SODIUM IONIZATION DETECTOR AND SENSOR

Final Technical Report

May 8, 1979

J. Hrizo and J. E. Bauerle
Materials Chemistry

Westinghouse Research and Development Center
Pittsburgh, Pennsylvania 15235

Contract EN-77-C-02-4197.A000

Department of Energy

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ABSTRACT

This report presents the work conducted on a basic technology development effort with the Westinghouse Sodium Ionization Detector (SID) sensor. Included are results obtained for three task areas:

- (1) On-line operational response testing - in-situ calibration techniques;
- (2) Performance-reliability characteristics of aged filaments; and
- (3) Evaluation of chemical interference effects. The results showed that a calibrator filament coated with a sodium compound, when activated, does supply the necessary sodium atoms to provide a valid operational in-situ test. The life time of new Cr_2O_3 -protected SID sensor filaments can be extended by operating at a reduced temperature. However, there also is a reduction in the sensitivity. Non-sodium species, such as products from a smoldering fire and organic aerosols, produce an interference response from the sensor comparable to a typical sodium response.

1. INTRODUCTION

This report describes the work performed on a DOE-funded program that involves basic technology studies for the Westinghouse Sodium Ionization Detector (SID) sensor. The SID sensor was designed to be used as a small, sensitive, sodium-to-gas leak detector for the Breeder Reactor Program.⁽¹⁾ Its performance in comparative testing with other sensors at the Liquid Metal Engineering Center (LMEC) has been excellent, and the SID has become a prime candidate for the sodium-to-gas leak detection system.⁽²⁾

The theory of the detector's operation, design, readout, and results of detector sensor tests have been reported;⁽³⁾ however, certain feasibility and instrument acceptability questions arose. The most important of these served as the basis for this program. Three tasks focusing on the sensor were performed:

Task 1: On-Line Operational Response Testing: In-Situ
Calibration Technique

Task 2: Performance-Reliability Characteristics of Aged
Filaments

Task 3: Evaluation of Chemical Interference Effects

Each task is discussed individually in the following sections of this report.

2. CONCLUSIONS

2.1 General

The program has met its technical objectives and performance on all tasks was attained. The performance of the Sodium Ionization Detector (SID) sensor indicates that it will serve as a practical instrumentation choice for sodium-to-gas leak detection. Specific conclusions for each of the program tasks follow.

Task 1: On-Line Operational Reponse Testing: In-Situ Calibration Technique

- A modified calibrator filament, coated with a sodium compound and located adjacent to the SID sensor, is capable of verifying the operational response of the detector sensor.
- Tests of the calibrator filament geometry configuration have shown that a helical Cr_2O_3 -protected filament or a straight SiO_2 -protected filament provide a suitable amount of sodium for verifying SID response.

Task 2: Performance-Reliability Characteristics of Aged Filaments

- Filament life tests show that the SiO_2 -protected material provides the best lifetimes.
- Lower temperature operation of the filaments extended the filament lifetime. However, the reduced thermal ionization efficiency decreased the sensitivity noticeably.

- Sodium sensitivity performance of aged SID filaments shows that the SiO_2 -protected filament material retains its sodium detection capability throughout the lifetime of the filament.

Task 3: Evaluation of Chemical Interference Effects

- Response tests of the SID sensor to dust species, for example concrete, soil, or insulation, did not produce any noticeable effect on the sensor signal. This lack of an effect may indicate the particulate size was too large and settling had occurred.
- An interference response of the SID sensor was produced by the smoke products from a smoldering fire. Also, aerosols of lubricating oils or electrical insulation oil produced an interference effect whereas the vapors of these liquids did not produce any noticeable signal.

3. TASK 1: ON-LINE OPERATIONAL RESPONSE TESTING - IN-SITU CALIBRATION TECHNIQUE

3.1 Task Objective

Develop a technique capable of confirming, upon command, that a remote, functioning, sodium ionization detector (SID) sensor is yielding the proper operational response. This technique should, if possible, also provide means of in-situ calibration of the sensor signal.

3.2 Calibrator Filament Geometry

The simplest form of an in-situ "tester-calibrator" would be a heatable filament coated with a sodium compound whose vapor pressure could be elevated by heating. A calibrator filament parallel to the SID sensor filament would accomplish this. Several possible SID sensor calibrator filament design variations are presented in Fig. 1.

