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Ag nanoparticle film-based gas sensors

Summary of findings for 2010

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Contract No. W7702-09R217

The scientific or technical validity of this Contract Report is entirely the responsibility of the Contractor and the contents do not necessarily have the approval or endorsement of Defence R&D Canada.

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Contract Report

DRDC Suffield CR 2011-014

September 2010

Canada

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Contract Authority: D. Pedersen, DRDC Suffield

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September 2010

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Abstract

This report summarizes the work done by Robert Scott's lab at the University of Saskatchewan as part of a contract to investigate the gas-sensing properties of Ag nanoparticle films prepared by Dr. Pedersen's lab (Soldier and Systems Protection Group) at DRDC Suffield, AB. Toby Bond was the researcher in the Scott group assigned to this project from its beginning in June of 2009 to the end of August 2010. This report covers results from Dec. 2009 to Aug. 2010.

Résumé

Le présent rapport constitue un résumé des travaux exécutés au sein du laboratoire de Robert Scott, à l'Université de la Saskatchewan, dans le cadre d'un contrat ayant pour objectif d'étudier les propriétés de détection de gaz de films de nanoparticules d'argent (Ag) préparés dans le laboratoire du groupe de protection des soldats et des systèmes de M. Pedersen (Ph.D.), à RDDC Suffield, en Alberta. Le chercheur scientifique du groupe de M. Scott attiré au projet, Toby Bond, en a assuré la direction dès le début, en juin 2009, jusqu'à la fin d'août 2010. Le présent rapport traite des résultats obtenus de décembre 2009 à août 2010.

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Executive summary

Ag Nanoparticle Film-Based Gas Sensors: Summary of Findings for 2010

Toby Bond; DRDC Suffield CR 2011-014; Defence R&D Canada – Suffield; September 2010.

Introduction or background: DRDC Suffield is actively researching and developing nanomaterials-based sensors for detection of toxic chemicals. A major aim of this effort is to develop personal exposure indicators that would warn a soldier of any respiratory hazard, as well as monitor the performance of protective equipment such as respirators. In this context, the sensors also serve as end-of-service life indicators for canisters that provide an indication when the canister capacity is exhausted. To be effective, the sensors must be able to detect toxic gases at low concentrations, before they cause toxicological effects that impair the soldier. To determine detection limits for chemical warfare agents and many toxic industrial chemicals, DRDC Suffield uses a static gas cell. The Scott group at the University of Saskatchewan, however, is able to measure detection limits for nanosensors under flow conditions, which are more realistic representations of the actual operational conditions. Accordingly, the Scott group has been contracted to study sensor performance under flow conditions.

Results: In its initial configuration, the flow system at the University of Saskatchewan was generating noisy data. To improve the system, electronic flow meters were installed with the effect of improving the signal to noise more than an order of magnitude. With the improved apparatus, nanosensors responses to water were then thoroughly studied. Exposure to relatively low concentrations of water was found to cause an increase in current. At exceptionally high concentrations, the water caused a decrease in current that sometimes irreversibly damaged the sensors. The detection limit for water was found to be 180 ppm. Preliminary results for HCl and NH₃ were also obtained but detection limits were not measured. The aging of sensors was also examined. Over a two week period, the sensors were very stable. During the first week following sensor fabrication at Suffield, the sensors exhibited a two order of magnitude increase in current flow through, likely due to the adsorption of trace amounts of water.

Significance: These initial results demonstrate the establishment of capability at the University of Saskatchewan to measure detection limits and other performance factors associated with nanosensor-type personal exposure indicators. This capability provides an independent verification of results generated in the Suffield labs. The detection limit measured for water is 180 ppm. This is 2 to 3 orders of magnitude higher than the detection limits measured by Suffield for a number of toxic gases. The results demonstrate a general insensitivity to water vapour, which is a desirable characteristic in sensors to be used in the field. The observed sensitivity to HCl also confirms results determined at Suffield. The aging study demonstrates that the sensors were extremely stable over a two week period.

Future plans: With capability established, the Scott group will now determine detection limits for a number of toxic gases of military relevance including ammonia and hydrogen chloride. The fundamental mechanism by which the nanosensors work will be elucidated. A comparison between the nanosensors and other state-of-the-art technologies, like metal oxide sensors, will be made to determine which technology is best as a personal exposure indicator.

Sommaire

Détecteurs de gaz à base de films de nanoparticules d'Ag

Toby Bond, Université de la Saskatchewan; RDDC Suffield CR 2011-014; R & D pour la défense Canada – Suffield.

Introduction : Les employés de RDDC Suffield exécutent des travaux de recherche importants sur l'étude et la mise au point de détecteurs à base de nanomatériaux ayant la capacité de détecter des produits chimiques toxiques. Les principaux objectifs de ces travaux comprennent la mise au point de dispositifs indicateurs d'exposition personnelle, qui permettraient d'avertir les soldats de tout danger de nature respiratoire et de surveiller le bon état de l'équipement de protection comme les appareils respiratoires. Les détecteurs peuvent aussi servir, dans ce domaine, d'indicateurs de la fin de durée de vie des boîtes filtrantes, lorsque la capacité de ces dernières est épuisée. Pour constituer des dispositifs efficaces, les détecteurs doivent pouvoir détecter de faibles concentrations de gaz toxiques, avant que ceux-ci aient des effets toxicologiques et nuisent à la santé des soldats. Dans les installations de RDDC Suffield, on utilise une cellule à gaz statique pour établir les limites de détection d'agents de guerre chimiques et de nombreux produits industriels toxiques. Toutefois, les membres du groupe de recherche de M. Scott, à l'Université de la Saskatchewan, ont la capacité de mesurer les limites de détection au moyen de nanodétecteurs, dans des conditions d'écoulement dynamique, lesquelles correspondent plus fidèlement aux conditions de fonctionnement réelles. C'est pourquoi un contrat a été conclu avec le groupe de recherche de M. Scott afin qu'on y étudie la performance des détecteurs dans des conditions d'écoulement dynamique.

