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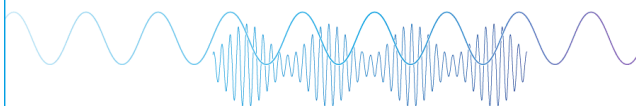
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ABSTRACT

Several experimental disciplines require simultaneous neutron flux and temperature monitoring. This paper provides the groundwork on a diamond sensor concept that could be used in harsh environments where the limits of current technologies are exceeded. Here, we present initial results on the functionality of a multimodal diamond sensor, which can simultaneously sense the presence of alpha particles at a few counts per second at temperatures up to 494 K. Such a sensor may also have applications where both charged particles or neutron detection and temperature measurement are simultaneously desired.

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While a multimodal diamond sensor has a multitude of potential applications, next generation nuclear power plants (NPPs) are an effective analog for numerous fields due to their harsh environments. Instrumentation and Control (I&C) and safety systems rely on stable and accurate sensor platforms. Sensor platforms, such as temperature and power level (neutron flux), of a nuclear power plant (NPP) are required to maintain safe operating conditions. Inside the core of a nuclear reactor, fast and reliable temperature and neutron flux monitoring is challenging due to harsh environments, and with some Generation IV reactors envisioned, current sensing technologies are not ideal. Furthermore, stable sensors for both high temperature and fast and thermal neutron flux in real time are a necessary tool in evaluating accident tolerant fuels and materials.

The primary challenge in temperature sensing is its operating range up to 1273 K and its reliability under extensive neutron/gamma ray irradiation.^{1,2} Current thermocouple technology is well-suited for current NPPs but seemingly unsuitable for high temperature and materials testing reactors. Thermocouple types K and N fail under neutron irradiation at temperatures above 1273 K, and types C and S have larger neutron cross sections, leading to transmutation and unacceptable temperature drift.^{3,4} Recent experiments with Mo/Nb thermocouples have not demonstrated suitable stability at high temperatures,^{1–3,5} but doped Mo-Nb1%Zr type thermocouples show some promise by exhibiting <2% drift at temperatures up to at least 1773 K.^{5–7} Modeling and experimentation of the transmutation of

Mo/Nb in thermal reactors indicate comparable levels of transmutation to type K thermocouples.⁸ Operating resistance thermometer detectors (RTDs) at high temperatures results in thermal expansion and changes in platinum within them, resulting in de-calibration.⁹ For this reason, they are generally kept at temperatures below 723 K for the highest accuracy applications. Johnson noise thermometers (JNTs) are under development in an attempt to overcome thermocouple instabilities and small signal analysis in RTDs operated in harsh environments.² This includes JNTs being implemented parallel to RTDs, which can provide correction factors to recalibrate RTDs during operation, mitigating the effects of drift and extending their operational lifetime.^{10,11} However, electromagnetic interference (EMI) that has impeded JNTs for in-pile temperature measurements and EMI subtraction via an antenna to remove the Johnson noise have resulted in a few percent error in an industrial setting at a few hundred degrees Celsius over a short duration, validating that EMI removal is possible.¹¹ Fiber Bragg Gratings (FBGs), which provide multipoint sensing along a single fiber, have indicated survivability to a fast neutron fluence of $\sim 10^{19} \text{ cm}^{-2}$ at 564 K for 22 days but resulted in temperature uncertainties from 277 to 350 K.¹²

In the case of power monitoring of a NPP, thermal neutron sensing is either ex-core, near-core, or in-core, using self-powered neutron detectors (SPNDs), fission chambers, or compensated ionization chambers. For Gen IV and experimental reactors, current commercial options are not able to sense the fast neutron flux in real time.

