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FFTF IN-LINE INSTRUMENTATION
FOR SODIUM PURITY MEASUREMENT:
STATUS REPORT

T. F. Demmitt

OCTOBER 1967

AEC RESEARCH &
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FFTF IN-LINE INSTRUMENTATION FOR SODIUM
PURITY MEASUREMENT: STATUS REPORT

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September 1967

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FFTF IN-LINE INSTRUMENTATION FOR SODIUM
PURITY MEASUREMENT: STATUS REPORT

T. F. Demmitt

ABSTRACT

The status of development work on in-line meters for measuring impurities in sodium is discussed. The meters are discussed in terms of their potential for useful service in the Fast Test Reactor (FTR).

The plugging meter, the electrochemical oxygen meter, the carbon diffusion meter, and the hydrogen diffusion meter are devices that should be developed in time for use in FTR during startup operations. The electrical resistivity meter may also find useful application at FTR if efforts to improve performance characteristics are successful. No other meters appear to have sufficient merit at present to warrant use in the FTR.

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FFTF IN-LINE INSTRUMENTATION FOR SODIUM PURITY MEASUREMENT: STATUS REPORT

T. F. Demmitt

INTRODUCTION

The Fast Flux Test Facility (FFTF) will be used to study the behavior of fuel and structural materials exposed to prototypic fast reactor environments. An important function of the FFTF is to provide reliable closed-loop testing under closely controlled and instrumented experimental conditions. The FFTF will provide fuels and materials irradiation capability as well as experience in design, testing, and operation of components and systems. The FFTF is to be responsive to all fast flux testing needs in the USA, including those of the Liquid Metal Fast Breeder Reactor (LMFBR) program.

One of the fundamental requirements for conducting a meaningful test program is the ability to accurately define the environmental conditions existing during the tests. Since one of the major variables in the FFTF test program will be coolant purity, it follows that adequate methods and hardware must be available to permit measurement of the purity. Impurity measurement can be performed either by withdrawing a coolant sample for analysis in external facilities or by evaluating the response from in-line process sensors permanently exposed to the coolant.

Programs are in progress in several laboratories to develop and characterize the methods and hardware required for both manual and in-line coolant purity measurement. Many progress reports and some review articles have been written concerning the results and direction of these programs. The function of this report is to provide an updated status report on the in-line coolant purity instrumentation development work, and to evaluate the potential for utilizing the results of this work to meet the specific needs of the FFTF.

SUMMARY AND CONCLUSIONS

This report reviews the pertinent information available concerning the status of various in-line meters for impurity measurements. The instruments are discussed in relation to their potential for useful service in the Fast Test Reactor (FTR) of the Fast Flux Test Facility (FFTF).

It is concluded that the plugging meter, the electrochemical oxygen meter, the carbon diffusion meter, and the hydrogen diffusion meter are devices that should be developed on a schedule compatible with FTR startup if possible. A prime requirement must be to provide proven models for use under the conditions which will be encountered in the FTR coolant systems. The electrical resistivity meter may find useful application at the FTR if current efforts to calibrate this device and to achieve increased sensitivity are successful. No other meters appear to have sufficient merit at present to warrant use at FTR. However, it is recognized that this conclusion could change as the result of continued development work on analysis methods.

The sodium purity measurement instruments needed for FTR are currently being developed under several AEC programs at sites other than PNL. At the present time the content, schedules, and goals of these instrument development tasks are not positively coordinated with the FTR schedule requirements. Redirection of these AEC sponsored instrument programs to insure provision of information needed on FTR decision dates is mandatory.

DISCUSSION

FFTF is an integrated system intended for fuels and materials evaluation studies. This complex consists of a fast reactor core (FTR), heat removal systems, fuel handling equipment, and various test and examination facilities.⁽¹⁾ The most important of these systems from the standpoint of sodium analytical chemistry are the open-core (primary) system, the secondary system, and the closed loops. Each of these

systems presents some unique impurity monitoring requirements, and each represents a different range of proposed operating conditions so that an independent evaluation of monitoring needs is necessary.

At the present time the ultimate operating conditions for the FTR coolant systems have not been definitely established. However, it is possible that primary coolant sodium temperatures to 1200 °F and closed loop coolant sodium temperatures to 1400 °F could be encountered. These conditions will necessitate careful control of the sodium coolant purity to minimize deleterious interactions with the structural materials and components that contact the sodium. Insofar as possible, it is desirable to automate the coolant analysis operations via use of in-line sensors, since the potential exists for using these devices to continuously monitor the coolant purity. Information obtained could be useful in establishing coolant purity trends and would assist in reaching operational decisions for the reactor and its experimental facilities. One very useful function the sensors could provide would be that of signalling operations personnel of possible off-standard conditions rapidly enough that effective remedial actions could be taken to correct the situation.

