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12. Fission and Corrosion Product Behaviour in Primary Circuits of LMFBR's. A Status Review of Work Being Undertaken in the UK

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1. INTRODUCTION

Safe and economic operation of Fast-Reactor systems require an understanding of how long lived isotopes (eg ^{54}Mn, ^{60}Co and ^{137}Cs)* and bulk corrosion products are released, transported and deposited in sodium circuits. For example build-up of radioactive species in those areas of the reactor which require periodic inspection or maintenance, such as pumps and heat exchangers, could make these operations both costly and difficult. Likewise build-up or the 'smearing' of corrosion products over pipework or heat-exchanger surfaces could effect the thermal-hydraulic performance of the system by the formation of deposits and roughening of pipework surfaces.

The UK programme to study fission and corrosion product behaviour in fast reactor systems is aimed at establishing:

a) the magnitude of material and radioactivity release to the primary coolant and the respective contribution from fuel cladding and structural components.

b) the pattern of corrosion product and activity deposition in various parts of the circuit.

c) the accuracy of estimates of activity levels deposited on the surfaces of circuit components liable to require maintenance or repair, and associated dose rates.

d) the effect of corrosion product deposition on circuit hydraulics and heat transfer.

2. CORROSION & MASS TRANSPORT BEHAVIOUR OF NON-ACTIVE AND ACTIVE CORROSION PRODUCTS

Non-Active Studies The aim of the REML experimental programme is to study mass transport of materials in sodium using both small and large loop facilities. The corrosion studies are primarily concerned with trying to understand the mechanism of stainless steel corrosion in sodium and special attention is being paid to the role individual alloying elements play in the corrosion process.

* ^{54}Fe (np) ⎫ ^{55}Mn (n 2n) ⎬	^{54}Mn	300 day half-life
^{59}Co (n γ) ⎫ ^{60}Ni (np) ⎬	^{60}Co	5.27 yr half-life
Fission product	^{137}Cs	30 yr half-life

The effect of heat flux on corrosion rate is also being studied and experiments are being carried out to establish what effect entrained particles in the sodium stream have on the corrosion behaviour of cladding alloys. An investigation using active materials, is also being mounted to study the physical and chemical nature of released material.

The deposition studies on the other hand aim to establish the chemical and physical nature of deposited material, the rate of build-up of deposits with time and whether material is eventually displaced to other sites around the circuit during continuous loop operation at oxygen levels in the range 5-25 ppm.

Other factors which can effect deposition behaviour in sodium systems such as rate of heat extraction, circuit geometry and temperature differential are being investigated in the Large Mass Transfer Loop which simulates the PFR primary circuits (see later). Additional work on the chemistry of the deposits will be undertaken using active materials to 'label' those constituents transported from the corrosion zone. The effect of oxygen level in the sodium on the nature of the deposit and on the kinetics of removal of these active constituents is to be investigated.

In parallel with the corrosion and deposition studies work is also in hand to investigate the mass transport behaviour of interstitial elements such as nitrogen and carbon in sodium systems. Part of this investigation will be devoted to trying to determine whether interstitial element transport can be associated with certain types of deposit, which are observed.

The facilities being utilised at REML to study these problems are the small corrosion-deposition loops (see Ref 1 and Fig 1), an active loop and the Large Mass Transfer Loop. The latter being a simulation of the prototype fast reactor primary circuit. The active loop and the large mass-transfer loop are additional facilities to those already described at the Bensberg meeting (2) and therefore their role in the REML programme will be briefly described.

The experiments that are to be carried out in the REML active sodium loop entail exposing irradiated specimens of stainless steel in the corrosion part of the loop and following the accumulation of activated isotopes such as 59Fe, 51Cr, 60Co, 54Mn and 172Ta by gamma-ray spectroscopy after their deposition from sodium at various points around the circuit.

More generally, experimental objectives will include efforts to determine the effects of oxygen level and circuit temperature differentials on deposit distribution. In areas of peak activity attempts will be made to obtain sufficient deposits for subsequent identification using X-ray diffraction, microprobe and chemical analysis techniques. An increase in bulk deposits might reasonably be achieved by increasing the corrosion rate through raising the oxygen level in the sodium.

The purpose of the Large Mass- Transfer Loop is to study the pattern of metal release and deposition in a simulated PFR primary sodium circuit. The design of the loop caters for a study of the release of corrosion products under near-identical conditions from a simulated core and neutron shield rod (NSR) assembly. This released material then passes through certain key items, which have been identified as the BCD and IHX tubes, where it may deposit. In addition to these critical areas deposition could occur in other areas of reactor such as the cold trap, the sodium pool or the lower end of the fuel pin assembly. Because of this possibility a simulation of the areas has been included in the design. (See Fig 2).

The loop contains a full size simulation of a PFR IHX tube and a length of BCD tube. In the design the IHX tube sets the scaling factor for the loop and where possible all other items in the various test sections are scaled to 1/10848 cf PFR sizes. Sodium flows, temperature, velocities, surface area to sodium volume ratio and Re Nos are taken from current PFR design and data sheets. The loop has now completed its first test period of 7 months.

Summary of Experimental Findings and Conclusions

Corrosion of stainless steel in liquid sodium. With the introduction of M316 stainless steel as the main fuel cladding alloy for the first and possibly subsequent charges in PFR recent REML studies have concentrated upon using this material to study stainless steel behaviour in sodium. These tests have now confirmed the early findings and more precise rate equations are available for the corrosion of 316 type stainless steels as

a function of temperature, oxygen level, velocity or Re No, time and position in the isothermal part of the circuit.

In order to establish whether these equations are valid for other types of sodium systems tests have also been carried out with a number of different loop configurations with different operating temperature differentials to verify whether sodium volume and/or loop geometry modify these equations. So far, none of these additional factors have been found to have a marked effect on the hot side corrosion values.

Microprobe analysis of the ferrite layer which is now commonly observed on M316 stainless steel after sodium exposure indicates that the layer generally contains around one per cent of nickel and 5 to 8 per cent chromium. This is to be compared with the approximate values of 12 and 18% respectively which are present in the as-received material. Also if one compares the corrosion behaviour of this 'alloyed-ferrite' in sodium with that of pure iron important differences are observed in response to changes in oxygen content and Re No of the sodium. For example the presence of small amounts of alloying elements across the ferrite layer on austenitic steels of the M316 type give a lower power dependence of corrosion on oxygen content than does pure iron which is substantially unalloyed ferrite. There is also some evidence that surface roughness is increased by the presence of alloying elements and this effect is enhanced at higher oxygen levels. Therefore it has been tentatively suggested that the point of inflexion in the corrosion rate - Re No relationship for the M316 materials occurs when the depth of surface roughness exceeds the thickness of the laminar sub-layer or diffusion layer.

Overall the oxygen impurity level in the sodium still remains the most powerful factor in controlling the rate of mass-transport of austenitic materials, and should the levels of oxygen increase in the sodium to produce high oxygen or 'fault' conditions than intergranular and sub-grain boundary penetration with eventual break-up of the surface to produce erosion damage must be expected. Such effects can however be eliminated by efficient control of cold-trapping procedure to maintain the oxygen level at the desired lower levels. For further details of this work see Ref 3 and for details regarding corrosion in simulated reactor situations see later under Large Mass-Transfer Loop Studies.

Deposition of steel material in sodium systems

Small loop systems The study of the deposition behaviour of corroded material in small or simple loop systems at REML has been carried out in two stages. The first stage consisted of simple studies which were designed to look for deposits in small loop systems and to quantify their rate of build-up in a simple sodium circuit over extended test durations. For this study four specimen holders were

welded into downstream and up-stream positions' of an REML test loop and these contained specimens which were exposed to sodium at velocities of $7\frac{1}{2}$ and 15 fps (Re No $4.7 \times 10^4 \sim 9.5 \times 10^4$ respectively), for 134 weeks at an oxygen level of 25 ppm. The release rate in the maximum temperature $600^\circ C$ part of the corrosion zone was 0.001 in/yr.

For the second stage of the study the loops have now been re-designed to accommodate more specimen holders and to achieve working temperatures more consistent with the reactor primary circuit. The number of specimen holders welded into the circuit are now twenty in number and with this increase it is possible to follow the effects of Reynolds number more accurately in various loop positions and also to make a comparison of the corrosion behaviour in 'multi-holder' loops (ie specimens in up-stream and down-stream positions) compared with the rates already observed in single holder loops.