Résultats : La configuration initiale du dispositif d'écoulement dynamique utilisé à l'Université de la Saskatchewan produisait des données comportant un bruit important. Afin d'améliorer son efficacité, on a installé des débitmètres électroniques, ce qui a permis d'accroître le rapport signal/bruit par plus d'un ordre de grandeur. On a ensuite utilisé le dispositif amélioré pour réaliser une étude détaillée de la réaction à l'eau des nanodétecteurs. Il a été établi que leur exposition à des concentrations d'eau relativement faibles provoque une hausse de courant. D'autre part, des concentrations d'eau exceptionnellement élevées provoquent une diminution du courant, ce qui entraîne, dans certains cas, la détérioration irréversible des détecteurs. La limite de détection établie pour l'eau est de 180 ppm. Des résultats préliminaires ont aussi été obtenus pour HCl et NH₃, mais leurs limites de détection n'ont encore été déterminées. On a en outre étudié le vieillissement des détecteurs. Ceux-ci sont très stables sur une période de deux semaines. Durant la première semaine suivant leur fabrication dans les installations de RDDC Suffield, les détecteurs présentent une augmentation de deux ordres de grandeur du débit de courant, laquelle est probablement attribuable à l'adsorption de quantités d'eau à l'état de traces.

Portée : Les résultats initiaux présentés démontrent la capacité des installations de l'Université de la Saskatchewan de mesurer des limites de détection fiables ainsi que d'autres facteurs de performance propres aux indicateurs d'exposition personnelle du type nanodétecteurs. L'utilisation de ces capacités permet d'effectuer une vérification indépendante des résultats obtenus dans les laboratoires de RDDC Suffield. La limite de détection établie pour l'eau est de 180 ppm, une valeur qui est de deux à trois ordres de grandeur plus élevée que celles des limites de détection mesurées dans les installations de Suffield pour un certain nombre de gaz toxiques. Les résultats indiquent que les détecteurs sont généralement insensibles à la présence de vapeur

d'eau, ce qui constitue une caractéristique recherchée pour tout dispositif de ce type utilisé sur le terrain. Les observations relatives à la sensibilité des détecteurs au HCl confirment aussi la justesse des résultats obtenus dans les installations de Suffield. Les résultats de l'étude de vieillissement des détecteurs confirment que ceux-ci sont très stables sur une période de deux semaines.

Recherches futures : Ses capacités de recherche de pointe étant maintenant reconnues, les membres du groupe de M. Scott peuvent maintenant entreprendre la détermination des limites de détection d'un certain nombre de gaz toxiques d'intérêt militaire, y compris celles de l'ammoniac et du chlorure d'hydrogène. L'exécution des travaux permettra d'élucider le mécanisme de fonctionnement fondamental des nanodétecteurs. On comparera aussi la performance des nanodétecteurs et celle d'autres technologies de pointe comme celle des détecteurs à base d'oxydes métalliques, afin de déterminer laquelle est la plus adéquate dans le domaine des détecteurs d'exposition personnelle.

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Introduction

This report summarizes the work done by Robert Scott's lab at the University of Saskatchewan as part of a long-term contract to investigate the gas-sensing properties of Ag nanoparticle films prepared by David Pedersen's lab (Soldier and Systems Protection Group) at Suffield, AB. Toby Bond was the researcher in the Scott group assigned to this project from its beginning in June of 2009 to the end of August 2010. This report covers results from Dec. 2009 to Aug. 2010 (a report describing the initial results is also available). A great deal of data is omitted from this report for the sake of clarity, but all the raw data, as well as more detailed reports that were submitted throughout the year, are available from the Scott lab upon request.

Background

The first months of the project were used mostly to determine appropriate storage and testing conditions for the sensors and demonstrate basic sensor responses to hexane, ethanol, and water vapour. The response is measured by applying a potential (usually 1 V) to the film and monitoring the current over time as the sensor is exposed to analyte gas. The concentration of analyte gas is controlled using 2 flow controllers: a clean channel with pure carrier gas (usually nitrogen) and an analyte channel with saturated vapour from a bubbler or a tank containing analyte gas (fig. 1).

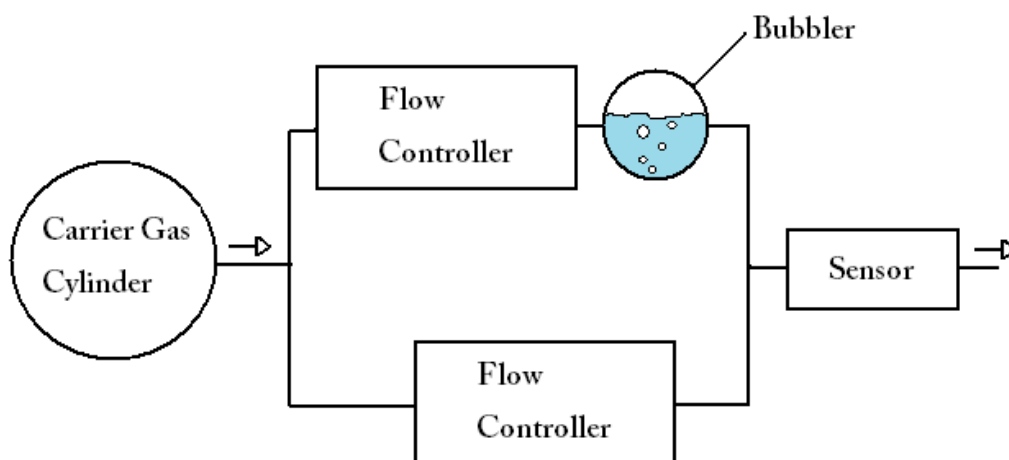


Figure 1: Schematic of flow apparatus.