While it is recognized that there are many sodium contaminants that are of sufficient importance to warrant development of analytical procedures to periodically measure their concentration,⁽²⁾ this report will be limited to those contaminants for which in-line monitoring capability is desirable. This report will also exclude consideration of analysis methods and equipment required to monitor the type and amount of radionuclides present in the sodium coolants. The impurities of major concern to FTR are oxygen, carbon, and hydrogen.

In actuality, there is a need for two classes of in-line instrumentation for sodium purity measurement: (1) general purity meters (analogous to the electrical conductivity meters which are widely used in water-cooled reactor systems), and (2) specific contaminant meters that can identify and measure individual contaminants.

Table I lists the various coolant systems for FTR and indicates the type of in-line instrumentation that presently appears to be best suited for use in these systems.

TABLE I. Summary of In-Line Sodium Purity Monitoring Instruments for FTR

System	Monitor Type				
	Plugging	Resistivity	Oxygen	Carbon	Hydrogen ^(a)
Open-core primary	yes	possible	yes	yes	yes
Open-core secondary	yes	no	yes	no	no
Closed loop primary	yes	possible	yes	yes	yes
Closed loop secondary	yes	no	yes	yes	no

(a) In-line hydrogen analyses are not considered necessary unless refractory metals are present in the system.

There are also other peripheral FTR sodium handling systems that will utilize in-line instruments. These systems will not be discussed individually, however, since in each case only plugging meters are recommended.

The instruments and systems applications summarized in Table I are described in more detail in the following sections. The sections include a status report on each instrument and a summary of the additional research and development efforts required to bring the meters to the point of suitability for FTR applications.

PLUGGING METERS

The plugging meter is currently the most widely used impurity monitoring device for liquid metal systems.⁽³⁾ The basic advantage of this type of meter is its simplicity and the fact that it is responsive to the general purity of the coolant. In this respect it is similar to the in-line

electrical conductivity probes that enjoy such widespread acceptance in water-cooled reactor systems. Plugging meters can therefore be used to provide rapid and fairly reliable indications of FTR coolant quality.

In its simplest form a plugging meter consists of a flow-through piping system including a sodium flowmeter, cooler, plugging orifice, and temperature sensor. A schematic diagram of this type of device is shown on Figure 1.⁽⁴⁾ Note that the plugging orifice is located downstream of the cooler, and the temperature is measured at this point. In operation, the temperature of the sodium is gradually reduced until evidence of a sharp decrease in flow confirms that the orifice is plugging. The orifice temperature corresponding to the flow change is regarded as the "plugging temperature."

Many variations on the basic meter design are possible, and a considerable number have been tried. The most significant are those directed toward making the meter into a continuous reading device. In essence, this is accomplished by providing feedback controls to permit operating the meter under incipient plugging conditions. Meters of this type are by their very nature more useful than those that operate intermittently and that require more supervision and data interpretation effort.

The most significant disadvantages of the plugging meter are that there is no standard accepted design for these devices, and the response of the meters varies widely for different impurities.

The plugging meters should be included in all major FTR systems containing sodium coolants to facilitate general purity measurements. The continuous reading instrument concept is preferable. However, in view of the fact that there is no proven device of this type available at present, it is not now possible to specify such a device for FTR service. Continued research and development effort is required in this area as well as effort to establish a standardized design for the intermittent-reading type. Support for work in this area is being provided within the USAEC Liquid Metal Fast Breeder Reactor (LMFBR) Program.⁽⁵⁾ Considerable program planning and integration will be required to insure

FIGURE 1. Schematic View of Plugging Meter

that the level and direction of the effort will be geared to provide the best instrument for FTR consistent with the schedule for startup of this system. This will necessitate critical evaluation of the LMFBR instrument development programs and redirection where necessary to meet FTR needs.

It must be mentioned at this point that there is a tendency at some installations to attempt to calibrate the plugging meter response in terms of the oxygen concentration of the sodium coolant. Although this procedure is approximately correct at relatively high oxygen concentrations, it results in progressively larger errors as the oxygen concentration is decreased since chemical species other than oxygen also produce plugging meter response. The practice of equating plugging temperatures and oxygen saturation values to establish even "equivalent" oxygen concentrations should be discouraged. Plugging temperature data should be reported in terms of temperature values only, and both scientific and operations personnel should interpret data on this basis.