So far one series of tests have been carried out in this loop at an oxygen level of 25 ppm. The duration of the test was 125 weeks and specimens were examined after 23, 39, 74, 99 and 125 weeks. The maximum temperature of the specimens in the corrosion zone was $675^\circ C$ and the minimum temperature in the deposition zone was $425^\circ C$. For most part of the test the velocity in the high velocity sections was 13 fps (Re No ~ 105) however during one period the velocity was increased to 20 fps to establish whether this had any affect on the pattern of deposition in various parts of the circuit. With the completion of this test another experiment at an oxygen level of 10 ppm has now started.

Before summarising our preliminary conclusions from these tests it is perhaps worthwhile making a few general comments regarding certain experimental findings. For example, during the initial stages of loop operation (ie 0-23 wks) the pipework surfaces could be divided into three main areas, namely a corrosion area which covers the region before and after the position of maximum temperature, a 'neutral' area which is up-stream and downstream of the corrosion zone and thirdly, an area of pronounced surface oxidation and deposits. This area was associated with the colder parts of the loop.

X-ray and microprobe analysis of the 'neutral' zone and the oxidised surfaces in the cold zone showed that surfaces were covered with ternary oxides based on sodium chromite. In the neutral zone the structure of the oxide was predominantly sodium chromite ($NaCrO_2$) whereas in the cold zone the chromite structure contained a mixture of iron and chromium and in certain regions sodium ferrate $(Na_2O)_2$. FeO was also identified. In addition to the various oxide formations X-ray analyses also identified certain types of carbide which again could be related to a particular part of the circuit. For example carbides of the $M_{23}C_6$ and M_6C type were associated with the corrosion or high temperature part of the circuit whereas M_7C_3 plus $M_{23}C_6$ carbides were a feature of the lower temperature part of the circuits. This infers that the sodium in these tests had a fairly high carbon potential which increased in the cooler parts of the system.

As the test proceeded it was found that the corrosion zone began to extend at the expense of the neutral zone (see Fig 3). This was probably due to erosion of the sodium chromite films. On the cold side of the loop (positions 4A to 8A) however, weight gains, which were the combined effect of initial surface oxidation and material transport, increased with increasing test duration and most marked effects were observed in positions 4A where metallic deposits were found to build-up with continuing exposure to sodium before they 'broke-away' to other parts of the circuit. In other parts of the circuit (ie positions 4 to 8A) general deposition of metallic particles was also observed but not to the same extent as in position 4A.

Turning now to more general conclusions it can be stated:

(1) The type of deposit in terms of particle size, general shape and composition did not radically alter with continuing exposure to sodium. This implies that the type of material being removed from the release zone is consistent if the chemistry and operating conditions remain constant.

(2) Irrespective of the position in the cold zone a large part of the deposit was composed of metallic particles of about 2 micron size. Certain particles are of sub-micron size while larger particles, which are probably entrained swarf, can be of the order of 25 microns or greater. Point analysis using microprobe and non-dispersive analysis plus chemical etching indicated the particles were ferritic. However, an area analysis using the microprobe analyser indicated that the general composition was similar to that of 18.8 stainless steel.

(3) In certain areas of the cold zone there was also evidence of surface break-up under the deposit which was probably caused by internal oxidation. This material which is slightly different in size also enters the deposit.

(4) The thickness of the deposit in any particular region can vary substantially. For example, the deposits in position 4A have been observed up to thicknesses of 0.010in.

A common characteristic of the particulate material is that it is porous and the conversion factor from weight to thickness assuming zero porosity needs to be multiplied by a further factor of x3 to obtain the observed thickness.

(5) From the operating standpoint slight reductions in cold side temperature did not induce the deposits to move to lower temperature parts of the circuit. Increasing the velocity from 13 to 20 fps seemed to have some slight effect in that deposits tended to build-up more in positions downstream from position 4A (see Fig 3).

(6) A common feature of the behaviour of the deposit is that all the major deposition occurred in the narrow bore specimens. Outside some slight corrosion of the wide bore specimens in those areas which were initially the 'neutral' zone, deposition in the wide bore specimens was negligible (see Fig 4). Similarly it is assumed deposition on associated pipework in the loop is also negligible.

(7) It has been established that deposits will grow to some limiting or critical thickness before they are mechanically damaged by the flowing sodium. Once this critical thickness, which can be of the order of 0.007 in has been reached the deposit is dislodged by the sodium and substantial material is removed to downstream positions. (Sheared and attached particles). (Fig 5 & 6)

(8) It is not possible to achieve a mass balance on the simple loops, because components such as the return side of the heat exchangers cannot be examined during the test.

Summarising these main points it is considered that in any analysis of element transport in loop systems long term tests are necessary to mitigate those short-term effects which are associated with surface structural changes of the specimen materials. For example, in the current tests it has been found that test durations beyond 23 weeks are required to obtain this situation. Up to this period element enrichment or depletion in a certain part of the circuit is not a function of element transport but is more a function of local surface or structural modifications caused by sodium chromite or carbide formations. During longer term operation these compounds will be removed to other parts of the circuit. However,

once the surfaces of the underlying pipework have achieved steady state composi-
tions then sensible estimates of element transport can be made.

Secondly, in the calculation of deposition rates for certain parts of a reactor
system cognisance has to be taken of what is occurring in other parts of the
circuit. For example in the second stage of testing no constant deposition rate
was found in any position because there were varying hold-ups of material in the
liquid sodium at different times. Some of this material was primary released
material while part of it was secondary deposits released from upstream positions.
This behaviour, which is probably typical of a high release rate situation
($\sim$ 0.002 in/yr metal loss) contrasts with our initial tests (maximum temperature
of 600°C and release rate 0.001 in/yr) where the deposition rate remained constant
over a period of 134 weeks and the deposit never reached unstable proportions.
Therefore in making a comparison between the deposition rates in various circuits
it is felt that loop design and specimen position must be taken into considera-
tion. This has been done in comparing Westinghouse findings (Ref 4) with REML
findings to arrive at a deposition rate for the type of particles under consider-
ation. The results which are illustrated in Fig 7 indicate that under com-
parable conditions of flow rate, oxygen level, loop position and temperature
a more or less constant deposition rate is obtained for the two experiments.
However it is to be noted that increasing the velocity markedly increases the
deposition rate.

Finally as regards the way this material deposits in the system it is highly
probable that the process is more associated with impaction and stiction and
not transport across say a laminar sub-layer. Calculations are required to
confirm this view. Nevertheless it is tentatively suggested that in
reactor systems fine particulate material in turbulent flow will tend,
because of the tangential forces on the particles, to be drawn to the wall
of the conduit where at high turbulence levels the resistance to mass-
transport is reduced and the probability of sticking is increased. With
continuing build-up of deposit and some local fusion or sticking of particles
the deposit can eventually become mechanically unstable in the sodium stream and
the material will break-away to reform in downstream high Re No positions.
However, as the apparent density of the deposit increases its terminal velocity
will also increase which means that agglomerated fine particles will tend to
sink in slow flow regions of the loop. This is thought to account for the high
accumulation of deposits located in the slow flow side of the loop heat
exchangers.

Large loop or simulated systems The first seven months of operation of the
large mass-transfer loop have now been completed. Although the commissioning
of the circuit proceeded satisfactorily the first period of testing has been
a 'shake-down' period for the loop, the operators and the various support
groups. For example, methods of loading and unloading specimens and holder,
which in some instances are 14 ft long, and the sampling of the circuit for
the relevant impurities have had to be evaluated.

For the first part of the test certain limits on circuit operation, especially
in the core test sections, were identified and lower sodium flows and temp-
eratures in one of the sections had to be accepted.

As regards loop operations generally it was found that the operation of two
circuits in contra-flow could be carried out satisfactorily and heat fluxes
of the order 2.4×10^5 BTUs/ft^2/hr and $\sim 2.0 \times 10^5$ BTUs/ft^2/hr were obtained
in core test sections one and two respectively. Although, as previously
mentioned, the sodium velocities were lower than those required, the Re No
values were sufficiently high $> 3 \times 10^4$ and the temperatures of 600 and 675°C

in the two test stations were considered reasonable for the first stage of
testing. In addition although the reduced flow had some effect on the flow
through the IHX and BCD section the changes were marginal and the rate of
heat extraction and ΔT along the specimen length were about the right order.

The sodium used in the experiment was taken from the PFR tank farm at DERE.
This sodium is of fairly high purity consequently it was difficult to main-
tain a high oxygen level in the loop sodium although the cold trap was opera-
ting at 180°C. Checks on the impurity levels were made using the plugging
meter however the recorded values varied during the test period because species,
other than oxide, were interfering with meter operation. Direct sampling of
the sodium was initiated half-way through the test and the oxygen levels recorded
over the last 3 months of loop operation were in the range 3-7 ppm.