Furthermore, the high temperatures in some reactor designs and testbeds (e.g., TREAT) are beyond their tolerance level. Fission chambers with ^{235}U and ^{238}U could also be used to get the combined thermal and fast neutron flux but not separately and convoluted with each isotope's response function (i.e., energy dependent cross section containing many resonances).² A high temperature fission chamber (HTFC), consisting of a miniature ^{242}Pu fission chamber compensated with a ^{235}U fission chamber or rhodium SPND, is under development to measure the thermal and fast neutron flux at the BR2 reactor,^{13,14} and the experimental data for thermal flux monitoring agree with the SPND after applying flux gradient correction factors.¹³ Similarly, the data for fast flux monitoring agree with models but require more corrections with larger uncertainties. Comparisons with the HTFC measurements and activation dosimetry data show some promise in the accuracy of these systems.¹⁴ Furthermore, a review on sodium-cooled fast reactors suggests that ^{242}Pu fission chambers are the ideal choice over compensated ion chambers and SPNDs.¹⁵ However, others point out that further development of fission chambers is needed due to high leakage currents and/or discharges during high temperatures, harsh environment operation.^{2,16,17} As of the publication date of this article, the electrical stability has yet to be solved for temperatures above 932 K.^{18,19} Micro-pocket fission detectors (MPFDs) are also under development for in-core neutron flux measurements.²⁰ However, fast and sensitive MPFDs have not been tested in high-temperatures and high-radiation environments and may encounter similar challenges. There is the potential to overcome the temperature and power (neutron population) monitoring challenges for Gen IV reactors using diamond. It has been reported in the literature that diamond is radiation hard, chemically inert, and has been developed for use in both charged particles and neutron sensing since the turn of the century. Applications are wide and range from micro-dosimetry²¹ to fusion diagnostics.^{22–25} Thermal neutron sensing with diamond is enabled with a conversion layer,^{26,27} and fast neutrons are sensed using nuclear reactions with ^{12}C .^{28–30} Furthermore, the large 5.5 eV bandgap of diamond yields potential for high-speed, high-temperature operation due to the excellent thermal conductivity of $\sim 2200\text{ W/m K}$ and lower band-to-band carrier generation,^{27,31–37} and recent research has demonstrated the potential of diamond as a thermistor for high temperature operation.^{37,38} In these reports, it was demonstrated that the prototype diamond thermistor remained accurate and stable after multiple temperature cycles between 773 and 1073 K over a period of 10 h. Furthermore, it was found that higher purity CVD diamond exhibited a higher resistivity as a function of temperature, yielding a higher operating range over HPHT diamond. This is advantageous for the potential to combine both neutron population and temperature monitoring using the same substrate. In this report, we present experimental results indicating the possibility of a semiconductor-grade diamond-based multimodal radiation and temperature sensor for Gen IV reactors.

To fabricate the multimodal diamond sensor, an electronic grade $4.5 \times 4.5 \times 0.5\text{ mm}^3$ single crystal CVD diamond from ElementSixTM was chosen for the sensor material. The diamond substrate was cleaned using *aqua regia* and piranha chemical processes. Planar contacts were formed on each side of the diamond substrate by sputtering 50 nm of chromium and capped with 200 nm of gold. Sputtering was followed by 873 K anneal for 20 min in a 20 mTorr argon environment to make the contacts Ohmic.^{39–41} The fabricated diamond detector

was placed inside a Janis VPF-800 cryostat to control the temperature. Electrical connections were made to the diamond detector using tungsten probes; one directly to the top of the diamond and the other to a copper foil that the diamond is placed on. The copper foil is electrically isolated from the copper cold finger of the cryostat via a sapphire disk (see Fig. 1). A Lakeshore 335 temperature controller was connected to the cryostat for both temperature control and measurement. The pressure inside the cryostat was approximately 50 mTorr during experiments but slowly increased during experimentation due to lossy connections from the thermocouples. Finally, a Spectrum Techniques 0.1 μCi ^{210}Po alpha particle source was placed inside of the cryostat to evaluate the radiation response of the diamond detector at various temperatures.

To measure the temperature (DC) and radiation response (high frequency AC) simultaneously, we used the pulse processing chain shown in Fig. 2. A 200 V bias was applied to the diamond substrate using the built-in differential high voltage source on a Keithley 6487 picoAmmeter. The positive pole was directed through the high voltage bias tee on a Cremat CR-150-R5 charge sensitive preamplifier evaluation board. The negative pole of the voltage supply was passed through the current sense input of the Keithley 6487 and grounded, enabling measurement of the DC leakage current of the diamond detector through the bias resistor (R_2) and the low pass filter (R_1C_1). The other electrode of the diamond detector was exposed to the ^{210}Po alpha particles to limit the potential response to air ionization. Radiation induced current signals in diamond are high frequency with a typical pulse width around 5–20 ns,⁴² which is sufficient to pass through the AC coupling capacitor (C_2) and into the Cremat CR-110 charge sensitive preamplifier. The output from the CR-110 was shaped using an ORTEC 572A linear amplifier and digitized with an ORTEC ASPEC-927 multichannel analyzer. The temperature was manually set via the Lakeshore temperature controller between 300 and 494 K. Once the temperature stabilized, we measured the DC through the Keithley 6487 picoAmmeter and the number of alpha-particle counts above the noise floor.