ELECTRICAL RESISTIVITY METERS

The electrical resistivity meter also indicates the general purity of the liquid metal coolant samples, since the electrical resistivity of the metal changes as the impurity types and concentration vary. In practice the device is sensitive to particulates and gas bubbles as well as to dissolved materials present in the coolant.^(6,7,8)

The resistivity meter concept has been developed to the stage where some models are available commercially. The most widely known example is the Blake Meter or Rhometer. A schematic diagram of this device is shown on Figure 2.⁽⁶⁾ The operating principle is based on the fact that a change in resistivity of the flowing sodium causes a shift in the phase of the alternating current in the secondary coil of the transformer (Figure 2). Determination of the phase shift between primary and secondary coils thus provides a direct measure of impurities in the sample.

FIGURE 2. Blake Resistivity Meter

The major disadvantage of this device is that the readings are appreciably affected by small temperature variations in the sodium since the temperature coefficient of sodium resistivity is large. Careful selection of a temperature compensating circuit is required to minimize the effects of this variable. This requirement involves significant engineering problems.

A secondary problem with the resistivity meter involves difficulties in calibrating the response. Blake indicated the theoretical sensitivity of the device should be 0.01% R (Resistivity) change per ppm of impurities in the sample. Calibration tests in USA laboratories have indicated values of 0.007 to 0.0139% R per ppm oxygen equivalent for general impurity species added to the sodium by raising cold trap temperatures.⁽⁷⁾ A value of about 0.004% R per ppm oxygen equivalent was obtained during injection tests where Na_2O was added.⁽⁸⁾ It has been estimated that the device has an ideal sensitivity to changes of about 0.01% R; however, practically it is questionable whether precision greater than 0.03% R could be achieved on a continuous basis during routine operation. The latter value corresponds to an impurity sensitivity limit of about 3 ppm using Blake's calibration relationship and about 7.5 ppm using the data obtained during the Na_2O injection tests described above.

In view of the problem described above and the recognized similarity in nature of response for the resistivity and plugging meters, the use of resistivity meters in the FTR coolant systems does not appear warranted since plugging meters will be used. It is recognized, however, that improvements in the concept could alter this conclusion prior to the time actual selection of FTR instrumentation must be made. One particularly encouraging development in this area involves the direct current resistivity meter being studied at LASL.⁽⁹⁾ This device differs from the Rhometer in several respects; however, the major one involves the fact that absolute rather than differential resistivity measurements are made. Continued evaluation of this device is desirable to establish

the actual operating characteristics. If performance of improved resistivity meters warrants their use, there are potential applications for these devices at FTR as indicated in Table I.

OXYGEN METERS

The continuous reading oxygen activity meter developed by the United Nuclear Corporation^(10,11) has undergone a considerable amount of performance testing under engineering test conditions.^(7,8,12) The device is specific for oxygen and is sensitive to very small changes in oxygen concentration. The meter has definite application potential for the FTR coolant systems as in Table I.

The meter sensor is a solid-electrolyte galvanic cell. A schematic diagram of the device is shown in Figure 3. The meter employs a thorium-yttria based ceramic electrolyte to separate the reference (copper-copper oxide) and sample (sodium-sodium oxide) half-cells. When the device is immersed in flowing sodium at elevated temperatures, an electromotive force (EMF) is developed that is proportional to the difference in potential of the half-cells. The electrical conductivity of the solid electrolyte is predominantly due to oxygen ion transport, although some electron conduction also occurs. Since the potential of the reference electrode is constant, changes in the cell EMF are primarily due to changes in the oxygen activity of the sample sodium. The cell EMF can therefore be calibrated in terms of sodium oxygen concentration. Since the relationship between cell EMF and sample oxygen concentration is logarithmic, the meter is extremely sensitive to oxygen. For example, changing the oxygen concentration from 1 to 2 ppm gives the same cell EMF change as going from 10 to 20 ppm. The device thus has good potential for use in even highly purified sodium such as will be present in the FTR closed loops.

The device has been developed to the hardware stage and is commercially available. Engineering tests in forced circulation loop systems have demonstrated that response of operating probes to oxygen additions as Na_2O , NaOH , and H_2O is rapid and reproducible. Addition

FIGURE 3. Electrochemical Cell

of mixed impurities by elevating cold trap temperature also causes response that correlates with oxygen measurements obtained by conventional sampling and analysis techniques.