A drop in current was observed for hexane and ethanol vapour, while a delayed increase in current was observed for water vapour. Reproducibility, signal drift, and signal noise were significant issues encountered during the initial testing. After trying a variety of storage conditions and carrier gases, it was found that the sensors were only stable under dry nitrogen

(both for storage and as a carrier gas). Even under these conditions, only some sensors (generally those that were less conductive – ie. lower particle density) yielded reversible gas sensing responses. The report from 2009 (mentioned previously) details these results as well as the apparatus used. Papers published by Pedersen *et al* describe the synthesis and characterization of the nanoparticle films used.

Experimental (upgrades to flow apparatus)

The previous flow apparatus employed analogue flow controllers which (fig 2a) had limited accuracy and had to be adjusted manually. Manual operation of the meter was somewhat cumbersome and required 30-40 seconds to adjust the meters to the desired analyte level. To overcome these limitations, considerable time was invested in building an apparatus using digital flow controllers which were both controlled simultaneously from a computer. The flow controllers used were two Smart Trak 2 Digital Flow Controllers from Sierra Instruments (fig. 2b).

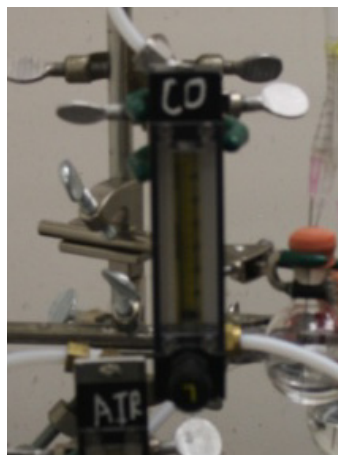


Figure 2: a) Analogue flow controller used in previous experiments
b) Smart Trak 2 Digital Flow Controller used in experiments from April 2010 onward.

The Smart Trak 2 is operated by setting a desired flow rate, or “setpoint,” which is achieved using a feedback system in the controller. The setpoint will usually be reached with high precision (± 0.01 scc/min) within 4 seconds, up to a maximum flow rate of 265 scc/min. The precision of the analogue flow controllers was approximately ± 8 scc/min. The controllers can be remotely operated from a PC using an RS-232 interface. Software was included with the controllers for this purpose; however it lacked two important features: the ability to operate more than one controller simultaneously, and the ability to save flow data recorded by the controller. To this end, we decided to write our own software using LabView – a graphical programming environment specifically designed for hardware interfacing and data acquisition. After several weeks of programming and debugging, a working program was successfully implemented which simultaneously operates both flow controllers and stores the collected flow data on the hard drive (see fig 3).

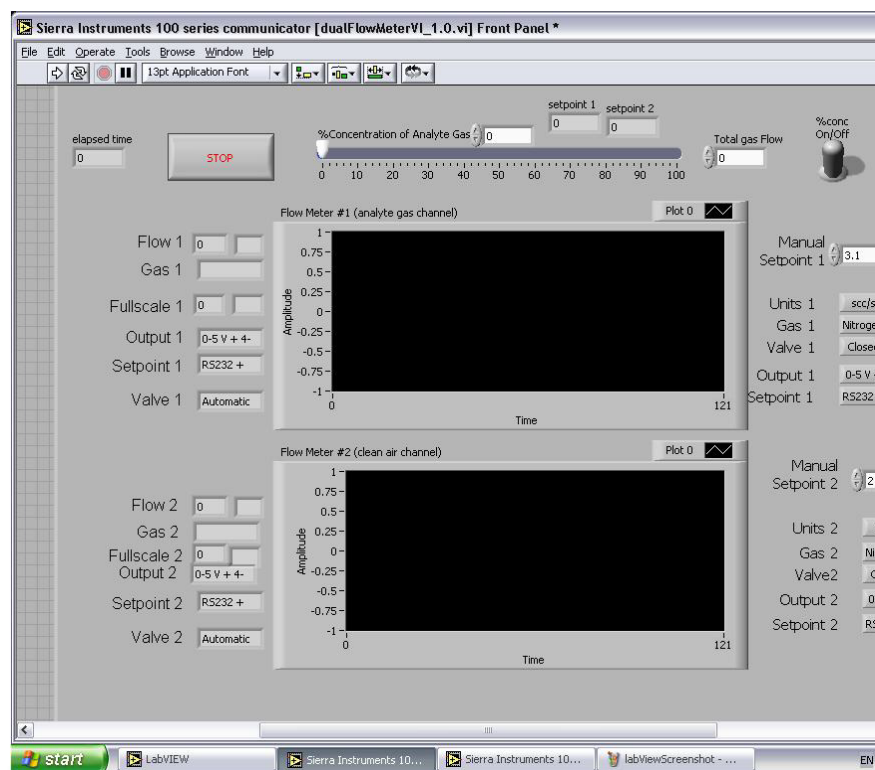


Figure 3: Graphical User Interface of in-house Flow Controller Software.

This data is displayed on the same graphs as the response data from April onward. To run the software, the user inputs a total flow rate that is maintained (which is simply the sum of the flow rates of the two controllers) and sets the %-concentration of analyte gas to be injected. The software then calculates the two setpoints based on these values and sends them to the controllers, which automatically adjust to their respective setpoints simultaneously. Although the program is fully operational, it still contains one bug which has eluded attempts to fix it, where certain setpoints (namely, at 80 scc/min and 120 scc/min) will cause the flow rate to drop to zero. A temporary workaround to this problem is to use setpoints that are 0.1 scc/min above or below these faulty setpoints, but it is hoped that a solution may still be found to this problem. In all other respects though, the apparatus functions with accuracy and injection times which are improved by orders of magnitude compared to the analogue setup.