A single straight wire calibrator filament (Fig. 1a) of the chromium oxide protected type material, coated with a sodium compound, was not effective in producing the required sodium signal. The available surface area of this straight wire design limited the quantity of sodium compound that could be applied.

A porous sheath design calibrator filament was constructed (Fig. 1c), consisting of a refractory tube placed over a thicker filament of the same Cr_2O_3 -protected material. This calibrator filament did provide the needed sodium supply for effective operation. However, the refractory nature of the sheath material required a higher operating temperature of the filament; this reduced its lifetime to only two hours.

A helical design for the calibrator filament (Fig. 1b) produced better results. This geometry had the advantage of increased surface area for retention of the applied sodium compound.

An SiO_2 -protected filament material was considered as the prime candidate for evaluation as the calibrator filament. This decision was prompted by successful longevity tests of this material as the SID sensor operating filament. However, due to the brittle nature of the SiO_2 -protected material, only the straight design could be tested. The silica (SiO_2) protective cover on this filament, formed during normal hot filament operation in an air atmosphere, resulted in a nonporous refractory sheath design.

3.3 In-Situ Tester-Calibrator Performance

The experimental apparatus for in-situ test-calibration of the sodium ionization detector (SID) sensor is shown schematically in Fig. 2. A conventional SID sensor unit was employed with a separately mounted calibrator filament. These were mounted on opposite sides of the detector housing. The detector and calibrator units have independent power supplies for heating and bias potentials.

The response of the SID sensor to a sodium signal generated by the Cr_2O_3 -protected helical design calibrator filament is shown in Fig. 3. The calibrator filament was tested both with and without a bias potential. The left curve was obtained with negative bias applied to the calibrator filament. The center curve depicts the response of the SID sensor to an initial negative bias of the calibrator filament with polarity changed to positive bias and then changed again to negative bias. The step increase in signal response of the SID sensor to a positive bias condition corresponded to an increase in the number of positive sodium ions generated by the calibrator filament. Upon returning the potential to a negative bias condition, it was observed that the SID sensor rapidly recovered to the initial signal. The third curve on the right of the figure shows the SID sensor response to an unbiased calibrator filament. The maximum signal response for this situation was intermediate to those obtained for negative and positive bias conditions. This displayed the generation of both positive and neutral sodium ions from the filament.

The SID sensor filament was normally operational throughout the test series, with the calibrator filament being activated to produce the sodium signal. A more critical test was performed by turning off the SID filament in order to simulate a sensor filament failure. The calibrator filament was operated under various bias potentials. These conditions are shown in Fig. 4. The curve on the left of the figure shows a slightly positive signal response, much reduced from the background signal, however, with no bias potential (0 volts) applied to the calibrator filament. The center curve depicts the essentially zero signal resulting when negative bias (-100 volts) was applied to the calibrator filament. The third curve on the right of the figure shows the undesirable condition of an apparently operational SID sensor when the calibrator filament has positive potential (+100 volts), although the SID sensor had an inoperative filament.

The helical design calibrator filament had a lifetime of about 65 hours for the operating conditions employed. Although this may seem to be of short duration, it should be kept in mind that the calibrator filament would only be used intermittently to provide a sodium source in order to verify that individual SID sensors were functioning.

It was considered that the SID sensor, during its lifetime, may be cleaned by a water and/or organic solvent wash following some test situations. This procedure could remove the sodium compound (needed to perform the on-line response test) from the calibrator filament. To test this possibility, the filament was washed with deionized water and then with acetone. Although the sodium compound (a water soluble material) had been evenly distributed over the calibrator filament upon application, a migration of the material occurred during response testing. This resulted in a material build up at the ends of the coiled Cr_2O_3 -protected filament. A flowing water stream did not remove this deposit. One filament failed by mechanical fracture when the filament was activated. The failure occurred at the coil termination where the material buildup had taken place. A second filament, although

not suffering failure, did not provide a signal during the on-line response test after the washing procedure. The sodium necessary for signal generation had been depleted from the central hot region of the activated calibrator filament.

The successful longevity of the SiO_2 -protected material used as the SID filament prompted an evaluation of it as candidate material for the calibrator filament. An SiO_2 -protected filament was mounted in the experimental apparatus as the calibrator filament. Although this material has an initially-high sodium content from the manufacturing process, it was coated with an additional sodium compound to ensure an adequate supply for calibrations.