From the standpoint of materials behaviour in the loop the various test sections
could be divided into weight loss and weight gain regimes. For example, the
weight loss values were associated with the top half of the heat flux region of
the core test specimens and the neutron shield rod specimens (see Fig 8)
whereas the weight gain sections were:

(a) the inlet to the core test stations (ie the part prior to the heat flux
zone plus half of the heat flux zone).

(b) the total length of the IHX tube (Fig a)

(c) the total length of the BCD tube

Briefly, the corrosion rate of the M316 fuel cladding material in the heat
flux zone increased with increasing temperature and the recorded maximum rate
of metal loss of 0.0002 in/yr, which occurred at the maximum temperature
of 675°C, is consistent with an oxygen level of 5 ppm if contribution from
specimens in upstream positions is ignored.

The surface structure of the steel in the core region is also typical of
that already observed in previous tests without heat flux. (Fig 10). There
is also evidence of ferrite layer formation both in the maximum temperature
position and position beyond this point which suggests that the sodium is not
completely saturated with soluble products after it leaves the core test sec-
tion. This view is further supported by the recorded corrosion losses on
the neutron shield rod specimens (Fig 8).

Once the sodium leaves the core area there is no further evidence of corrosion
and weight losses give way to weight gain. The recorded weight gains (see
Fig 9) were relatively small with the exception of a layer of small particles
on the surface of the IHX tubing metallographic examinations suggest there is
no build-up of deposits in either the IHX or BCD tubes. However, the small
particles which cover parts of the heat exchanger tubing surface are of
some interest. For example, it has been found that they vary in size down the
length of the tube, ie they get larger as the sodium becomes colder, and
secondly they contain high levels of Mn and Ni (see Fig 11).

Although it is not possible at this stage to say how the particles are formed,
two possibilities are suggested. First the particles which have the character-
istic structure of gamma-manganese are either residues caused by the reduction
of any oxide films which formed during the first few weeks of sodium exposure.
High levels of manganese and chromium could be anticipated in certain types of
oxide layer. Second, the manganese along with the nickel could have been
transported in solution in the sodium from the core test section to the IHX

where they have interacted and precipitated to form polyhedral particles. Whether this finding is fundamental to the transport of manganese in sodium systems can only be clarified by further testing, which should produce an increase in the number of precipitated particles.

Summarising the large mass-transfer findings the preliminary evidence suggests:

1) Corrosion losses in reactor systems will be apparent over approximately half of the heat flux zone. These losses will be a maximum at the maximum temperature of the pin and will decrease as the sodium moves out of the heat flux zone.

2) The corrosion potential of the sodium in the large mass-transfer loop is sufficiently high to cause corrosion of the neutron shield specimens and therefore it is possible that these components in the reactor will add to the corrosion product burden in the system.

3) Within the limits of the sodium chemistry capability during the test the effect of heat flux on corrosion rate does not appear to be substantial. Tests will continue at higher values of heat flux and sodium velocity to confirm this view.

4) The amount of bulk corrosion products observed in the IHX and BCD section is small as to be negligible. A mass-balance of the system indicates more material has been lost than gained and therefore it is assumed that the sodium pool or the cold-trap have collected most of the corroded material. It is intended to carry out a more detailed examination of this part of the circuit after an extended period of loop operation.

5) The finding of Mn-Ni rich particles in the heat exchanger could have some bearing on the way Mn and Ni are transported in sodium systems. More experimental work is required to confirm this finding. In addition the absence of bulk sodium chromite films on the various material surfaces is of interest in other areas of reactor operation.

Active Studies   The main features of the Harwell mass transfer loop were described in detail at the last specialists meeting. (See Ref 2).

Simulation of reactor conditions in the loop extend, at present, to establishing the relevant temperature, flow and material conditions for a single tube of the intermediate heat exchanger (IHX) of a typical fast reactor. The corrosion test section itself is exposed to flowing sodium at a high velocity and the highest temperature of the circuit. The test section is, at present, not heated, although it is considered desirable to simulate the heat flux expected from the surface of a fuel pin. It is intended that heat flux simulation will be provided through the installation of an electron bombardment heater, currently being tested, into the circuit.

The main method of controlling the oxygen level in the circuit is by means of a forced circulation regenerative cold trap. This is air-cooled and is located on a bypass to the main flow. As an additional means of controlling the oxygen level, a duffusion cold trap is included in the main circuit. Analysis for oxygen in sodium is by the vacuum distillation method using sodium samples taken by tap sampling into a dry box purged with 10 l/m of argon at a purity of < 1 ppm oxygen and moisture. With this arrangement it is intended to study the effect of varying the oxygen content on release rates and the influence of both a forced circulation and diffusion type cold trap on activity profiles.

In the experimental programme various aspects peculiar to irradiated steel release are also being considered. In summary, these are:

(a) influence of possible variations in the concentration of trace elements on levels of induced activity

(b) reliability of neutron activation cross-sections used in deriving induced activity levels, and the consequences on circuit activity assessments

(c) error assessments on released activity predictions for specified reactor conditions.

To provide additional quantitative data, selected pipework is removed from the circuit and treated to isolate active and inactive corrosion products. In the case of the active species the washings can be chemically analysed and then counted, using a prepared standard, to yield the amount of "active" material. Activity measurements in this case are made using a multi-channel gamma spectrometer.

Summary of Main Conclusions from the Active Studies

Transport of Fe-59

1. Fe-59 transport from a pure iron source in a liquid sodium system is characterised by its appearance as a deposit on pipework from immediately downstream of the hot corrosion zone. Comparable amounts of iron are found on sections of pipework that are isothermal or have a temperature gradient along them. Iron movement from other parts of the circuit indicates a similar behaviour.

2. Although increased deposition of active iron with increased velocity is observed and bends induce preferential deposition the migration of deposited activity is a much slower process than the initial deposition process.

3. Deposits up to 5 microns thick have been produced after experiments of 3000 hr at high oxygen levels. Visually the deposits range from a brown patchy film appearing at low flow rates to a uniform dark grey film at high flow rates. Auger spectroscopy identifies elements such as Fe, O, Cr, Ni, C, Na as major constituents.

4. Photomicrographs of exposed surfaces reveal a combination of grain boundary dissolution and deposits consisting of regular and irregular shaped particles mainly in the 0.1 to 10 μ dia range. Butanol washing of the exposed surfaces removes from the loop pipework a fine inactive black powder which is partially magnetic and which contains the elements Al, Si, Ca, Cr, Fe, and Ni. The predominant size range is 0.1 to 10.0 μ.

5. The experiments with iron have indicated some pseudo steady-state deposition rates for parts of a sodium circuit of around 0.1×10^{-3} mg/cm^{2}hr or 0.04 mils/yr, equivalent to 30 μ after 30 years. This is not to deny the occurrence of localised deposition in excess of this.

Transport of Mn-54

6. The transport of Mn-54 differed from iron in that with Mn-54 there was preferential migration to the colder parts of the circuit including cold traps and areas where temperature and/or geometrical discontinuities exist.

Corrosion and transport studies on pure cobalt

7. A corrosion rate equivalent to 0.07 mils/yr at 585°C, ~14ppm oxygen and 25 ft/sec has been obtained for this material. This low corrosion value which is about six times lower than the corresponding value for stainless steel is attributed to the inhibiting effects of iron and nickel deposits which were observed to form on the cobalt at equivalent rates of 0.18 and 0.03 mils/yr respectively. Co-60 deposits forming as peaks on the heat exchanger were capable of being removed by changes in sodium velocity.

Decontamination of active material

Information is being collected on the effectiveness of a variety of decontaminants for active deposits. In terms of the assumed criteria of maximum decontamination coupled with minimum iron removal a KOH solution seems at present to be the most favourable. Liquid sodium does not appear to be an effective decontaminant.

3. FUEL AND FISSION PRODUCT BEHAVIOUR

The UK programme on fission product behaviour was reviewed in 1971 for the IWGFR by Thorley and Davies (5, 6). Studies, at that time consisted of observations from the Dounreay fast reactor (DFR) primary circuit, supplemented by laboratory investigations of fundamental properties such as solubilities and adsorption kinetics in sodium systems.

Investigations since 1971 have concentrated in somewhat different areas with studies on fuel behaviour and fission product release within the fuel pin, release mechanisms to the circuit with particular reference to the behaviour of short lived fission products in relation to failed fuel detection systems.

A surveillance programme to monitor events and behaviour within the prototype fast reactor (PFR) has been set up. Assessments have been made of activity levels within the circuit due to fission product activities. Significant limitations have been met in the translation of information from the DFR primary circuit examination to future fast reactors, since much of the information from DFR is only applicable to that reactor. The driver charge of the reactor is a vented metallic uranium-molybdenum alloy fuel. This results in a high background circuit activity against which the relatively small perturbations produced from the various experimental fuel pins are difficult to observe.