Results and discussion

Although initial results have been obtained for hexane, ethanol, and water vapour, we decided to focus on the response to water vapour in an effort to demonstrate reproducible results and to obtain a calibration curve and detection limits for at least one analyte. Though much of the actual testing focused on water, some preliminary results were also obtained for ammonia, aqueous hydrochloric acid vapour, and hydrogen chloride gas.

Since the switch to ultrapure nitrogen as a carrier gas and atmosphere for storage, baseline currents have been relatively stable under storage. Table 1 shows the baselines of a batch of sensors from April during the testing period. A baseline signal is no longer recorded after the sensor has been exposed to analyte (which often irreversibly alters the baseline).

Table 1: Baseline currents of Ag nanoparticle films stored under ultrapure nitrogen atmosphere (baseline not measured after exposure to analyte).

Sensor number	Baseline current @ 1V - before shipping (A)	april 13	April 14	April 15	April 17	April 18	April 21	April 22	April 25
1	6.0×10^{-7}	4.37×10^{-5}	3.18×10^{-5}						
2	9.5×10^{-8}	1.39×10^{-4}	1.36×10^{-4}	1.36×10^{-4}					
3	8.0×10^{-7}	1.09×10^{-4}	1.08×10^{-4}	1.12×10^{-4}	1.14×10^{-4}				
4	2.0×10^{-6}	3.40×10^{-4}	3.57×10^{-5}	3.65×10^{-4}	3.70×10^{-4}	4.04×10^{-4}	3.98×10^{-4}	4.14×10^{-4}	
6	4.0×10^{-7}	6.02×10^{-5}	1.00×10^{-4}	1.00×10^{-4}	1.01×10^{-4}	1.02×10^{-4}	1.01×10^{-4}	1.03×10^{-4}	1.03×10^{-4}

The baseline drift observed during initial experiments is also no longer observed, making calibration more feasible. Despite the stability in baselines, the sensors still cease to yield a response to analyte gasses after 1-2 weeks (even when exposed only to nitrogen atmosphere).

Response to water vapour

It has been known since early on in the project that the sensors are sensitive to moisture (which likely contributed to the signal drift observed initially). We decided to focus our attention on water vapour in the hope of obtaining more consistent, reproducible results for a single system. Water vapour turned out to be a fairly elusive analyte, giving seemingly conflicting responses with different batches of sensors. Many tests indicated that the response of water was an increase

in current, which was the opposite response of all other analytes tested to date. This increase was characterized by delayed, terraced, and unpredictable spikes in current, a typical example of which is given in Fig. 4.

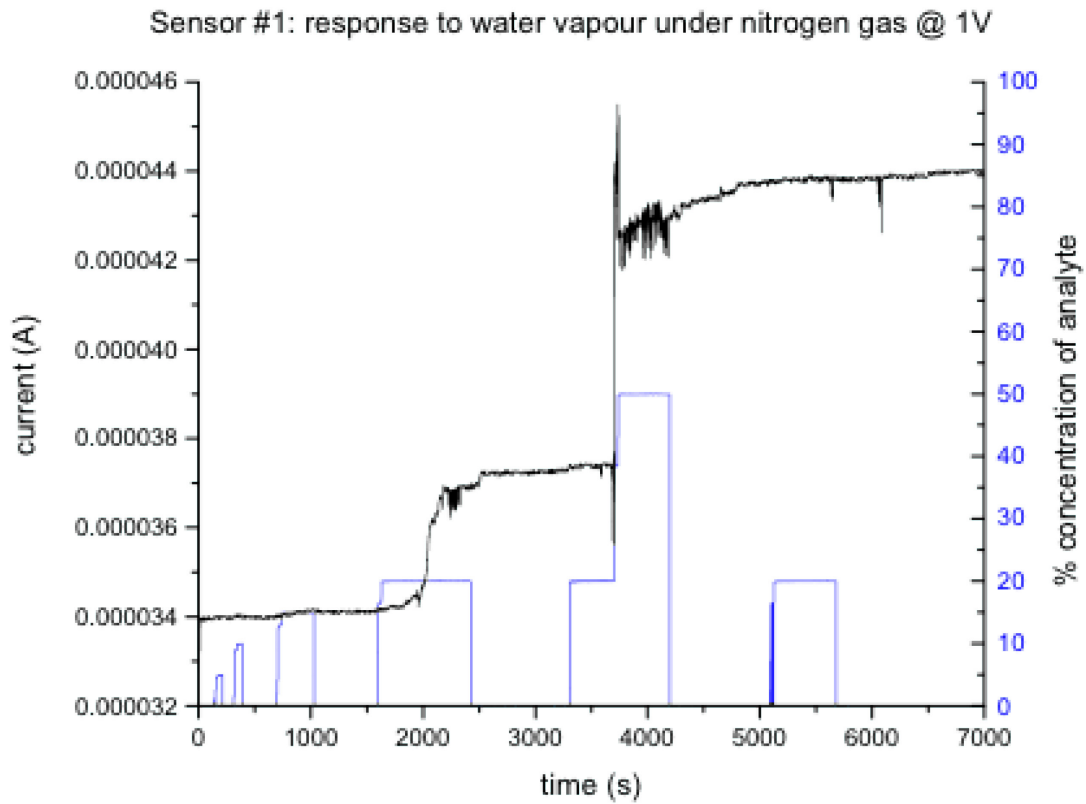


Figure 4: Typical erratic response of sensor to water vapour.