Although the oxygen meter appears to be a highly useful and versatile unit, it is important to realize that there are some areas where additional effort is required to evaluate and describe the performance characteristics. These include the following:

- Solid electrolyte (ceramic) tube fragility and metal-to-ceramic sealing techniques require further evaluation.
- Calibration curves may vary for different probes. These variations may be the result of variations in electrolyte impurity concentration effects on oxygen ion and/or electron conduction.
- Meter readings vary as the result of changes in γ -radiation field intensities.⁽¹²⁾ The significance and nature of these variations is not understood at present but must be described to assess suitability for service in radioactive sodium.
- Ambient room temperature fluctuations cause oxygen meter variations.
- Temperature limitations currently restrict operation of the probes to well below the ultimate values planned for FTR coolants, and the temperature coefficients (mV/°C) vary somewhat between different probes.

Some progress is being made toward identifying and resolving the development items described above. However, an accelerated effort will be required to achieve the goal of having proven hardware available for service in FTR systems at startup.

At the present time there is no truly promising alternative to the electrochemical meter for performing in-line oxygen concentration measurements in FTR sodium systems. The only other device that has received much attention for in-line oxygen measurements is the niobium corrosion meter. This device has been described previously by Simons et al.⁽²⁾ as follows:

"Davis and Draycott⁽⁹⁾ describe an impurity-sensing device which measures impurity levels indirectly, although measuring directly the corrosiveness of contaminated sodium. Essentially, determinations are made of liquid metal flow through two multihole orifice plates in parallel, one plate fabricated of niobium and the other fabricated of stainless steel. In principle, sodium containing a certain level of oxygen will corrode the former at a certain rate, while the latter will be unaffected. Hence, differential flow through the orifice pair will occur as time passes, and its magnitude will indicate a corrosion rate, which is relatable to oxygen level. The operating temperature level is in the 400 to 600 °C range."

Very little information is available concerning performance of this meter as regards sensitivity, response time, maintenance requirements, specificity, etc. However, on the basis of available data it does not appear that the device would be suitable for FTR service requirements. Further developments in this area should receive careful attention, and interest in the meter could receive considerable impetus if the electrochemical meter does not prove to be as useful as presently indicated.

CARBON METERS

At present there is no proven device available for in-line carbon measurements in sodium. However, United Nuclear Corporation is presently pursuing laboratory studies of a very promising device for this purpose.^(13, 14) The technique is similar to the electrochemical oxygen meter insofar as it facilitates measurement of the chemically active (diffusible) carbon species present. This is a particularly significant advantage in the case of carbon where existence of particulate material has already been demonstrated in some sodium systems, and where correlation of carburization data in terms of total carbon concentrations present has not given satisfactory results. The device has definite application potential in FTR systems, as indicated in Table I.

The carbon meter involves a metallic diffusion membrane which contacts sodium on one side and a decarburizing gas supply on the other. A schematic diagram of the meter is shown on Figure 4.⁽¹³⁾ Present versions of the device employ iron as a diffusion membrane and a water-hydrogen-argon mixture as the decarburizing gas. The meter operates

FIGURE 4. Schematic Diagram of UNC Carbon Diffusion Meter

at about 1400°F, and carbon diffusion rates are calculated from measurements of the rate of formation of carbon monoxide in the gas stream. Actually, the carbon monoxide is converted quantitatively to methane by passing the gas over a ruthenium catalyst bed, and the methane is then measured to assess the carbon transfer using a flame ionization detector.

Results obtained to date are highly encouraging, it appears the device has sufficient sensitivity to permit active species carbon measurements at concentrations at least down to 1 ppm. Response time of the instrument is such that stable response to concentration changes can be obtained within a matter of minutes, which is adequate for all proposed FTR applications. To date it appears the device is specific for carbon. That is, there is no measurable interference due to the presence of impurities normally encountered in reactor coolant sodium.

The primary technical problem with the meter at present is variation of the carbon transport rate across the diffusion membrane. This effect could be the result of progressive carburization of the membrane material. An alternative possibility is that accumulation of corrosion products on the membrane as the result of oxidation by the moist decarburizing gas may retard carbon diffusion directly or indirectly by interference in the gas-solid reaction kinetics. To date it appears that carburization is a relatively unimportant factor and that oxidation effects are controlling. The full significance of this effect has not been defined as yet and currently occupies the number one priority of the program. The effect will certainly be a major factor in assessing the suitability of various alternate membrane materials (e.g., nickel) for this application and the useful lifetime of the carbon meters.