Release of gaseous and volatile fission products from oxide fuel.

The release of stable fission product gases from experimental fast reactor oxide fuel pins has been measured by fuel element puncture analysis of the plenum gases. The amounts released depend upon burn-up, fuel fabrication route and fuel configuration. Values range between 50-90% release for fuel in excess of a few per cent burn-up; a systematic dependence of gas release against burn-up has been observed for vibro-compacted fuel(7).

Extensive movement of caesium has been observed in oxide fuel pins even at low heat ratings. Most observations have been made on fuel which is stoichiometric or slightly sub-stoichiometric (mean O/M ratio 1.98-2.00 clad temperatures 300 to 550°C). Concentrations near the fuel periphery are typically two or three orders of magnitude greater than those near the pin axis. The extent of axial migration in the intact pins is limited. The migration to the periphery is taken as evidence of release from the fuel indicating qualitatively that releases of caesium are of similar magnitude to those of the stable gases.
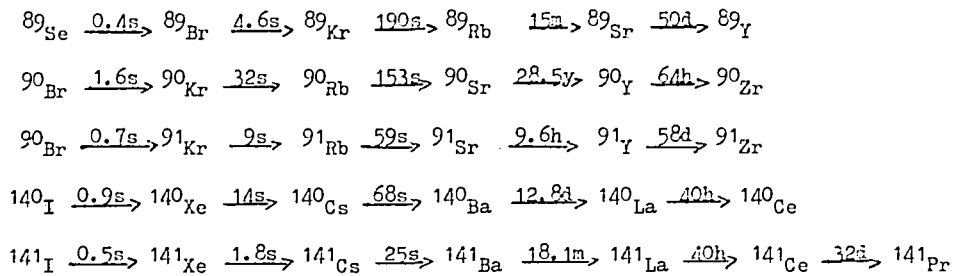
As the release of short lived volatile fission products is of interest to failed fuel detection systems. An experiment has been undertaken in DFR with a special instrumented fuel pin equipped with a gas flow system continually purging the plenum space. The experiment was run to 2% burn-up and the emission of gases from 3 minute half life up to 5 days was measured and compared with stable gas release(8). These experiments have been supported by small scale experiments conducted in a Materials Testing Reactor (MTR) in which the release of short lived and volatile fission product species has been measured from small specimens heated electrically and irradiated to high burn-up at fast reactor ratings(9). This study has shown that the initial stage of the release process from the site of generation to the surface of the fuel grain may be defined by a diffusion controlled process for which values of activation energy and diffusion coefficient may be assigned. Comparison of the emission of differing species demonstrates further that, to a first approximation, the release is controlled by half life in the manner expected from diffusion theory and that a single relationship describes the release of xenon, krypton, iodine and, within rather broader limits, tellurium. This behaviour is demonstrated in Fig 12.

The formulation of a model based on the diffusion data obtained from the small scale experiments which, when combined with the temperature profiles and form factors within a fuel pin allows an upper estimate of releases within the pin to be assigned. The predictions can only be regarded as upper estimates as the only delay in the release process is that afforded by the fuel grain. Some additional attenuation of release is expected due to delay within the pore structure or gas passages within the pin. Estimates of releases based upon these assumptions are shown in Table 1. A comparison with predicted and measured releases from the DFR experiment previously described(8) is shown also. Agreement is good for species greater than 9 hour half life, but becomes progressively worse at the shorter half lives presumably due to the increased effect of delay within the gas passages of the pin experiment. In the DFR experiment, however, some of the subsequent delay may have been due to the connection via a short length of small bore piping between the fuel pin and the collector chamber from which the gas was sampled.

Release of non volatile species

The amount of quantitative evidence that has been obtained on the migration or release of non-volatile species from fast reactor fuel is small. Some evidence from DFR on the stability of lanthanide species is discussed in subsequent sections on release to the sodium circuit. The behaviour of the noble metal fission products is well known from the characteristic inclusion phases which are formed extensively throughout the fuel(10). Examinations of the fuel matrix have shown that in the high temperature regions the majority of noble metal fission products are in the inclusions: the same is probably true at the lower temperature although this cannot be resolved from the surrounding matrix. The high chemical stability of these compounds makes further migration interaction improbable unless the oxygen potential is increased substantially above that found in the stoichiometric fuel.

A release mechanism via a volatile precursor activity has been shown to exist in small scale MTR experiments, where fission product deposits from gas flows passing over the specimens have been analysed. The more important fission product chains showing this effect are:

$$^{89}Se \xrightarrow{0.4s} {}^{89}Br \xrightarrow{4.6s} {}^{89}Kr \xrightarrow{190s} {}^{89}Rb \xrightarrow{15m} {}^{89}Sr \xrightarrow{50d} {}^{89}Y$$

$$^{90}Br \xrightarrow{1.6s} {}^{90}Kr \xrightarrow{32s} {}^{90}Rb \xrightarrow{153s} {}^{90}Sr \xrightarrow{28.5y} {}^{90}Y \xrightarrow{64h} {}^{90}Zr$$

$$^{90}Br \xrightarrow{0.7s} {}^{91}Kr \xrightarrow{9s} {}^{91}Rb \xrightarrow{59s} {}^{91}Sr \xrightarrow{9.6h} {}^{91}Y \xrightarrow{58d} {}^{91}Zr$$

$$^{140}I \xrightarrow{0.9s} {}^{140}Xe \xrightarrow{14s} {}^{140}Cs \xrightarrow{68s} {}^{140}Ba \xrightarrow{12.8d} {}^{140}La \xrightarrow{40h} {}^{140}Ce$$

$$^{141}I \xrightarrow{0.5s} {}^{141}Xe \xrightarrow{1.8s} {}^{141}Cs \xrightarrow{25s} {}^{141}Ba \xrightarrow{18.1m} {}^{141}La \xrightarrow{40h} {}^{141}Ce \xrightarrow{32d} {}^{141}Pr$$

Material will be released to the extent of the gaseous pre-cursor; in many instances this is small due to its short half-life.

Fission Product release from failed pins

Information on fission product release to the primary circuit has been obtained from the post irradiation examination of DFR experimental failed fuel. The determination of residual caesium from a number of failed fuel pins has revealed a consistent pattern in which between 10 and 20% of the generated caesium is remaining. This confirms the conclusion that caesium releases are of similar magnitude to the stable gases and demonstrates further that the released caesium is readily released through the defect. The general experience is that sodium has been drawn into the fuel pin and the plenum space generally contains at least some NaK (the DFR coolant is NaK). Little can be said about the rate of caesium extraction from the pin since the time of failure is not generally known.

Information on the behaviour of lanthanide elements has been obtained from microsamples taken from cross-sections of intact and failed DFR fuel pins. Measurements on ^{144}Ce are presented below as the ratio of observed to calculated concentrations together with the standard deviation on the observations. Calculated values are obtained from a knowledge of fuel rating and burn-up taken with the appropriate fission yields.

	Ratio ^{144}Ce observed: ^{144}Ce calculated average	standard deviation	Number of observations
Intact pins	0.989	0.069	39
Failed pins	0.976	0.048	12
All pins	0.986	0.061	51

These results suggest that there are no significant differences between failed and unfailed fuel: samples in the region of the defect did show a greater scatter, however, than those from the bulk of the fuel pin. The results confirmed that no axial or radial migration of cerium occurred even in highly rated oxide fuel. A slightly different picture emerges for neodymium where evidence of some depletion of Nd was obtained giving observed/calculated ratios of Nd down to 0.85 within 100 mm of a defect. Comparable losses of other rare earth elements seem probable and the effect may be associated with compound formation between sodium and rare earth elements.

The release of short lived gaseous fission products, and delayed neutron emitting activities have been measured for the calibration of failed fuel detection equipment. Experiments under conditions of flowing sodium have demonstrated that releases from a failed pin exceed the recoil emission from the exposed area of fuel. This conclusion is to be expected since release to the free volume within the pin will occur by diffusion as described in the previous section and by fission recoil. The delayed neutron activities are principally halogens which have an appreciable solubility directly in the coolant[11], and may be expected to pass rapidly into the sodium coolant. A similar mechanism will be operative for the release of fission product gases via their halogen precursors which in many instances have appreciable half-lives. Studies of fission product gas release usually measure the combined release of the gas itself together with that released directly from the fuel as the halogen precursor. It has been shown that the proportion released via the precursor is in many instances a substantial fraction of the total emission[12].