The response of the SID sensor filament to the SiO_2 -protected calibrator filament is shown in Fig. 5. It is seen that with positive bias (+100 volt) applied to the calibrator, the magnitude of the signal response is an order of magnitude greater than the signal produced when there is no bias (0 volt) potential. It also is interesting to note what happened when the SID sensor filament was deactivated the last five minutes of the test in order to simulate a SID filament failure. There was no apparent change in the sensor signal when the SID filament was off. The signal spike at the end of the trace was a result of reactivating the SID filament prior to turning off the calibrator filament. This spike resulted from the rapid volatilization of the sodium, deposited on the filament during the period it was off (cold), when the filament was reheated. By contrast, when there was no bias potential applied to the calibrator filament, the response of the sensor went essentially to zero signal with the SID filament rendered inoperative. When the SiO_2 -protected calibrator filament was operated under negative bias, a copious quantity of electrons was generated. This had a pronounced effect on the SID sensor by driving the normal positive ion current signal to negative electronic current signal, resulting in the instrument panel readout meter moving off scale, below zero.

The washing procedure was performed on the SiO_2 -protected filament. A deposit remained upon the filament after this treatment.

This filament fractured when reactivated. The failure is believed to be due to insufficient drying of the filament after the washing procedure.

The reduced signal generated after the calibrator filaments had received a wash treatment suggests that an additional supply of sodium-bearing material should be applied to the calibrator filaments in the event of their contact with any water.

4. TASK 2: PERFORMANCE-RELIABILITY CHARACTERISTICS OF AGED FILAMENTS

4.1 Task Objective

Determine the functional performance characteristics of aged sodium ionization detector (SID) filaments. Also, develop methods for increasing the likelihood of their reliable long term operation.

4.2 Filament Material Lifetime Testing

An operating SID sensor filament undergoes changes in its physical and chemical properties with time as a result of being maintained at a high temperature. Material is continually being vaporized from its surface, and the protective surface oxide layer grows at the expense of underlying filament material. Moreover, the filament becomes more fragile with age.

A historical review of previous SID filament failures has indicated that two modes of failure occur: (1) normal, long-life failure and (2) early life failure. The normal failure mode is characterized by the uniformly slow attrition of filament material. This condition eventually leads to a "hot spot" situation. The "hot spot" location is usually near the central section of the filament where the temperature is the highest, and where the filament fails.

The second type of failure, early life failure, occurs at a flaw (surface scratch, impurity, or inclusion) which raises the local filament temperature at that location. Growth of the flaw is accelerated by the high temperature and leads to an early failure. The failure site may be located any place along the filament. (During the period of this study, SID sensor filament failures were reported by personnel of Energy Technology Engineering Center (ETEC). The time to failure for two filaments were three and twelve months. See Appendix A for this discussion).

The filament lifetest system has been described in a previous study.⁽³⁾ The test system makes use of individual power supplies (A.C.) for each filament so that they may be adjusted for temperature independently. The filament holders and gas enclosures were constructed of Pyrex. A vacuum pumping system was used to maintain flowing room air around the filaments.

The filament materials were tested at 1100°C (200°C above normal temperature) in flowing air. However, a number of filament failures occurred during these accelerated life tests. Hence, the operating temperature of the remaining filaments was reduced from 1100°C to 900°C in order to evaluate the sodium sensitivity response of the aged filaments.

The lifetime data obtained for the filament materials tested in this program are summarized in Table I. Data include the lifetime in hours and filament operating temperature. The filaments are listed in order of increasing thickness. It is clear from the listed lifetimes that the larger the diameter for a given filament material, the longer its lifetime.

Two materials that form a protective chromium oxide film on the filament wire surface received more attention. These materials were more easily formed into coil configurations. This provided a longer filament with increased surface area. All the filaments were initially operated at 1100°C. A number of filaments have several hundred hours at 1100°C, but were listed as operating at 900°C since this was the temperature for most of the time of test.

4.3 Sodium Sensitivity Testing

The filaments of an operational sodium ionization detector (SID) sensor are expected to have the capability of operating for long periods of time (months or years). At any moment they may be called upon to detect a possible sodium leak. This may occur shortly after the installation of a new filament, or at a much later date. Consequently, the response performance characteristics of aged filaments are necessary to establish their continued suitability with time.