When diamond is operated as a temperature sensor, it is a negative temperature coefficient (NTC) thermistor. This means that there is an exponential decrease in resistance with increased temperature, caused by thermal excitation of charge carriers across the bandgap. In semiconductors, the temperature dependent resistivity $\rho(T)$ is given as

$$\rho(T) = \rho_{\infty} \exp\left(\frac{E_a}{kT}\right), \quad (1)$$



FIG. 1. Diamond detector mounted to the cold finger of the Janis VPF-800 cryostat.

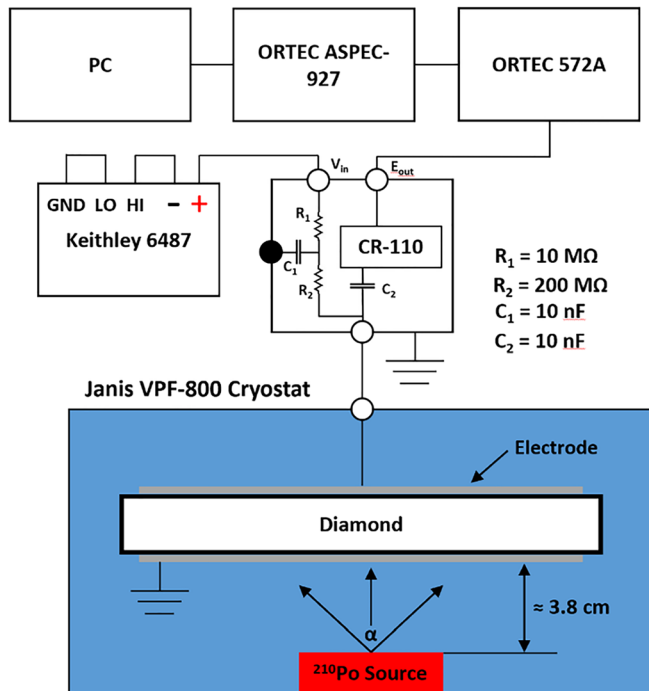


FIG. 2. Graphical representation of the experimental setup and pulse processing chain for the diamond substrate operating as both a thermistor and a radiation detector. R_1 and C_1 represent the low pass filter, R_2 is the bias resistor, and C_2 is the AC coupling capacitor to the CR-110 charge sensitive preamplifier.

where ρ_∞ is the resistivity at infinite temperature, E_a is the activation energy, T is the temperature, and k is the Boltzmann constant.^{43,44} For a thermistor of fixed dimensions and constant resistivity, the resistance (R) can be expressed as

$$R = R_\infty \exp\left(\frac{A}{T}\right), \quad (2)$$

where R_∞ is the resistance at infinite temperature and A is a material constant.³⁸ This is the basis for the Steinhart–Hart relationship given in Eq. (3) that enables determination of the substrate temperature from the observed leakage current (resistance) at some temperature T . Here, B_1 , B_2 , and B_3 are coefficients determined by fitting to experimental data,

$$\frac{1}{T} = B_1 + B_2 \ln(R) + B_3 [\ln(R)]^3. \quad (3)$$

Therefore, at each temperature T , the DC through the diamond was measured to determine its resistance. The temperature and resistance data were used to determine the three coefficients in the Steinhart–Hart relationship.