Toward the end of a given sensor's testing life, this increase would no longer be observed, and eventually the current began to decrease as water vapour was applied which continued until the sample was no longer conductive within our detection limits ($I < 0.14 \text{ nA}$). It was found in later experiments that this increase in current is cumulative at fairly low concentrations of water: In one experiment (fig. 5) a sensor was exposed to a constant concentration of water vapour (10% of saturation) until the current reached a plateau. When the water concentration was increased, the drop in current was observed which leveled out when the water was shut off.

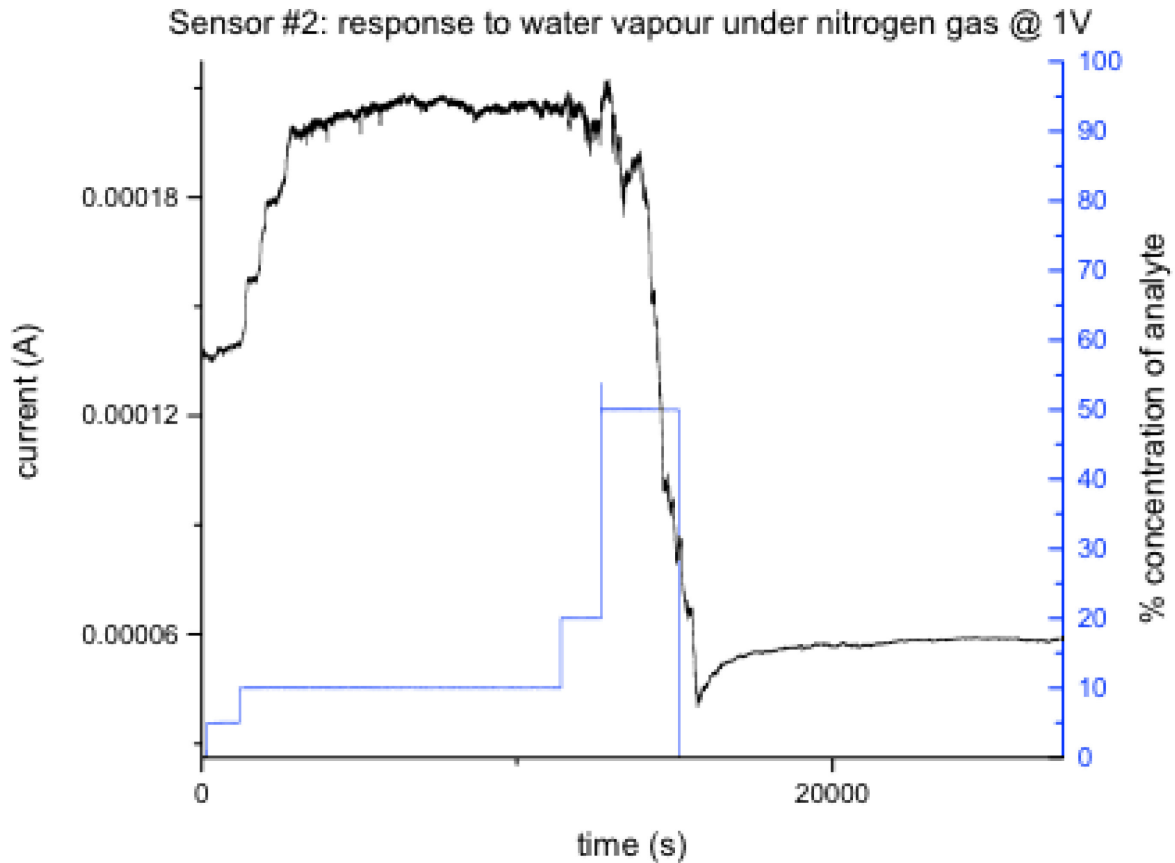


Figure 5: Rise and fall of current with constant exposure to water vapour.

Although all sensors displayed this type of response eventually, some sensors (mostly those with lower particle density) demonstrated a different type of response initially, where the current dropped in response to applied water vapour, before the rise in current. In June of this year, one sensor in particular (which was low-density, having an initial baseline current of 3.61×10^{-6} A) produced this type of response reproducibly enough that it was used to make a 4-point calibration curve, the raw data for which is shown in Fig. 6.