The filament materials used in previous studies⁽⁴⁾ to determine the mean time-before-failure were not operationally tested with sodium (i.e., aged in air only). The sodium testing was then performed on new detector sensor filaments. In this study, the sodium sensitivity response tests were performed on the filaments that survived lifetime aging determinations.

The sodium sensitivity tests were performed in a "model SID" which utilized one Pyrex enclosure originally used for filament lifetime determinations. This permitted the filament holder, with the test filament attached, to be removed from the lifetime test apparatus, and inserted into the model SID enclosure. The collector plate of the model SID was positioned in the enclosure so as not to interfere with the filament and arranged so that adjustments could be made to have a constant filament-to-plate separation gap (Fig. 6).

There was a delay before the actual sensitivity tests were performed. The filaments were operated in a flowing air stream during life tests; this was room air pulled through the life test apparatus by means of a vacuum pump. After the filaments were mounted in the sodium sensitivity model SID apparatus, tank nitrogen gas was used as carrier gas for the sodium aerosol. The background ion current response of this model SID was quite large due to the accumulation of dust from the air, presumably corresponding to the ionization of sodium in the dust particles. A conditioning of the filament was required, i.e., operating the filament in flowing nitrogen gas a few days, before the ion current began to approach a steady low value in the range of 0.1 nanoamperes.

The apparatus used for the sodium aerosol generation in these sensitivity tests consisted of a single element bubbler unit containing an aqueous solution of the sodium compound with a gas supply line. A baffle, prior to the gas exit port, prevented larger particles, i.e., greater than 2 μm size estimated from a prior study, from entering the sample line. The gas stream containing the aerosol was then passed through the model SID cell containing the filament to be tested.

This arrangement, although sufficient for the sensitivity test, produced a moist particulate. This moist condition probably had the effect of lengthening the recovering time, i.e., the time needed for the ion current signal to return to the normal background value when a stream of dry purge gas was passed through the model SID cell.

In order to overcome this effect, a modification was made in the aerosol generation system. An additional chamber with a secondary low pressure gas supply was located downstream from the aerosol generator. This provided a drying station for the generated aerosol mixture, thus reducing the moisture level. An additional benefit was that the aerosol concentration could be varied over a much larger range so that it was possible to check the linearity of response of the filaments. The sodium sensitivity test apparatus is shown schematically in Fig. 6.

Table II lists the filaments that had been tested for sodium sensitivity response. The data include the filament style, viz., straight wire or coil geometry; the filament lot; the operating time in hours that the filament was on life time testing prior to the sodium sensitivity test; and the signal response of the model SID utilizing that filament. The table includes both the background ion current and the signal ion current response to the sodium aerosol. Also included are the types of response for the filament. That is, whether a slow, medium or fast response was observed when the sodium aerosol was introduced into the model SID, or the speed of recovery to background ion current value after the purge gas was passed through the cell. Remarks for the filaments include: the apparent filament condition, which in general were clean except for life tested filament I-16 which had a thick oxide coating; and the filament operating temperature difference, i.e., whether the filament temperature at the time of sensitivity testing was higher or lower than the normal filament operating temperature of 900°C.

Two of the filaments listed had their response characteristics obtained after the drier-dilution chamber was added to the sodium sensitivity apparatus. It is apparent that when these filaments were operated at higher-than-normal operating temperatures, they showed a more rapid signal response. A number of filaments were tested at lower-than-normal operating temperatures, with sodium aerosol diluted as above, but are not included in Table II. The signal response for these filaments did indeed decrease when tested at the lower operating temperature, indicative of reduced thermal ionization efficiency. The dilution of the sodium aerosol also contributed to the reduced ion current signal response.

Figures 7 to 12 show the response of the model SID sensor to sodium aerosol using various Cr_2O_3 -protected filaments. Not all filament response tests are shown since the lack of signal current sensitivity disqualified the results obtained during the lower operating temperature runs.

5. TASK 3: EVALUATION OF CHEMICAL INTERFERENCE EFFECTS

5.1 Task Objective

Determine the response of the Sodium Ionization Detector (SID) sensor to possible spurious chemical interference agents originating from materials which are likely to be present in a Liquid Metal Fast Breeder Reactor (LMFBR) plant.

