C) ratio is 73:1.

Table 2. Detector evaluation for detector P84648A with shaping time of 6 μs.

Bias (kV)	RESOLUTION (keV) ^{60}Co		^{57}Co		Pulser	P/C	Eff.	Comment
3.5	1.68	3.11	0.87	1.63	0.75	53.5	20.6%	Test in LN_2
4.0	1.70	3.10	0.86	1.58	0.74	52.5		
4.5	1.70	3.12	0.91	1.70	0.78	51.8		
5.0	1.73	3.19	0.95	1.72	0.78	53.5		
Bias overnight								
3.5	1.65	3.06	0.84	1.53	0.72	54.8	20.4%	
3.5	1.71	3.10	0.84	1.55	0.76	52.5	20.5%	Test in LAr

Table 3. Detector evaluation for detector P3488B with shaping time of 6 μs.

Bias(kV)	RESOLUTION (keV) ^{60}Co		^{57}Co		Pulser	P/C	Eff.	Det. Temperature
4.0	2.01	3.77	1.25	2.25	1.14	73.2	95.4%	93 K
Bias overnight								
4.0	1.99	3.69	1.25	2.30	1.17		97.9%	

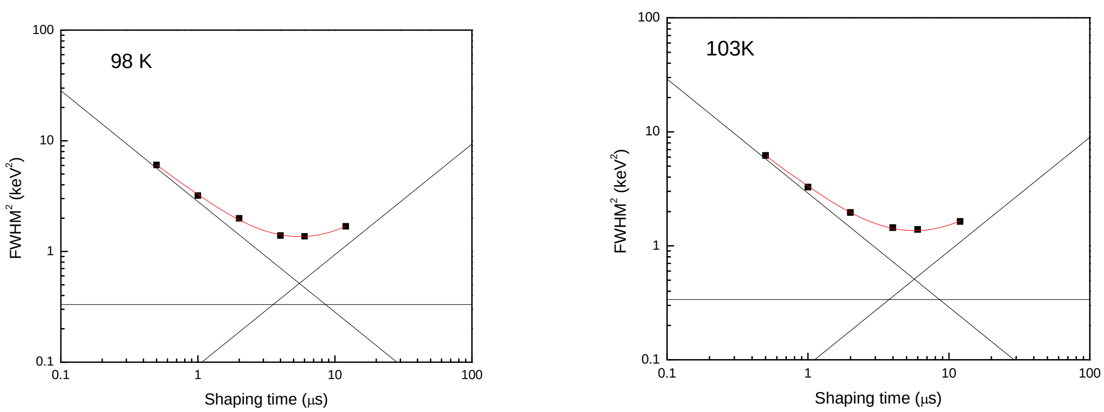


Figure 3. Noise analysis of detector P3488B. The pulser FWHM measurements were carried out at 98 K and 103 K, respectively. The three straight lines in each figure represent series, parallel, and 1/f noise, respectively.

Sources of electronic noise in germanium detectors are associated with detector capacitance, leakage current, and intercontact surface noise. Therefore, the total noise encompasses three independent categories which sum in quadrature as given by the following expression

$$FWHM_{pulser}{}^{2} = N^{2}_{series} + N^{2}_{parallel} + N^{2}_{1/f} = \frac{k_1}{\tau} + k_2\tau + k_3 \ . \qquad (1)$$

Series and parallel noise correspond to detector capacitance and leakage current, respectively, and are functions of amplifier shaping time. The 1/f noise component is partly contributed by the detector intercontact surface and is not a function of amplifier shaping time (Bertolini and Coche, 1968). The pulser FWHM data as a function of shaping time from 0.25 μs to 10 μs were measured for the detector P3488B at 98 K and 103 K. The data ($FWHM_{pulser}{}^{2}$) were fitted with equation (1) using a nonlinear least squares method. The fitted curves are shown in Figure 3. The three components of the noise are individually shown in both figures as straight lines. At 98 K, the fitting parameters

k_1, k_2 and k_3 are 2.82, 0.09, and 0.33, respectively, and at 103 K, the fitting parameters k_1, k_2 and k_3 are 2.89, 0.09, and 0.34, respectively. The *1/f* noise contribution from AHSi passivation is lower than that for the detector previously fabricated with the standard SiOx passivation technique where k_3 was 0.61 (Martin, et al.,2009).

Temperature stability

Surface channel effects can greatly compromise detector performance if germanium surfaces are not effectively passivated. In addition surface channels may be temperature sensitive which can further deteriorate HPGe detector performance at higher operating temperatures (Hull, et al., 1995). Depending on the efficacy of a surface passivation in eliminating surface channels, the detector resolution and noise can be greatly improved if the passivation is stable over a broad temperature range. This may not only improve detector resolution at higher temperatures, but also reduce the input power of the cryocooler and mitigate the associated microphonics noise.