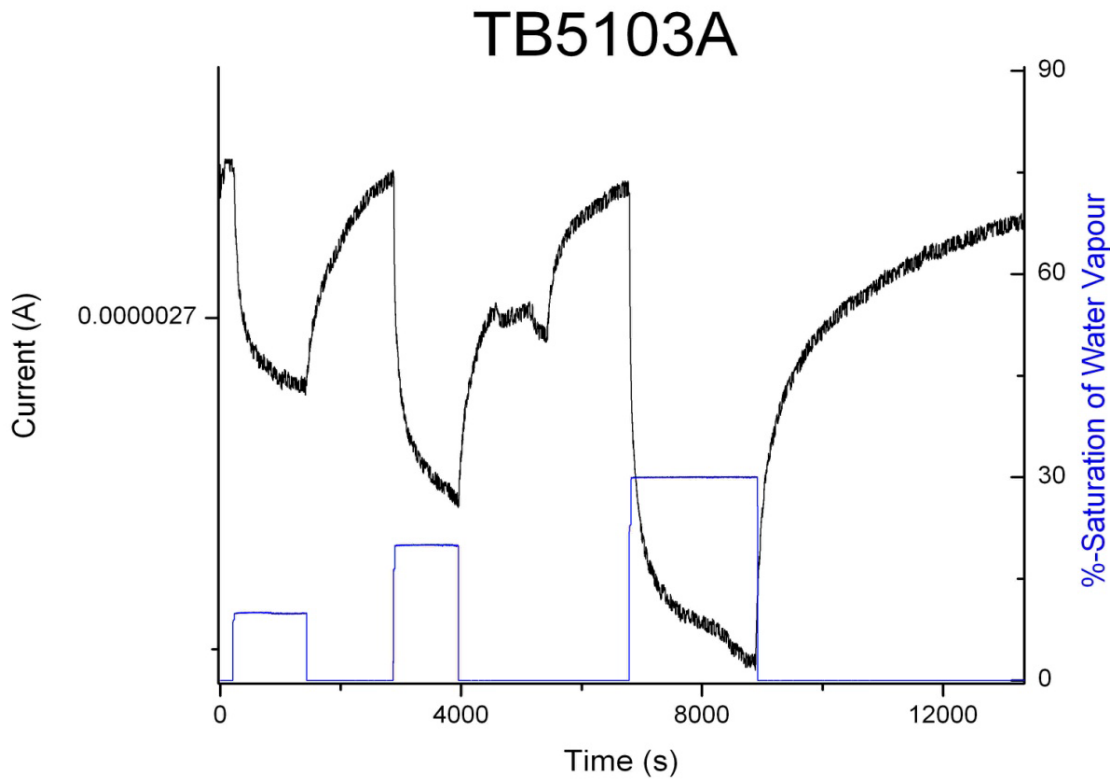


Figure 6: Reversible response to water vapour used for calibration.

After this calibration was performed, the sensor was accidentally exposed to 100% saturated water (due to the previously mentioned software/hardware bug) which caused an abrupt drop and subsequent spike in current. After drying under nitrogen flow for several hours, a reproduction of the 10% measurement was attempted, which did result in a drop in current, but the subsequent recovery exhibited the rise-and-fall behaviour observed in previous experiments (fig. 7).

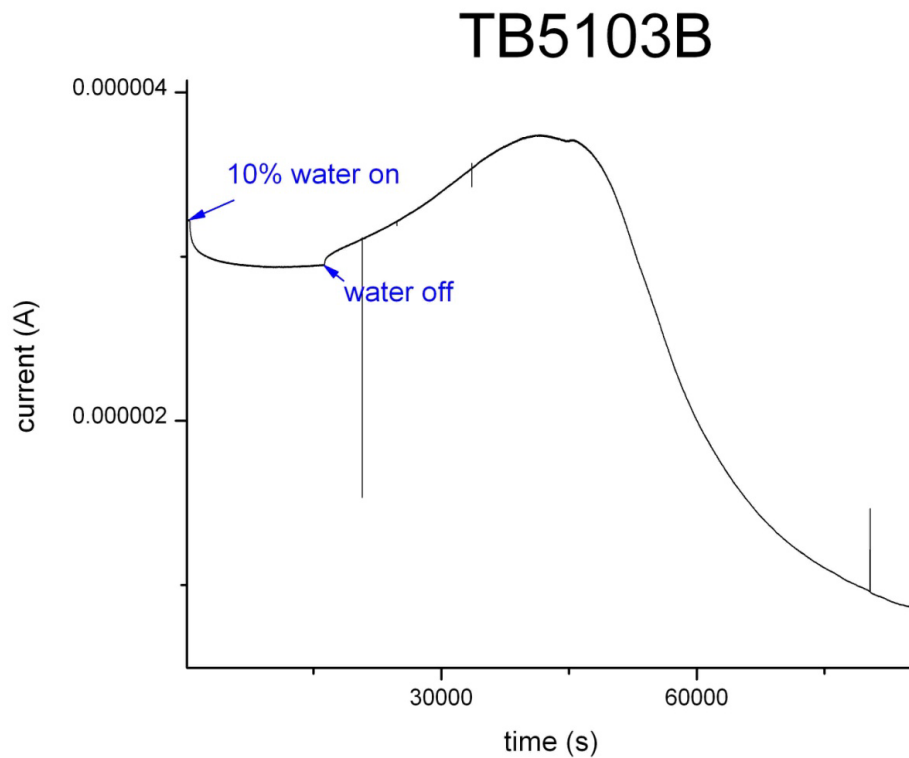


Figure 7: Rise and fall of signal from sensor used to make calibration curve (after calibration data was collected).

The current continued to drop until the sensor was no longer conductive. This seems to suggest that lower-particle-density sensors do indeed respond in a reversible manner to water vapour until the sensor is rendered inoperative by whatever processes cause this rise-and-fall response to occur.

In order to construct a calibration curve for water vapour, we measured the response as the change in current as a percentage of the initial baseline ($\Delta I/I_0$) and plotted it against the mole fraction of water (calculated by multiplying the mole fraction of saturated water at 21 C by the flow rate fraction of the water vapour channel). The resulting data is given in table 2.

Table 2: Signal and concentration data used for calibration curve.

%-saturation of water	Mole% of water (@ 21 C)	I_0 (A)	Response I (A)	ΔI (A)	%-change ($\Delta I / I_0$)
10	0.2461	2.766×10^{-6}	2.672×10^{-6}	9.941×10^{-8}	3.40
20	0.4922	2.765×10^{-6}	2.615×10^{-6}	1.150×10^{-7}	5.42
30	0.7383	2.761×10^{-6}	2.544×10^{-6}	2.175×10^{-7}	7.88

The resulting plot shows a linear calibration curve (including a current change of zero for the absence of water) with little error (fig. 8).