5.2 Identification of Interference Species

The sodium leak detection lower limit for the SID sensor is determined by the magnitude and variability of the background ion current. This background current can be affected by the presence of chemical interferences near the detector sensor. These interferences produce relatively minor ion currents compared to sodium. Thus, in order to choose the optimum leak detection setting for the sensor, the possible range of sensor response under realistic plant operating conditions should be known. A number of chemical interference species have been identified and include: dust, smoke, aerosols and vapors.

The dust particles could be present in the ambient atmosphere from concrete, a major structural material; soil, which tends to adhere not only to shoes but to clothing as well; and insulation, which is used to fill voids around various piping and electrical installations. Smoke particles could be produced from over-heated electrical cables or localized fires such as paper or tobacco. Aerosol contaminants may be produced by rotary equipment such as motors or bearings. Vapors evolve from the use of organic solvents and from the outgassing of binders used in insulation. Thus it is seen that in a structure as large as a LMFBR containment building, the possible sources of chemical interference species will be a function of the construction material and equipment.

5.3 Environment Chamber Testing

A fixed volume (0.76 m^3) cubical environment chamber was used to evaluate chemical interference effects. A fan provided turbulent mixing of the chamber air. This was necessary to avoid flow stratifications in the region of the SID sensor. The fan was located in an upper corner of the chamber and operated at low to medium speeds in order to avoid cooling the filament of the SID sensor.

In the initial test of the SID's ion current response to cigarette smoke in moving air, there was a rapid depletion of the interference species. This was attributed to deposition of the smoke particles on the chamber wall. When the smoke was introduced slowly into the chamber in still air (fan off) by means of a 5 cc hypodermic syringe, the smoke stratified. In this situation the response or lack of response of the SID sensor depended upon the relative position of the smoke layer in relation to the sensor; when the SID sensor was located on the same plane as the smoke, then a rapid and continuous signal was observed; otherwise there was no response. If the fan was turned on to circulate the air, the SID sensor then responded, but the signal soon diminished indicating loss of the smoke particles by deposition on the chamber walls. This effect is shown for cigarette smoke in Fig. 13.

To correct this wall deposition effect, the chamber was modified by relocating the fan at the bottom center of the chamber, and the fan was provided with a baffle plate. This is shown schematically in Fig. 14. The fan in this configuration moves a fraction of the total chamber air through its sub-enclosure. The remaining portion of the air recirculates in the larger upper portion of the chamber in which the SID sensor is positioned. This air flow pattern was confirmed by observing smoke streams emitted from a hypodermic needle. Although the problem of particulate deposition on the chamber walls was not completely eliminated, the time interval available for monitoring the SID sensor ion current was extended.

5.4 Evaluation of Interference Species

The tests performed in the environment chamber have shown that, in general, the smoke produced from a smoldering fire can produce a

continuous signal response by the SID sensor. This was true also for smoke produced from paper, fiberglass insulation, tobacco and electrical insulating materials. When the materials were heated more rapidly and ignited, producing a flaming fire, there was an initial rapid response of the SID sensor. However, the initial signal amplitude and the duration of the signal were not as pronounced as the signal which resulted from smoldering material. Evidently, the interference species from these materials were consumed in the flaming fire, yielding products which did not affect the SID sensor. The response of the SID sensor to smoldering fire particles of cigarette, rubber and polystyrene are shown in Figs. 15, 16 and 17.

The presence of vacuum pump oil or an electrical insulating oil in the chamber did not produce any marked effect upon the SID sensor signal, presumably because their vapor pressures were very low. These liquids were placed in an open dish and inserted into the base of the chamber. The vapor was circulated throughout the chamber since the dish location was near the circulating fan air stream. Lighter molecular weight materials, such as kerosene and organic solvents, which have a higher vapor pressure, did produce a slow, gradual increase in the background signal of the SID sensor. With removal of these liquids, the SID sensor ion current signal gradually decreased to its original background level. The SID response to kerosene vapor is shown in Fig. 18.

The vapor of a vacuum pump oil did not affect the SID sensor signal as mentioned above; however, when this same oil was injected into the chamber as an aerosol, there was a definite interference signal produced which is shown in Fig. 19. The signal response was rapid and contained numerous spikes of one to two nano-amperes. The spiked signal resulted from the individual aerosol particles. The signal response of the SID sensor to an electrical insulating oil aerosol was very similar in nature. There was a rapid initial peak signal of one to two nano-amperes and numerous spikes of decreasing amplitude as the aerosol was depleted from the circulating air stream.