Loss of fuel from failed elements

Loss of fuel material from failed pins by spalling, coolant corrosion or compound formation is a potential source of appreciable coolant activity. Accordingly, experimental fuel discharged from DFR is routinely examined for failures and evidence of fuel loss. The most direct measurement is by comparison of pre and post irradiation weights but this is subject to uncertainty due to the loss of a significant weight of fission product gas (>0.5 g in some high burn-up pins) and because, after failure, variable amounts of reactor NaK are drawn into the fuel pin. These uncertainties lead to the situation that losses of less than 0.1 g of fuel are not readily detected. The extent of fuel loss from failed fuel in DFR has been assessed by this means. In 90% of the cases examined, fuel losses were below this limit of detection. In the 10% of failures where significant fuel loss was observed a common feature was the appearance of clad melting due to local overheating.

The degree of fuel loss is likely to be influenced by the occurrence and extent of sodium uranate formation between the oxide fuel and the coolant. However, the extent of reaction has been shown to be limited by achieving low oxygen potential conditions and some control of the reaction can be achieved by these means.

Fuel and fission product behaviour in the circuit

Much of the UK experience on fuel and fission product behaviour in sodium circuits has come from DFR and has been described previously[6]. This work is, however, of limited application due to the design of the driver charge of the reactor. The evidence from DFR has presented a clear picture on the behaviour of caesium; it is apparent that the majority will be released to the circuit. This will represent a major source of activity in the coolant. Further, due to the long half-life of ^{137}Cs, the activity will build up throughout the life of the reactor cumulatively from successive failures.

The adsorption of caesium onto steels under sodium has been studied in the UK[13]. Adsorption was shown to occur reversibly but there are considerable variations both in the extent and rate of adsorption which may be due to variations in the deposition surface. Complete desorption was found at $800^{\circ}C$. Evidence for the deposition of caesium has been obtained from the analysis of DFR cold trap baskets. Caesium is found at greater levels than expected from that associated with the retained coolant in the basket, but still at very low concentrations compared with the total caesium in the circuit. The complete removal of the activity has proved difficult possibly due to adsorption or the formation of a caesium chromite phase. Caesium enrichment in the vapour phase over the primary coolant is expected from the higher volatility than sodium. Values of relative volatility α_{Cs} of between 60 and 100 are expected under reactor conditions where α = mole fraction Cs in gas/mole fraction Cs in liquid.

The behaviour of iodine in the circuit has received particular attention because of its importance as a potential radiological hazard. Thermodynamic calculations have shown that iodine should exist as iodide in liquid sodium[14]. Methyl iodide has been shown to be unstable with respect to sodium; caesium iodide similarly is expected to decompose to sodium iodide at the concentrations expected under reactor conditions[15]. Some evidence for the collection of iodine as iodide within the reactor cold trap has been obtained from analysis of the DFR cold trap baskets. This observation is in accordance with the prediction from studies of sodium iodide solubility measurements[11]. The conclusions reached on the reversible adsorption of iodine and its ease of removal by simple decontamination procedures[6] are confirmed by experience from the DFR cold trap baskets.

Some attention has been given to the behaviour of tellurium for which a relatively high solubility in sodium at low temperature has been reported indicating that the material is unlikely to precipitate in the cold trap. Significant levels of tellurium have been observed in DFR cold trap baskets however. This activity is not readily removed by mild decontamination procedures suggesting that the tellurium is adsorbed or bound to the steel surface in a relatively firm manner. The behaviour of other metallic species has not been of concern from circuit activity aspects. They do not feature prominently in the analyses of coolant sodium, probably due to their low solubilities and high retention within the fuel element.

Several of the fission product elements and indeed the fuel itself, can be shown by thermodynamic calculation to exist in the oxide form in contact with liquid sodium containing typical oxygen levels of a reactor coolant. These elements include the lanthanides, alkaline earths, zirconium and niobium, and they are expected to show very low solubilities in the sodium. Some complication, as already discussed for (U,Pu) oxide, may be encountered due to formation of compounds with sodium of the double oxide type. Although not proven, this behaviour is unlikely to have a large effect on the insoluble nature of these species. The principle concern for these species, therefore, lies with their behaviour as particulate activity. This is borne out by the experience from the DFR primary circuit where it is believed that the principle oxide forming fission products are present as a suspension of fine insoluble particles in the coolant, which tend to deposit on metal surfaces[6]. This view is supported by the analysis of cold trap deposits where some accumulation of this material occurs and is relatively easily removed by simple washing procedures.

Activity assessments within a fast reactor primary circuit

In order to assess the relative importance of the different fuel and fission product activities to the problems of circuit activities, assessment has been undertaken using the configuration of a 1300 MW(e) fast reactor system. With the present state of knowledge, there are a large number of uncertainties, both in the releases to the circuit and the subsequent behaviour within the circuit. Simplifying assumptions have, therefore, to be made in several areas. The number of failed pins that can be tolerated in the reactor is unknown. Some compromise operating philosophy is likely to emerge from experience once the reliability of the fuel and the consequences of failure are fully appreciated. Currently these factors are unresolved, but operation experience from experimental pins in DFR suggests that burn-ups exceeding 10% may be achieved reliably without failure. Calculations here have been undertaken for continuous operation with one failed pin; releases for operation with larger numbers can be obtained by direct scaling.

The first step in assessing activity build-up within the circuit is to define the release from the failed pin. For this to be done effectively, all the mechanisms outlined must be considered for the various elements concerned. This exercise has been attempted in a UK assessment with the conclusion that the principle mechanisms for release, at least for the more severe failures will be fission product diffusion from the fuel to the free space within the pin and transfer to the coolant and fuel loss with its accompanying burden of fission products. Fission product diffusion is assessed either directly from experimental fuel pin experience in DFR or by using the diffusion model described to set maximum releases for the shorter lived activities. Fuel loss is taken as 1% of the fuel weight which is in accordance with DFR experience for the more severe types of failure. On average this latter figure is likely to be an overestimate. These assumptions are incorporated in the fuel pin release factors given in table 2 for the principle fission product activities. These were then applied to a fission product inventory calculation from standard UK computer codes for a single fast reactor pin containing 150 g of fuel as metal and taken to a mean burn-up of 7.0% at a mean fuel rating of 180 $W.g^{-1}$. The activities per pin are given in table 2 for conditions of 15 day and 1 year cooling periods which are taken as appropriate times for maintenance operations.

Further factors that have to be considered to obtain activity levels plated in the circuit are the build-up of activity due to successive failures and plate out characteristics around the circuit. The dwell time of a subassembly within the reactor will be approximately one year and, as failure is more likely at end of life, it is assumed that it remains in the failed conditions for the last 3 months of operation. Thus continuous operation with one failed pin on this basis means 4 failures per year or 40 failures summed over a 10 year period. For an element such as caesium the activity released to the circuit will be that from the whole burn-up period rather than just that generated whilst the pin is in the failed condition. The question of plate out is highly complex and, in the absence of reliable information, the calculation for the activity deposition in an intermediate heat exchanger (IHX) is simplified by the assumption that the fission product activity deposits within the IHX's and not elsewhere. This assumption is justified on the basis of the very large area afforded by the IHX's compared with elsewhere. The 1300 MW(e) reactor is assumed to have 6 IHX's. Calculations of the γ dose rates developed within one single IHX from fission product activity after 10 years continuous operation with one failed pin are presented in table 3, for decay time of 15 days and one year. Two plate out conditions are considered: One is 100% plate out of all activity including caesium. The other is 0.1% plate out of species miscible with sodium, eg Cs, Rb, I, Te, Ag, Sb, Sn; all other species plate out 100%. The table demonstrates the overriding importance of caesium and its plate out behaviour. Should the 0.1% plate out for caesium be justified, then the behaviour of other activities such as Ru/Rh, Zr, Nb and Ce/Pr become significant. It is of note that, whilst several activities are decayed after one year, several of the longer lived isotopes persist and give rise to appreciable activity levels.

Future work programme

The principle studies on fission product behaviour in primary circuits will be undertaken in PFR for which an extensive programme of measurements are planned. This includes coolant monitoring for fission product activity, examination of deposition foils from various places within the reactor, and the examination of components removed routinely or for special purposes during the course of reactor operation. A series of artificially defected pins will be irradiated early in reactor life to establish detection sensitivity and resultant activity in the circuit.

These reactor studies will be supported by some materials testing reactor experiments of defected fuel pins and other fuel specimens in sodium capsules for the detailed examination of fuel sodium reaction phases and the amount and nature of fission product phases entering the sodium. The control of fuel sodium reactions in oxide fuel is being investigated as part of the UK fuel development programme by the determination of oxygen potential changes with burn-up and evaluation of oxygen redistribution phenomena as a function of mean oxygen to metal ratio.