Calibration Curve of Water Vapour Response for Sensor #3

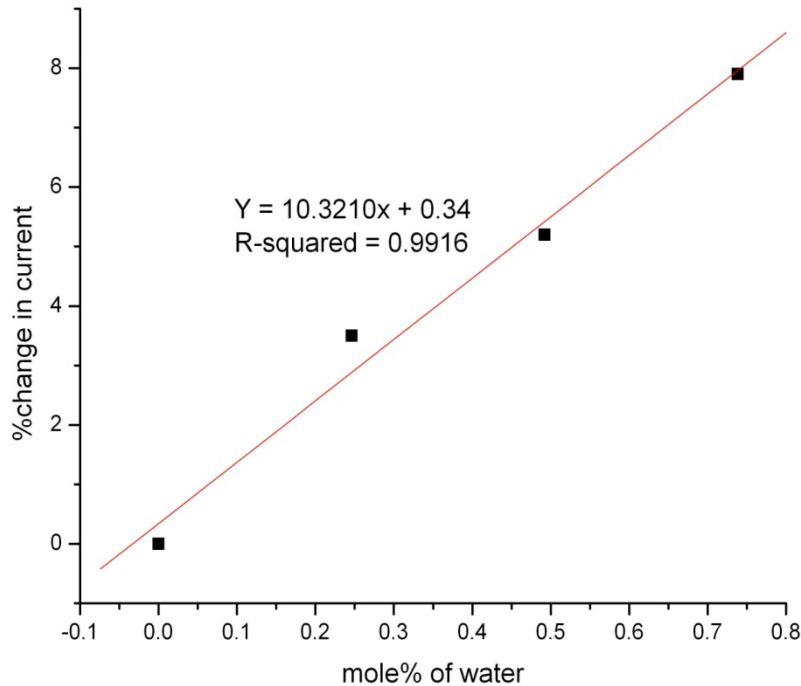


Figure 8: Calibration curve of water vapour.

The noise in this particular experiment has an outer range of 4.862×10^{-9} A, which corresponds to 0.1758 % of the initial baseline (2.766×10^{-6} A). This can be used to estimate the detection limit by assuming that the limit is about 3 times the noise range. Using the calibration curve, the detection limit for water under these conditions is approximately 180 ppm.

Hydrogen chloride and ammonia gas

Although most of the sensors were used up in tests with water, some initial tests were also run with aqueous hydrochloric acid vapour, dry hydrogen chloride gas, and ammonia gas. The concentration of the hydrogen chloride and ammonia gasses was adjusted using a similar setup as shown in the experimental section, but the analyte channel consisted of a cylinder of analyte hooked up directly to one flow controller (which was then mixed with nitrogen flow from the clean channel). The hydrochloric acid vapour was injected using the same method used for other vapours.

In general, both the aqueous hydrochloric acid and hydrogen chloride gas showed a similar rise and fall in current observed with water, but on a much smaller timescale (especially for hydrogen chloride). The response to hydrochloric acid vapour is shown in fig. 9.

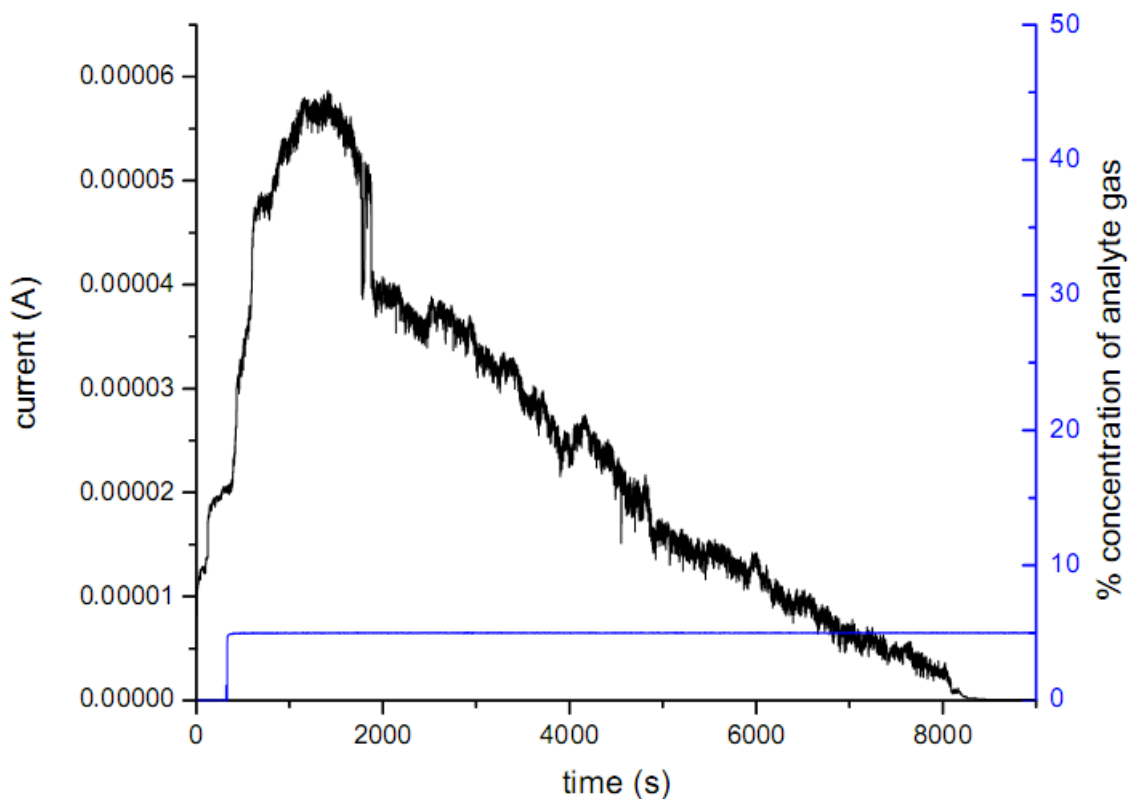


Figure 9: Response to aqueous hydrochloric acid vapour.

The noise in these experiments is markedly higher compared to the response for water (possibly due to oxidation of the film by chloride during the experiment). The response to HCl gas is similar, but much more rapid (fig. 10).