The admission of dust particles into the environment chamber did not produce any noticeable response by the SID sensor. Sources of dust that were tested were: construction sand, soil and concrete. This neutral effect may indicate that the particulate size was so large that settling was occurring. Alternatively, one would not expect a pronounced SID response to large particles since their low specific surface area makes them less able to interact with the surface of the SID filament.

The results of Task 3 of this study are in agreement with a recent report⁽⁴⁾ that shows interference signals were produced under similar conditions.

Signature Sheet

Final Technical Report

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1a.

Straight-Wire Design

1b.

Helical Filament Design

1c.

Porous Sheath Design

Fig. 1 — Sodium ionization detector (SID) calibrator filament configurations

Fig. 2 – Schematic of the in situ calibration technique for the SID

Fig. 3 — Response of SID sensor to sodium signal generated by Cr₂O₃-protected calibrator filament under biased and unbiased conditions

Fig. 4 — Response of SID sensor to Cr_2O_3 -protected calibrator under condition of simulated SID filament failure. Various calibrator bias potentials indicated.

Fig. 5 – Response of SID sensor to SiO₂-protected calibrator filament with various bias potentials (indicated)

Fig. 6 — Schematic of sodium sensitivity test apparatus for aged filaments

Fig. 7 — Sodium sensitivity response of model SID utilizing Cr_2O_3 -protected filament I-8, straight style, 2100 hours life test, (A) no dilution of sodium aerosol, (B) 50% dilution of sodium aerosol, (C) 75% dilution of sodium aerosol

Curve 716596-A

Fig. 8 — Sodium sensitivity response of model SID utilizing Cr_2O_3 -protected filament I-16, straight style, new filament

Curve 716603-A

Fig. 9 — Sodium sensitivity response of model SID utilizing Cr_2O_3 -protected filament N-12 straight style, 550 hours life test

Curve 716604-A

Fig. 10 — Sodium sensitivity response of model SID utilizing Cr_2O_3 -protected filament N-12, straight style, 1840 hour life test

Fig. 11 – Sodium sensitivity response of model SID utilizing Cr_2O_3 -protected filament N-16, coil style, (A) new filament, lower temperature, (B) 456 hour life test, operating temperature of 900°C

Fig. 12 — Sodium sensitivity response at 900°C of model SID utilizing Cr_2O_3 -protected filament N-20, straight style, 760 hour life test

Fig. 13 — SID ion current response to cigarette smoke, showing the rapid depletion of interference species when air circulating fan was turned on

Fig. 14 — Schematic of environment chamber to test SID ion current response to various chemical interference species

Fig. 15 — SID ion current response to 35 ppmv cigarette smoke

Fig. 16 — SID ion current response to smoldering rubber

Fig. 17 — SID ion current response to smoldering polystyrene (Note current scale change between 6 and 10 minute intervals)

Curve 716601-A

Fig. 18 — SID ion current response to kerosene vapor at 1.8×10^{-3} atm. pressure

Curve 716607-A

Fig. 19 — SID ion current response to aerosol from
(A) vacuum pump oil, and (B) electrical insulating
oil

TABLE I

Observed Lifetime of Chromium Oxide Protected Filaments

<u>Lot Filament</u>	<u>Lot Diameter (mils)</u>	<u>Indiv. Filament Lifetime (hours)</u>	<u>Temperature (°C)</u>
N-9	9	120	1100
		264	"
		237	"
		168	"
		240	"
		648	900
N-12	12	459	1100
		213	"
		889	"
		3097	900
		1313+	"
N-16	16	672	1100
		408	"
		429	"
		359	"
		1848	900
		1172	"
N-20	20	1488	1100
N-12 Coil	12	3264+	900
		6984+	900
N-16 Coil	16	3144+	900
		5952+	900
N-20 Coil	20	3204+	900
I-8	8	96	1100
		46	"
		94	"
		188	"
		2103	900
I-12	12	360	1100
		334	"
		330	"
		166	"
		1174+	900
I-16	16	2327+	900
I-12 Coil	12	312	1100
		144	"