Laboratory work on the deposition of caesium will be undertaken to determine the extent and mechanism of caesium uptake by steel surfaces. Subject to priorities, some studies of fission product behaviour will be mounted in some of the UK out-of-reactor sodium loops. Further work on fundamental properties such as solubilities and the behaviour of oxide and metal particulates in sodium will be undertaken as effort allows.

4. THE ROLE OF SODIUM CHEMISTRY IN CORROSION AND MASS-TRANSFER PROCESSES

It has already been shown in an early part of this paper that the mass-transport of materials in sodium is sensitive to the levels of impurity in the system. Notable impurities are oxygen which effects the corrosion rate of stainless steel and carbon which can interact with certain elements such as Fe and Mo to form spallible carbides of the M_6C type (Fe_4Mo_2C). Therefore from the standpoint of minimising mass-transport effects in reactors a knowledge of how these impurities behave in large systems is required along with reliable methods of measurement and control.

In the UK, basic sodium chemistry studies are conducted principally at CEGB, BNL and AERE Harwell. For the purposes of this paper these studies may be divided into the categories.

a) Analytical techniques for measurement of impurities in sodium in terms of both thermodynamic activity and concentration.

b) Control of impurity levels.

c) Solubility studies and determination of the nature of species in sodium.

Analytical Techniques

Trevillion (BNL) has recently written a comprehensive review[16] of sodium analytical techniques, and work at BNL is largely concentrated on those topics which were recommended for further study in the review. In the context of fission and corrosion product behaviour in sodium circuits, oxygen is considered to be the principle impurity of interest.

Oxygen Analysis

The following techniques for oxygen determination are being investigated in the UK:

(i) Vacuum distillation: Trevillion (BNL) has conducted analyses of vacuum distilled samples taken from an in-line sampler on a bench-top electromagnetically-pumped loop[17]. The aim of the experiment was to determine the effects of cold trap operation and of sampler design and operation on the analytical figures and the corresponding blank analyses. Optimised operating conditions resulted in a blank value of + 1.5 ppm on concentrations in the range 3-5 ppm.

At higher oxygen concentrations (up to 25 ppm) the blank analyses were higher, this was rationalised in terms of particulate migration from the cold trap and it must be concluded that the precision of vacuum distillation analysis may be compromised by the presence of particulate material.

In order to make vacuum distillation more specific for oxide, methods for determining other species are being developed. A technique for determining carbonate has been developed by Gwyther and Trevillion. This involves titrating coulometrically the distillation residue for total alkalinity, slightly acidifying to liberate CO_2, removing the CO_2 by bubbling argon and back-titrating coulometrically.

(ii) Equilibration techniques: Hooper and Trevillion[18-20] have made a theoretical assessment of the vanadium wire equilibration technique and it is found that thermodynamic equilibrium was not achieved in the application of the technique as described by workers in USA, and that the technique is non-general. Experimental studies at BNL confirm the theoretical assessment; diffusion barriers are formed at the vanadium surface at oxygen levels in sodium of 4-20 ppm (at $750°C$), producing an oxygen concentration gradient in the specimen, and at very low oxygen levels the peritectoid phase "V_9O" precipitates on cooling the equilibration wire subsequent to exposure.

Consideration of niobium and tantalum as alternative equilibration metals (in Ref 18) shows that, in the absence of interfering reactions, the measurable range of oxygen activities in sodium is greatly increased compared to that for vanadium. However, application of these metals demands accurate measurement of much lower concentrations of oxygen in the refractory metal compared to the case for vanadium.

(iii) Electrochemical cells: Extensive programmes on oxygen meter development are being carried out both at AERE Harwell and at CEGB BNL; in many ways these programmes may be considered complementary.

At Harwell, Asher et al[21] are testing meters which incorporate $ThO_2 - 7.5$ wt % Y_2O_3 electrolyte, fabricated in tube form in the Metallurgy Division, and a platinum/air reference electrode. The aim of these tests is to optimise both electrolyte tube production and meter-mounting technology in circulating sodium systems. To date 6 meters have been tested at ca $405°C$ in a small thermosyphon loop. The longest lifetime achieved was 2040 hours and the other 5 meters failed after much shorter periods. All the meters showed a drift in voltage output but, more importantly, some gave an output which could not be rationalised in terms of exposure conditions and sudden fluctuations in output were superimposed on the steady drift.

At BNL, Simm and Smith (22, 23) are testing meters which incorporate $ThO_2 - 7.5$ wt % Y_2O_3 electrolyte, fabricated in tube form by Zircoa, and a platinum/air reference electrode. The aim of these tests is to evaluate long-term meter performance with respect to output stability, achievement of theoretical emf's and reactor application.

Three meters have been tested; two are still functioning well after 7500 and 5000 hours respectively, the third failed after 5500 hours as a result of studies on the effect of cycling the temperature gradient along the tube. The meters are calibrated by making known additions of oxygen gas to the known (and constant) amount of sodium, after an initial uranium-gettering has reduced the indigenous oxygen level to a neglibible amount. Important observations are:

1) For approximately the first 800 hours the meter output is subject to a steady upward drift averaging 3.6×10^{-5} volts/hour.

2) After this 800 hour period the meter voltage becomes very stable and the emf's generated are close to theoretical if the equation for solubility of oxygen in sodium due to Noden (BNL)(24) is applied to the oxygen additions.

3) The correlation with theoretical emf's could be improved further by increasing the length of electrolyte tube exposed at temperature.

4) The oxygen activity of sodium is strongly affected by hydrogen. Over the range of conditions studied the oxygen activity was suppressed by ca 10% on addition of an effective 1 ppm hydrogen (measured using the hydrogen meter — see below). It is considered that this would be the discrepancy attached to vacuum distillation analyses under these conditions.

5) For a reactor instrument having good lifetime and stable output, at least 3 requirements must be met. The operating temperature must show variations $\leq \pm 1°C$ from the set temperature (in the range 400–500°C). The rate and extent of changes in ambient temperature must be minimised. A method of periodic recalibration must be devised; use of a characterised cold-trap in the meter sub-loop is favoured for practical reasons.

(iv) γ-photon activation analysis: Thompson and Hartlib (Harwell) are developing a technique in which drip samples of sodium are taken through a "gate valve" system and sealed in situ for subsequent activation analysis. This analytical route may reduce contamination problems inherent in other analysis techniques.

Carbon Analysis

Analytical techniques for carbon have also received a good deal of attention in the UK.

At Harwell, Asher, Kirstein and Tolchard are developing a carbon meter which works on the following principle: Carbon in sodium diffuses across an α-iron membrane and reacts with an oxide film, which has been grown on the other side of the membrane, to form carbon monoxide. The carbon monoxide is swept away by an inert gas stream and analysed quantitatively. In conjunction with tests on the meter, considerable experience has been gained on the use of nickel equilibration tabs for carbon activity determination.

At BNL, Gwyther and Hobdell are developing an electrochemical carbon meter. This similarly incorporates an α-iron membrane which allows diffusion of carbon from the sodium. Within the membrane is the molten $Na_2CO_3-Li_2CO_3$ electrolyte and a centrally positioned reference electrode of α-iron saturated with carbon.

Both the Harwell and the BNL carbon meters will be inserted into the PFR primary circuit.

Hydrogen Analysis

As outlined above, hydrogen may influence significantly the oxygen activity of sodium. An electrochemical hydrogen meter has been developed at BNL(25). The cell incorporates iron membranes, which allow diffusion of hydrogen. The membranes separate the sodium and the reference hydrogen atmosphere from the calcium hydride/calcium chloride electrolyte. Cell emf's are 95% of theoretical.

Control of Impurity Levels

The principal means of controlling impurities in LMFBR sodium circuits remains cold-trapping. Hebditch, Haigh and Gliddon(26) (CEGB Marchwood Engineering Laboratories) are studying cold-trap performance from basic principles in order to improve understanding of processes necessary for efficient cold-trapping, particularly with respect to sodium oxide. Parameters which affect crystal nucleation and growth and mass transfer coefficients are defined. The relation of crystal growth and mass transfer theories is discussed and the error in making mass transfer/heat transfer analogies is stressed. It is concluded that improved cold-trap performances should be possible if the controlling crystal growth processes are properly understood; there is scientific evidence in favour of mesh-packed cold-traps. Finally, these workers apply their theories to data due to McPheeters(27) and obtain a better fit for the clean-up curve compared to McPheeters own treatment.

At BNL the nature and kinetics of interactions between other non-metal impurities in sodium (hydrogen and carbon) and sodium monoxide are under investigation. These studies should lead to better understanding of the species controlling cold-trap behaviour. Also workers at BNL in collaboration with DERE are analysing PFR primary circuit cold-trap deposits by techniques which avoid possible decomposition of species of interest (for example, extrusion of a rod-form sample of the PFR sodium, containing the precipitate of interest, for X-ray crystallographic analysis).