The present development program that United Nuclear Corporation is pursuing includes design and construction of fully operational prototype meters suitable for routine use in field trials. These prototypes will then presumably undergo intensive engineering testing to determine performance characteristics in operating test loops. Following successful completion of this field testing, it is likely that limited production of commercial devices would occur for use by interested organizations.

There is a definite need for an in-line carbon analyzer for use in the FTR sodium systems. This promising program should be pursued as rapidly as possible with the ultimate goal being to insure availability of fully tested and proven meters, including demonstration tests in radioactive sodium, prior to the startup of the FTR.

HYDROGEN METERS

Hydrogen as an impurity in FTR sodium coolant systems is a moderately important variable. Although hydrogen present as NaH or NaOH is not a major factor in overall corrosion behavior of structural and cladding materials, it is a well known fact that hydrogen can cause low-temperature embrittlement of refractory metals such as zirconium, niobium, and tantalum. This can cause undesirable changes in physical properties of these materials. Thus, careful control of hydrogen will be required in all FTR coolants where refractory metals may be present, including the open-loop (primary) coolant. There is also some speculation that hydrogen may interact with other impurities, such as, oxygen and carbon, to affect corrosion processes involving these impurities. Potential applications therefore exist for in-line hydrogen meters in both the open-core and closed loop primary coolants at FTR as indicated in Table I. Secondary coolant applications are not considered necessary since air blast heat exchangers will be used in FTR rather than steam generators as a final heat dump.

An in-line, continuous readout meter for hydrogen analysis in sodium was developed at Atomics International.^(15, 16) The device has been further refined to include an improved method for pumping, collecting, and measurement of the hydrogen and is currently undergoing extensive engineering tests by Atomic Power Development Associates personnel.⁽¹⁷⁾ A schematic diagram of the meter is shown in Figure 5.

The hydrogen meter measures active (diffusible) hydrogen. It is based on the principle that hydrogen present in sodium will diffuse through a metallic membrane, from which point it can be collected for analysis. At present, the diffusion membrane being evaluated is a bimetallic diaphragm composed of a nickel tube plated with palladium. The sodium coolant contacts

FIGURE 5. Schematic Diagram of APDA Hydrogen Diffusion Meter

the nickel surface, and the hydrogen diffuses through the membrane to the outer palladium surface where it is catalytically oxidized to water by a flowing argon-oxygen gas mixture. The water produced is measured using a Consolidated Electrodynamics Corporation moisture monitor. The rate of moisture accumulation can thus be calibrated in terms of the hydrogen concentration in the sodium if process variables can be held constant during the measurement, and if all hydrogen-containing compounds can be destroyed thermally to release the hydrogen for measurement.

Proof-of-principle tests have been completed and demonstrate that the meter is capable--under laboratory conditions at least--of performing the required monitoring functions. The meter appears to be specific for hydrogen and capable of measuring hydrogenous impurities such as NaH, NaOH, and H₂O added to the sodium coolant. Response time of the instrument is short enough that changes in concentration can easily be detected in a matter of minutes, and the meter is capable of measuring concentrations as low as a few ppb according to APDA.⁽¹⁷⁾

At present there are two main problems associated with the hydrogen meter probes tested: (1) the response from probe to probe is not uniform; that is, individual calibration curves are required, and (2) the probes show a tendency toward variation in response with time, perhaps as the result of partial poisoning of the palladium catalyst surface or loss of surface area. Since the measurement technique is fundamentally sound, it appears likely that the problems described will succumb to further efforts to improve the probes, and reproducible results will eventually be obtained.

Although this device has not yet undergone extensive testing in radioactive sodium, there would not appear to be any significant potential for a major effect of radiation on meter response. Continued testing and evaluation of the meter is warranted with the ultimate objective of providing proven meters for FTR use on a schedule that is consistent with the projected startup date for the reactor.

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M. Rau
W. E. Roake
J. D. Schaffer
F. A. Scott
F. A. Shadel
D. W. Shannon
D. E. Simpson
C. R. F. Smith
R. J. Squires
G. H. Strong
C. D. Swanson
J. C. Tobin
K. G. Toyoda
M. Vogel
D. M. Walley
J. H. Westsik
L. A. Whinery
R. D. Widrig
N. G. Wittenbrock
M. R. Wood
F. W. Woodfield
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