⁺Filaments still in operation

TABLE II

Sodium Sensitivity Response of Cr₂O₃-Protected Filaments

Lot	Filament Style	Life Time Hours Before Test	% Aerosol Dilution	Background Current nA	Na Signal Current nA	Response	Recovery	Remarks *
N-12	Straight	550	0	0.012	1.2	Fast	Medium	Clean Filament
	Straight	1841	0	0.01	0.4	Fast	Medium	Clean Filament
N-12	Coil	(New) 0	0	0.03	1.8	Fast	Medium	Clean Filament
N-16	Coil	456	0	0.14	4.2	Fast	Fast	Clean Filament
N-16	Coil	(New) 0	0	0.02	0.8	Fast	Medium	Lower Temperature
I-7	Coil	(New) 0	0	0.01	2.9	Fast	Fast	Clean Filament
			0	0.01	1.15	Slow	Slow	Lower Temperature
			75	0.01	3.2	Fast	Fast	Higher Temperature
			50	0.01	7.0	Fast	Fast	Higher Temperature
			0	0.01	8.4	Fast	Fast	Higher Temperature
N-20	Coil	216	0	0.01	4.0	Fast	Fast	Clean Filament
N-20	Straight	760	0	0.01	4.4	Fast	Slow	Lower Temperature
I-8	Straight	738	0	0.06	0.25	Medium	Medium	Lower Temperature
I-8	Straight	2103	50	0.01	0.3	Fast	Fast	Higher Temperature
			25	0.01	1.0	Fast	Fast	Higher Temperature
			0	0.01	2.9	Fast	Fast	Higher Temperature
I-16	Straight	(New) 0	0	0.032	1.2	Fast	Fast	Clean Filament
			0	0.025	0.93	Fast	Fast	Lower Temperature
I-16	Straight	2330	0	0.012	0.11	Slow	Slow	Heavy Oxide on Filament

* - Normal Operating Temperature 900°C
 Higher Temperature - 1000°C
 Lower Temperature - 800°C

APPENDIX A

SID Filament Failure

It has been observed that most of the SID filaments (SiO_2 -protected type) fail near the cooler end regions rather than in the central region where the temperatures are highest. The specific mode of failure involves a progressive thinning of the filament, accompanied by a whitish powdery growth as if a severe chemical attack were responsible. Figure A-1 shows a photograph of this type of failure. X-ray diffraction analysis of the white material indicated only original materials were present. The most likely source of such attack involves flux residue (fluorides) from the brazing process by which the cermet filaments are attached to the nickel end connecting tabs. Fluorides are known etchants for the cermet filament material. Indeed, the purpose of the flux is to remove the surface oxides so that the brazing alloy can wet the underlying filament material and thereby make a good bond. After each filament is brazed to the end tabs, it is given a boiling water treatment to remove any excess flux. A visual inspection is then made to verify that no surface flux residue remains. Despite these precautions, a recent series of tests on filament ends near the brazed zone indicated that almost half of them still had traces of chemically detectable flux present. The Bernite flux detection method,* which is sensitive to less than 10 micrograms of flux per square inch of surface, was employed.

Our present hypothesis is that the flux may be absorbed within the filament during the brazing process, either going into

* Bernite Products, Inc., Carle Place, N.Y. 11514

pores or combining with the oxide component of the cermet filament itself. If this hypothesis is correct, then the boiling water treatment would not remove this absorbed flux; subsequent operation of the filament would probably exhibit a slow corrosive attack due to the presence of this residual flux and the slightly elevated temperatures near the filament ends.

One obvious solution to this problem would be to eliminate the use of flux entirely. We have tested several fluxless brazes in vacuum, but with poor results; the braze would not wet the protective SiO_2 layer on the filament surface. A second scheme, which we have not tested; involves the use of a very high temperature fluxless braze to platinum; it has been reported that platinum and the cermet filament material form a eutectic, which could make this a feasible process for bonding. A third method would involve metallization of the filament ends by standard techniques (evaporation, sputtering), followed by a vacuum or furnace braze to the nickel support tabs.

It is our opinion that the fluxless bonding of filaments to their end support tabs is achievable, and that this would immediately result in reliable filament lifetimes of one year to two years. This is based on the excellent appearance of the central high temperature zone of SiO_2 -protected filaments after long term life tests.

Fig. A-1. Exhibiting powder type growth on aged filaments at the point where they were brazed to their support tabs.