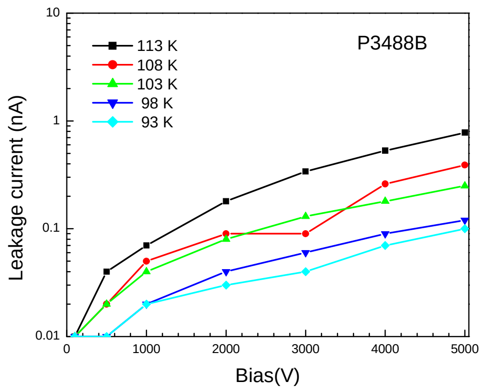


Figure 4. I-V characteristics of detector P3488B at various detector temperatures.

Lowering the cooling power of the CP-5 allowed the progressive increase of the detector temperature from 93 K to 113 K. At each temperature, the detector leakage current and resolution were measured to determine the temperature effect on detector performance. Figure 4 shows the detector leakage current as a function of reverse bias at various temperatures. As can be determined from the graphs, the leakage current gradually increases as the temperature increases. However, it is observed that increasing detector temperature by 20 K from 93K does not compromise its resolution and noise performance. As shown in Table 4, the FWHM and FWTM for ^{60}Co (1.33 MeV) and ^{57}Co (122 keV) sources and FWHM for pulser, as well as the P/C ratio and relative efficiency for the detector P3488B at various temperatures do not change significantly. Compared to the standard SiOx passivant from previous investigations, detector P3488B with AHSi as its passivant demonstrates excellent resolution and noise performance over a wide temperature range. Our earlier data for the detector with the standard SiOx passivant showed that the detector became much noisier once the detector was warmed to above 103 K. The improved temperature stability reported here is due to the unique properties of the AHSi films produced by the sputtering technique developed during this research.

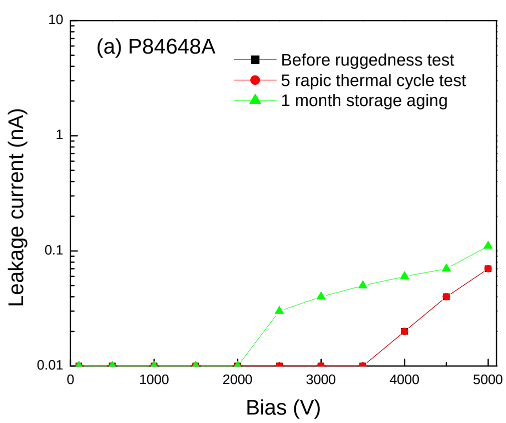
Table 4. Detector evaluation for detector P3488B at various detector temperatures.

Bias (kV)	RESOLUTION (keV) ^{60}Co	^{57}Co	Pulser	P/C	Eff.	Det. Temperature
4.0	1.99 3.69	1.24 2.30	1.17	70.0	100%	98 K
4.0	1.96 3.64	1.24 2.33	1.18	69.0	98.1%	103 K
4.0	1.95 3.60	1.26 2.39	1.16			108 K
4.0	1.97 3.62	1.26 2.31	1.14			113 K

Detector Ruggedness

To examine the AHSi passivation method for ruggedness, two tests were carried out for both detectors P84648A and P3488B: rapid thermal cycles and atmospheric aging. Plots of leakage current as a function of reverse bias for the ruggedness tests are shown in Figure 5 for both devices. The leakage current characteristics before and after the ruggedness tests were measured, providing a direct comparison of leakage performance. Five rapid thermal cycle tests were performed for each detector from 87 K to approximately 300 K. No change of film color and morphology were observed during the tests. The leakage current of detector P84648A showed no change after five thermal cycles, however, a slight increase of leakage current for detector P3488B occurred after five rapid thermal cycles.

The atmospheric aging test was carried out by storing both detectors in a nitrogen-purged dry box over an extended period of time. After one month of dry box aging, the leakage current for detector P84648A increased at 3000 V reverse bias from 0.01 nA to 0.04 nA. The increase in leakage current was negligible since a subsequent retest showed the same resolution and noise as they were prior to the ruggedness tests. For the device P3488B, the leakage current improved during the one month aging test, indicating a moderate improvement of the surface condition. The AHSi passivant performance was not compromised by storage/aging in a clean and dry storage environment, which contrasts with the standard SiOx passivant. Our previous results had shown that the standard SiOx passivant introduced much more leakage current and noise after a four week aging process. These results demonstrate that the detector fabricated with the new AHSi technique as its passivant can have a much longer shelf lifetime than the one with SiOx.