The ability of the plugging meter to monitor the tendency for species to precipitate, possibly causing reductions in heat transfer or blockage, makes the device a useful reactor instrument. At REML (Refs 28 and 29) both automatic and manual plugging meters (APM and MPM) have been developed. In the presence of a number of precipitating species the APM takes a considerable time to reach the highest temperature of interest and unstable traces may be obtained for precipitates having poor mechanical stability. Both these difficulties can be overcome using the MPM, and use of the APM and MPM as complementary instruments provides a comprehensive picture of all the saturation temperatures in a system.

Solubilities and Nature of Species in Sodium

Iron solubility

Workers at Harwell are studying the solubility of iron in sodium. In these experiments ^{59}Fe is used as source and sampling is carried out at temperature by puncturing the containment capsule. A straight line relationship is obtained between log [solubility] and reciprocal temperature using sodium gettered by uranium foil and the values obtained are similar to those of other workers. At high oxygen activity (ca $\frac{2}{3}$ saturation) a parallel relationship is obtained but the iron concentrations are typically 20 times higher; for example, 0.75 ppm at 400°C and 70 ppm at 700°C compared with 0.036 ppm and 3.5 ppm in the gettered series. Furthermore, a linear relationship between log [solubility] and log [oxygen activity] is obtained at oxygen activities above 0.1 (estimated on the basis of solubility data).

The deposition rates of iron from saturated solution onto nickel has also been measured in sodium gettered by uranium and in sodium having oxygen activity 0.6 on the basis of solubility data. The observed deposition after 48 hours is shown below:

Temperature (^{o}C)	Fe Deposition $a_{O_{Na}}$ 0 (ug cm^{-2})	Fe Deposition $a_{O_{Na}}$ 0.6 (ug cm^{-2})
400	0.1	10
600	0.6	1000
800	1.0	80000

Iron particulate formation has also been studied at 727°C as a function of oxygen activity. Iron is added as Fe_2O_3 and the behaviour of the free iron following reduction is observed. At high oxygen activities large crystals (2–3 mm long) are formed after two days; crystal growth of this type does not occur in gettered sodium.

Carbon Movement and Carbide Formation

Studies of this topic are being conducted both at Harwell and at BNL in conjunction with carbon meter tests. At Harwell the carbon uptake in nickel foils and the stainless steel pipework is measured at various positions and hence temperatures in a thermosyphon loop.

At BNL the effects of sodium at different carbon activities on the composition and microstructure of 316 steel are being studied. The carbon activity in a typical isothermal capsule test is set constant by use of a selected carbon source or sink and is monitored with the electrochemical carbon meter. Results of the experiment at unit carbon activity have appeared in the open literature (30).

The carbon meter has been used to follow carbide transformation in steels exposed to sodium (31) and a programme is underway to define the composition of carbides, with respect to substitutional elements, as a function of carbon activity.

Other work at BNL, which will be described in more detail in a separate presentation (32) is intended to improve understanding of the role of sodium chemistry and metallurgical structure in determining mechanisms and kinetics of corrosion and deposition processes. Briefly these studies may be categorised as follows:

1) The effect of sodium chemistry on steel microstructure is being studied.
2) The effective effusivities and diffusivities of both substitutional and interstitial elements in corrosion-affected surface layers, and in deposition regions are determined.
3) Compound formation. In addition to the studies on carbide formation outlined above, the thermodynamics, structure and growth mechanisms of compounds such as ternary oxides and intermetallics are being studied.

5. REACTOR SURVEILLANCE

In addition to the laboratory studies a programme of work is in hand at DERE to study corrosion and deposition in various parts of the Prototype Fast Reactor (PFR). These studies are mainly concerned with the measurement of selected fuel pins, the measurement of small lengths of fuel pin (mini-pins). This is to establish more accurately changes along the length of a full-sized pin, and the measurement of specially prepared metal coupons. All these different samples are situated in the core of the reactor so that

corrosion and deposition can be assessed under realistic conditions of irradiation, coolant flow, impurity level and temperature.

Complementary to the core surveillance programme a study is also to be undertaken to evaluate material transport in other parts of the primary circuit. This programme, aims to monitor both materials behaviour and the coolant composition in various parts of the reactor circuit. Typical examples of the position identified for this work are:

a) inlet and exit positions below and above the reactor core,

b) positions away from the reactor core such as the inlet to the IHX and the sodium pool,

c) a position where specimens can be exposed to both cover gas and liquid sodium so as to investigate 'interface' effects, and

d) the cold-trap facility.

Coolant samples will also be extracted from the inlet to the IHX, the sodium pool and the cold-trap.

The types of measurement which will be carried out on the various samples will involve both weight and diametral changes followed by metallographic examinations to establish the structure and thickness of any deposited or depleted layer. Where necessary certain specimens will proceed for further analysis using microprobe and activation techniques for an assessment of any concentration changes. During the cleaning of the sample the various solvents will be monitored to ascertain whether removal of deposits, fission products or fissile material occurs during this part of the handling procedure. Also separate specimens will be further treated using acid pickling solutions to remove successive layers from the sample to provide a check on deposit thickness and also to estimate the levels of penetration of any active constituents.

6. SUMMARY OF THE UK POSITION

Corrosion and Activity Release

Bulk corrosion of fuel clads Provided REML predictions of corrosion rate are confirmed by the PFR data the situation with respect to bulk corrosion appears to be satisfactory. Using the best estimates of corrosion rates provided by REML and appropriate details of design and operating conditions, definitive estimates of the quality of material released to the coolant as a function of oxygen content and clad temperature can be prepared, a) to assist calculations of activity release, and b) to allow more realistic assessment of hydraulic as well as activity problems arising as corrosion material deposits accumulate.

Activity Release from Clad/Structural Materials

Uncertainties in activation cross-sections added to the uncertainties in bulk corrosion rates raise the overall uncertainty in activity release rates to ± 75%. Error factors ranging between 2 to 20 were reported for the reactions governing circuit activity – ie Fe54 Mn54, Ni58 Co58, Ni60 and Co59 Co60. These deficiencies are being remedied by AERE. To ensure strict comparability to activity release estimates from different sources, an appropriate body should examine activation cross-sections currently in circulation and issue an approved list of spectrum weighted or energy related cross-sections for general use in calculations. Additionally, measurement

of activity levels in relevant materials after irradiation under closely controlled conditions would provide an invaluable check on the cross-sections recommended.

Activity Release from Fuel Pins

The uncertainty in heavy atom and fission product yield data is generally within the range $\pm$ 20% and could be reduced to $\pm$ 5% as a result of experiments in PFR.

With the exception of the rare gases and certain volatile elements, the rates of release of fission products from a failed fuel pin are not based on experimental evidence, but are arbitrarily assigned on the basis of inferred release mechanisms. Fortunately, the release rate for Caesium 137, which because of its long half life dominates long term fission product activity levels, is better established than the majority.

The figures used for fuel loss are best guesses which, it is hoped, can be substantiated by data from PFR. The influence of coolant/fuel chemical interaction on fuel loss from a pin will, however, to appreciable and because of associated alpha activity hazards this factor may strongly affect decontamination and effluent disposal procedures. Evidence on the effects of fuel/coolant reaction is being sought, therefore, by examination of defect pins in DFR, PFR and MTR, by alpha activity measurements on PFR coolant samples and from laboratory studies of reaction kinetics at AERE. Methods of reducing interaction effects, eg by the use of low O/M fuel, are also being investigated in and out-of-pile.

Recent calculations[33] suggest that the long lived activity released to the coolant in large FR's in the form of corrosion products (estimated at 60 Kg/year from core pins) is 5 x 10⁴Ci, a figure which is matched by the activity arising from fission products at a pin failure rate of 200 per year. These figures assume reactor operation for 1 year and a 15 day decay period in each case. Because of the long half life of Caesium 137, however, fission product activity will overtake corrosion product activity, even at a considerably lower failure rate, towards the end of service life.

Deposition

Corrosion Products

The aims of laboratory experimental work in this area have already been outlined.

Tabs are to be inserted around the PFR primary circuit in an endeavour to monitor bulk corrosion product and activity deposition rates in different locations.

Crud collection clusters are provided to check on the particulate content of the coolant and chemical analysis of coolant samples will yeild information on the build-up of soluble impurities. Deposition within the core will be assessed qualitatively by inspection of fuel pins and sub-assembly internals and by analysis of anomalous temperature increases in sub-assembly coolant outlet temperatures. A special fuel cluster is proposed which is designated to furnish precise data on deposition, as well as corrosion rates within the core zone. Provision has also been made for extracting samples of sludge from the bottom of the reactor tank.