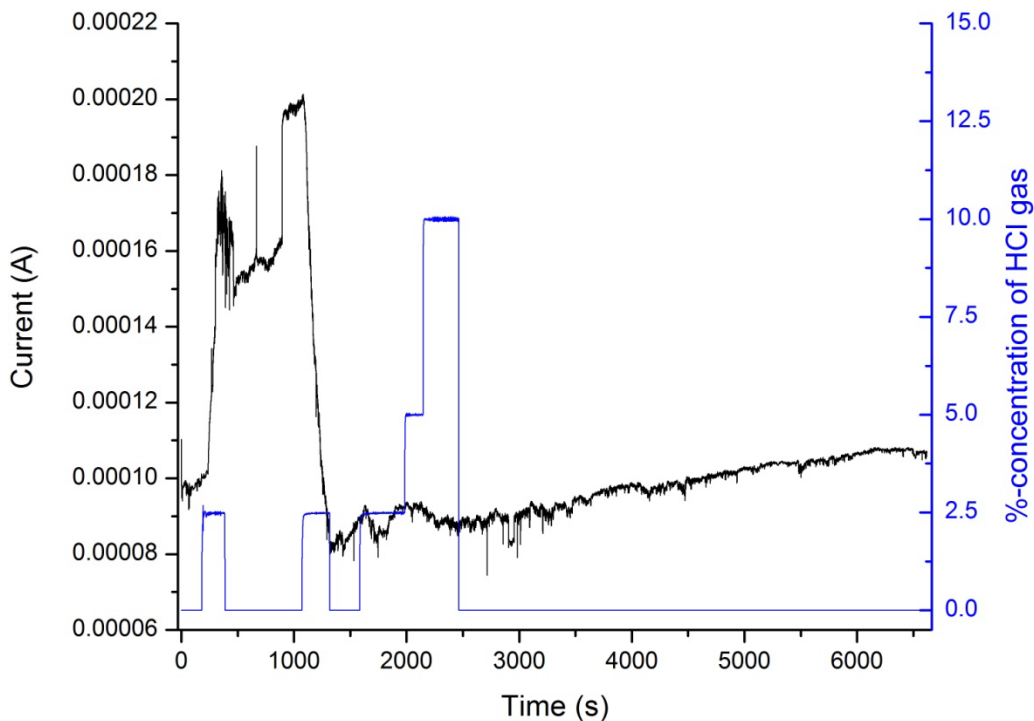


Figure 10: Response of the hydrogen chloride gas.

It is interesting to note that the current did not immediately drop to zero after the rise and fall in signal, but simply stopped responding to further injections of HCl gas. After this experiment, the sensor was re-examined after 5 minutes and did show zero conductivity.

Ammonia gas was tested once with a fairly high-conductivity sensor (with a baseline of 0.5 mA) which did not show any response even at the highest allowable concentration (1000 ppm – the concentration of ammonia gas in the analyte cylinder). This result should not be taken as conclusive, as a lower particle density sensor could still yield a response, but there is nothing to report for this initial experiment.

Conclusions and future work

Although many sensors did not exhibit a useful response to water vapour, sensors with sufficiently low particle density do respond to water vapour with a drop in current which is reversible until sensor function is lost, at which point there is a rise in current with a subsequent drop to zero conductivity. This reversible response can be calibrated and shows a linear relationship between the drop in current (relative to the baseline) and mole fraction of water vapour present in the cell. Using this information, the detection limit for water of the film tested is estimated to be 180 ppm.

Hydrochloric acid and HCl gas both show a comparatively rapid increase and decrease of current. This effect, observed for both water and HCl, is likely due to different processes governing the respective increase and decrease in current. For example, the increase could be due to a change of the dielectric constant between barriers while the decrease could be due to structural rearrangement or oxidation of the film (formation of silver oxide or silver chloride). A single experiment with ammonia gas has shown no response up to 1000 ppm, but it is still possible that a sensor with lower particle density could show a response (as has been observed with other analytes in the past).

Using digital flow meters has allowed us to see much more clearly when there is a delay between the injection of analyte and the observed response, as well as perform calibration with significantly greater accuracy. Sensor responses are still too unpredictable at this point to use automated injection to test the sensors without human intervention, but if the sensors can be made to respond more predictably, higher throughput could be achieved by adding automation features to the software, allowing testing and calibration to occur overnight without supervision.

Experiments to be performed in the near future include reproduction of the calibration curve using another low particle density sensor for comparison, performing a calibration with another analyte such as hexane, (which has shown a proportional response in the past) and testing of the sensors with new gases, including repeated tests with ammonia. Since the lower particle density films appear to respond much better, investigating in greater detail the relationship between particle density and detection limits should also be one of the long-term goals of the project.

An effort was made to set up a cell to test other types of sensors (such as metal oxides) that can be compared with these films under the same conditions. Unfortunately, problems with one of the flow controllers did not allow for this to be completed before the end of the summer, but this will be one of the next tasks to be performed by our group.

Despite initial difficulties with reproducibility, the results from this past year have been encouraging, and it will be interesting indeed to see if such a linear calibration curve can be produced for other analytes. Although this project is still at the stage of basic research, the potential for these films as novel gas sensors is certainly promising.

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This report summarizes the work done by Robert Scott's lab at the University of Saskatchewan as part of a contract to investigate the gas-sensing properties of Ag nanoparticle films prepared by Dr. Pedersen's lab (Soldier and Systems Protection Group) at DRDC Suffield, AB. Toby Bond was the researcher in the Scott group assigned to this project from its beginning in June of 2009 to the end of August 2010. This report covers results from Dec. 2009 to Aug. 2010.

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nanosensors, nanoparticles, sensors, gas sensors, hydrogen chloride

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