In Fig. 3, the transient dark current and temperature vs time is provided at a cryostat setpoint of 400 K. The oscillation in the temperature above and below the setpoint is normal operation for the cryostat, which is caused by the heater kicking on and off. The dark current through the diamond oscillates with the temperature but lags by approximately 25 s. We believe this lag is due to the faster

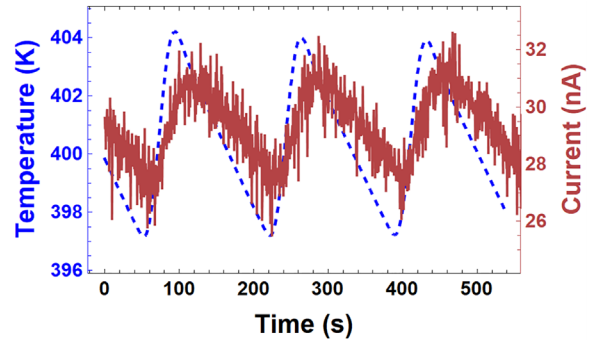


FIG. 3. Transient current (solid red line) and temperature (blue dashed line) observed when the cryostat is set to 400 K.

temperature sensing of the thermocouple embedded in the copper cold finger vs the delayed heat transfer through the sapphire disk and into the diamond detector. To extract the correct current for temperature, we correlated the peaks in the oscillations to correct for the time lag. Error in the current is represented as the standard deviation of values observed across the peak region. The resulting resistance at each temperature is provided in Fig. 4, which also includes the Steinhart–Hart fit.

The Steinhart–Hart relationship fits well with our experimental data. We found that the three coefficients B_1 , B_2 , and B_3 , were $(3.5 \pm 0.6) \times 10^{-3}$, $(-1.8 \pm 0.4) \times 10^{-4}$, and $(2.8 \pm 0.3) \times 10^{-7}$, respectively. These values do not agree with the coefficients determined by Kin *et al.*,³⁸ where the signs of B_2 and B_3 are flipped and our coefficients are all larger by a factor of 22 for B_3 and less for the other two. Other works use empirical polynomial fits on temperature^{45,46} or include the even polynomial terms of the natural log of the resistance,⁴⁷ which was found not to significantly reduce the residuals.⁴³ The temperature coefficient [$1/T$ from Eq. (3)], across a temperature range of 301–492 K for our diamond detector, was found to be $(-5.219 \pm 0.007) \times 10^{-3} \text{ K}^{-1}$, which also does not agree with other literature values within our error.^{38,46} However, the coefficients are dependent on the material properties of the diamond substrate used, which varies greatly between growth cycles and manufacturers, so

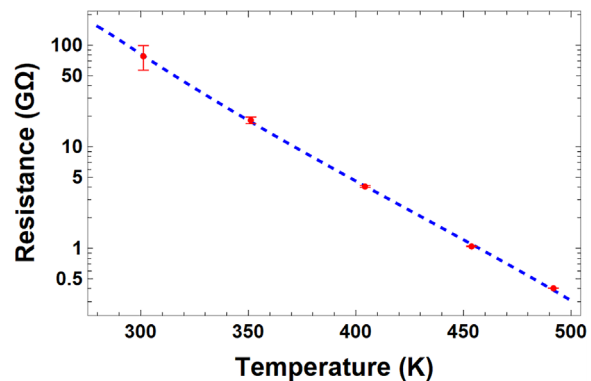


FIG. 4. Measured resistance of the diamond detector as a function of temperature. The Steinhart–Hart fit to the dataset is represented by the dashed line.

general agreement with other literature may not be appropriate. It is worth noting that the samples used by Kim *et al.* were HPHT and boron doped single crystal CVD diamonds with 300 μm thickness, and the samples used by Werner *et al.* are a combination of heavily boron doped and undoped polycrystalline CVD diamond with a total thickness of 5 μm on a p-Si substrate.^{38,46} When comparing results to our 0.5 μm thick single crystal CVD diamond, these differences could explain the discrepancies observed.

At each temperature shown in Fig. 4, the observed count rate of the diamond detector from alpha particle exposure was simultaneously measured through the AC coupling capacitor into the Cremat CR-110 charge sensitive preamplifier. The net count rate observed at each temperature is provided in Table I, where it is evident that the diamond detector responded the same above the noise floor. These results demonstrate that a diamond detector can indeed operate as a multimodal detector for both temperature and radiation counting simultaneously.