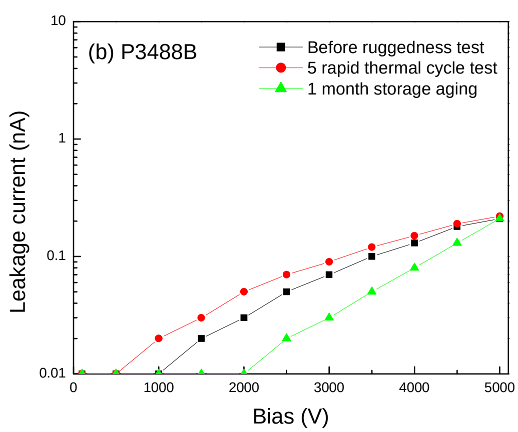


Figure 5. I-V ruggedness tests including five rapid thermal cycles and one month storage aging for the two SEGe detectors (a) P84648A and (b) P3488B, respectively.

CONCLUSIONS AND RECOMMENDATIONS

Two HPGe detectors were successfully fabricated with AHSi film as the intercontact surface passivant using a RF magnetron sputtering technique. Detector test results demonstrated acceptable resolution and noise performance, suggesting the effectiveness of a sputtered AHSi technique in passivating germanium surfaces for high resolution germanium detectors. The fabricated detectors tolerated temperature cycling between room temperature and cryogenic temperatures, were stable with time without compromising resolution or efficiency, and tolerated ruggedness tests by maintaining acceptable performance after thermal quenches and atmospheric aging. Low leakage current was achieved suggesting that AHSi films are electrically stable and are capable of withstanding strong electric fields. Excellent temperature stability of resolution and noise will allow detectors to operate at higher temperatures. This will help to mitigate microphonics induced by cryocoolers by allowing them to operate at lower input power. Low 1/f noise contribution from the intercontact surface will greatly benefit small germanium detectors for which ultra-low noise is required.

This research demonstrated that amorphous hydrogenated semiconductor films can be used as passivants for germanium surfaces for high resolution germanium detectors. In the next phase of this research the feasibility and performance of AHGe films will be investigated. It is expected that AHGe may have better performance in passivating germanium surfaces than AHSi. It is intuitive to believe that superior interface between germanium surfaces and AHGe passivant can be achieved since interface bonding stoichiometry should be better. At the interface, the ordered Ge atomic lattice may seamlessly morph into a disordered Ge network that removes strained Ge–Ge bonds. Any unsatisfied bonds created during the film deposition will be mitigated by the creation of Ge–H bonds. Figure 6 displays initial results of the sputtered AHGe film as the passivant for a HPGe detector along with the collected ^{57}Co spectrum. Low leakage current and acceptable resolution and noise have been accomplished, suggesting that AHGe films may be useful in passivating germanium surfaces for HPGe detectors.

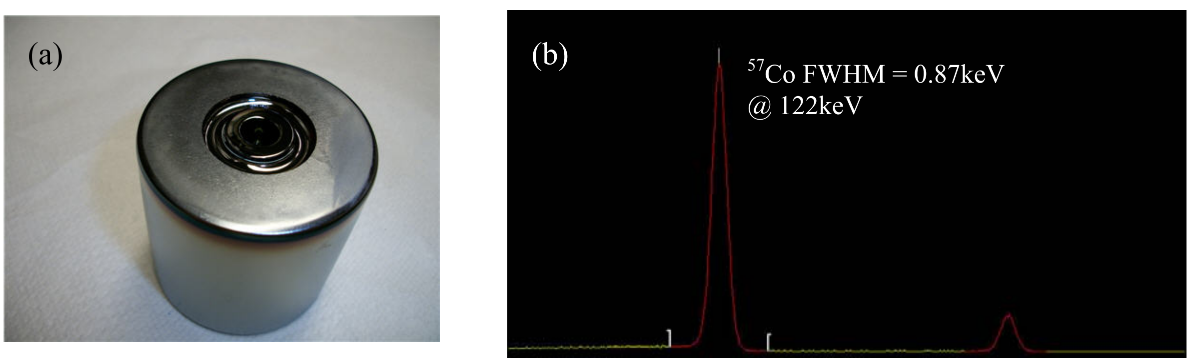


Figure 6. (a) SEGe detector P84648A for which the intercontact surface was passivated with the sputtered AHGe film. (b) ^{57}Co spectrum showing a resolution of 0.87 keV at 122 keV.

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