While the work presented here only tests the multimodal diamond sensor during irradiation with alpha particles, it is reasonable to consider that similar results could be achieved under neutron irradiation. The neutron response to diamond has been well demonstrated, providing options for neutron spectroscopy and counting.^{30,48} The $^{12}\text{C}(n,\alpha)^9\text{Be}$ reaction results in a full energy peak that is well separated from other neutron reactions when the neutron source is monoenergetic. When the neutron source is polyenergetic, the output spectrum is a combination of elastic and inelastic reactions as a function of the incident neutron energy, resulting in little spectroscopic information without spectral unfolding. Furthermore, the lower energy region of the spectrum will likely be contaminated with gamma rays, but if the diamond is kept thin, then simple threshold discrimination could be used to remove the gamma ray contamination. Still, evaluation of the performance of a diamond-based fast neutron counter at 423 K exposed to 10^6 n/cm² s showed 90% stability and still performed well after 100 h of exposure.³⁴ However, even with the large 5.5 eV bandgap of diamond, experiments have shown that diamond suffers from a loss of spectroscopic capability at temperatures around 500 K.^{32,49–52} This is consistent with our results shown in Fig. 5, where a strong drift in the full energy peak location occurs when we reach a temperature of 473 K.

A recent study suggests that decreasing the detector thickness and varying the contacts may improve the spectroscopic performance at higher temperatures.⁵³ Here, they found that varying the electrode type has a slight effect on the response of the detector. Using double chromium contacts and a 100 μm thick diamond performed well up to 543 K, and double tungsten contacts on a 500 μm thick diamond resulted in a loss of spectroscopic performance around 510 K. Using chromium and a p-type diamond layer as the other contact, they

TABLE I. Observed count rate vs temperature of the diamond detector exposed to a ^{210}Po alpha source.

Temperature (K)	Count rate (s^{-1})
300	0.53
350	0.54
400	0.49
494	0.52

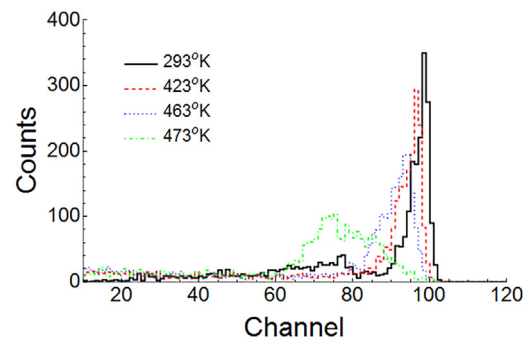


FIG. 5. Response of a diamond detector to ^{210}Po alpha particles at 300 and 473 K. The onset of spectral degradation is noticeable as the temperature approaches 500 K.

found that it performed well up to 600 K for a 100 μm thick diamond, but a 500 μm thick diamond lost its spectroscopic performance above 500 K. The authors attribute this to polarization effects, but polarization at the contact or within the bulk should be present regardless of the detector thickness, so further studies are needed to understand these observations. Another recent study has shown that a recovery in CCE begins at around 660 K, even after an initial reduction in CCE due to radiation damage induced by 5 MeV protons at fluences up to 10^{13} cm⁻².⁵² Although the mechanism of this recovery is not well understood, thermal de-trapping of charge carriers and defect annealing are suspected to play a key role. Further study of diamond sensor performance at higher temperatures and harder radiation environments, with regard to detector preparation schemes, is required to better understand the mechanisms behind their operational limits. Initial results on this recovery at higher temperatures are encouraging though, as it indicates that reliability and performance may be maintained at higher temperatures, even where damage due to fast neutrons is likely.

In summary, we have experimentally demonstrated that a diamond detector can be used as a multimodal device by acting as both a thermistor and a radiation detector at the same time. This was accomplished by measuring both the DC (thermistor) and AC (radiation-induced pulses) signals simultaneously. Changing the radiation source from alpha particles to neutrons should not result in a change in our conclusions given the extensive literature on diamond-based neutron sensing. Extending the multimodal diamond device for use in nuclear reactors requires additional investigation at higher temperatures, but its current operational capabilities may be sufficient for other applications such as well logging.⁵⁴ These investigations include understanding the effects of the sensor design to include thickness, substrate processing techniques, device architecture, and a precise understanding of the electrical properties and concentration of defects, all as a function of temperature. Finally, evaluation of the stability of diamond under intense neutron and gamma radiation fields at elevated temperatures is needed to understand its stability, lifetime, and ability to discriminate against low-energy deposition gamma interactions for operation at Gen IV and test reactors.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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