In the long term, integration of data from standard loops, the mass transfer loop and PFR will lead to the development of a generic model describing corrosion, transfer and deposition phenomena. If it should transpire that deposition is sensitive to environmental conditions such as surface quality, turbulence, flow history, temperature gradients and method of cold trap or reactor operation, then any alteration in the circuit materials the heat exchanger design, inlet and outlet temperatures and probably the cold trapping system may have to be evaluated on a large loop simulation.

The influence of cold trap operating and design characteristics on deposition processes is a relatively unknown quantity which merits study since the cold trap is, in principle, the ultimate sink for all coolant impurities, including corrosion products, fission products, titium and possibly fuel debris. Observations on the Harwell Mass Transfer Loop have shown clearly the preferential migration of important corrosion isotopes to the coldest section of a diffusion cold trap but evidence is otherwise lacking. Efficiency in attracting corrosion products could, therefore, be a crucial factor in determining designs of the cold trap and associated decontamination facilities.

It was noted that deposits in a standard loop reached a critical thickness then sheared off the substrate. Although this experiment was carried out under conditions which should not occur in a large reactor, this type of deposit behaviour should be investigated further since it has implications on reactor safety which may demand additional protection of the core against the sudden intrusion of debris.

Activity Deposition

Observations suggest variations in the deposition behaviour of active isotopes eg Mn appears to migrate preferentially to the cold trap while Fe59 and Co60 - and the bulk of the stainless steel - are likely to deposit in the cold zone immediately down-stream of the hot zone, ie in the lower part of the IHX. Thus activity monitoring is not necessarily a reliable method of locating bulk corrosion product deposits.

The Harwell Mass Transfer Loop also simulates heat exchanger tube conditions, but is simpler in construction than the REML rig, is manufactured from 316 stainless steel, accommodates active specimens and allows continuous monitoring of activity around the circuit. Decontamination techniques will be tested at the end of each active run.

Fission Product Deposition

Virtually no information on fission product or fuel material deposition behaviour is available although their location, chemical form and adherence may be critical factors in the determination of decontamination and effluent treatment procedures.

Caesium activity dominates fission product activity in the long term and may eventually exceed the activity due to corrosion products. Its fate, therefore, is vitally important. The possibilities include:

a) retention in the coolant - highly probable in view of its solubility in sodium; caesium activity in sodium withdrawn in or on components will, therefore, contribute to effluent disposal problems.

b) plate-out on stainless steel or other material surfaces; plate-out fractions as low as 0.1% are frequently employed in estimating dose rates but it is known that caesium may be adsorbed to a much greater extent under favourable surface energy conditions.

c) transfer of a proportion of the caesium to the blanket gas and its escape to the reactor hall by leakage, to the atmosphere during controlled discharges via the stack, and to the blanket gas clean-up plant.

d) transfer to surfaces above the coolant level as 'frost', which behaves as a solid phase at temperatures as high as 500°C. Since the vapour pressure of caesium is high relative to sodium, enhancement of caesium levels by one or two orders of magnitude above that of the coolant must be expected in 'frost' deposits.

e) plate-out or deposition in the cold trap; in view of the high solubility of caesium in sodium at all temperatures, deposition in the cold trap should not occur because of immiscibility considerations, but may plate-out on the extended surfaces within the trap because of surface reactions with stainless steel or impurities already precipitated thereon.

The Effects of Deposition on Circuit Hydraulics and Heat Transfer

Although simple calculations suggest that deposit build-up should not adversely affect heat transfer values the chief cause for concern is that marked surface roughness, or local build-up of deposits will lead to pronounced increases in pressure drop. Information is, therefore, required on the coolant flow, surface energy and temperature conditions promoting or obviating deposit irregularity. Certain of these aspects are currently being covered in the UK programme.

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4) Dr K Q Bagley FETD, for permission to reproduce item 6 'Summary of UK position' from one of his papers on this topic.

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TABLE 1

Emissions of Short Lived Fission Products from Fast Reactor Oxide Fuels Expressed as Per Cent R/B

Fuel	Centre temp °C	Isotope and Half-Life														
		^{85M}Kr 4.4h	^{87}Kr 78m	^{88}Kr 2.8h	^{89}Kr 3.2m	^{90}Kr 33s	^{91}Kr 10s	^{133}Xe 5.3d	^{135}Xe 9.2h	^{137}Xe 3.7m	^{138}Xe 17m	^{139}Xe 41s	^{131}I 8.1d	^{133}I 21h	^{132}Te 78h	^{87}Br 55s
PFR Pellet	1460	2.8	1.5	2.2	0.3	0.1	0.07	13.6	3.9	0.3	0.7	0.1	16.4	5.9	10.9	0.2
PFR Vibro	1800	5.5	3.1	4.5	0.6	0.3	0.1	21.8	7.7	0.7	1.5	0.3	25.1	11.1	18.5	0.3
DFR 238	2100	12.3	7.6	10.4	1.8	0.7	0.4	34.2	15.9	1.9	3.8	6.8	37.8	20.8	30.2	0.9
DFR 238 exptl. results	1.25-1.5% burn-up	3	1.0	1.5	0.03	-	-	30	7 (135M 1.2)	0.05	0.2	-	-	-	-	-
	1.75-2.0% burn-up	5	1.6	2.2	0.03	-	-	38	11 (135M 1.1)	0.05	0.2	0.004	-	-	-	-

TABLE 2

Principle Fission Product Activities and Pin Release Factors

One pin - 150 g metal at 180 Wg^{-1} and 7.0% mean burn-up

Activity	Half-Life	Curies per pin		Pin Release Factor
		15 day	1 year	
91-Y	58.51d	407	6.5	0.01
95-Zr	63.98d	711	17.3	0.01
95-Nb	35.15d	791	36.6	0.01
99-Mo	66.02h	27.8	-	0.01
103-Ru	39.35d	799	1.8	0.01
103m-Rh+	57 m	783	1.8	0.01
106-Rh+	30.35s	435	225	0.01
111-Ag	7.45d	17.4	-	0.5
125-Sb	3.89d	5.01	-	0.4
125-Sn	9.64d	9.03	-	0.5
127-Sb	3.89d	5.01	-	0.4
127m-Te	109 d	9.8	1.1	0.8
129m-Te	33.52d	27.1	0.02	0.7
129-Te+	69 m	19.4	0.02	0.4
131-I	8.04d	219	-	0.5
132-I+	2.28h	41.9	-	0.2
132-Te	78.2 h	41.8	-	0.4
134-Cs	2.07y	0.03	0.02	0.8
136-Cs	13.0 d	2.9	-	0.6
137m-Ba+	153.5 s	30.0	29.3	0.8
140-La+	40.2 h	532	-	0.01
141-Ce	32.5 d	614	0.35	0.01
144-Pr+	17.28m	415	176	0.01
147-Nd	10.98d	159	-.	0.01

NOTE:

+ denotes daughter product
Parent activities are listed below

103-Ru $t_{\frac{1}{2}}$-39.35d
106-Ru $t_{\frac{1}{2}}$-368.8d
129m-Te $t_{\frac{1}{2}}$-33.52d (0.634 of yield ---> 129-Te)
132-Te $t_{\frac{1}{2}}$-78.2h
137-Cs $t_{\frac{1}{2}}$-30.13y
140-Ba $t_{\frac{1}{2}}$-12.79d
144-Ce $t_{\frac{1}{2}}$-284.4d

TABLE 3

I.H.X. Dose Rates from Fission Product Activities

Accumulated doses after 10 years operation with one failed high burn-up pin having 3 month residence time.

Activity	Half Life	DOSE RATE - mR/h			
		100% Plate out		100% Plate out + 0.1% miscibles	
		15d decay	1 y decay	15d decay	1 y decay
91-Y	58.5d	0.2	0.003	0.2	0.003
95-Zr	63.98d	66.4	1.6	66.4	1.6
95-Nb	35.15d	42.7	2.0	42.7	2.0
103-Ru	39.35d	4.8	0.01	4.8	0.01
103-Rh	57m	3.1	0.007	3.1	0.007
106-Rh	30.35s	30.2	15.6	30.2	15.6
111-Ag	7.45d	2.6	-	0.003	-
125-Sb	3.89d	8.6	-	0.009	-
125-Sn	9.64d	13.5	-	0.013	-
127Sb	3.89d	12.0	-	0.012	-
127m-Te	109d	12.9	1.5	0.01	0.002
129-Te	33.5d	9.8	0.006	0.01	-
129mTe	69m	7.0	0.006	0.007	-
131-I	8.05d	383	-	0.4	-
132-I	2.28h	193	-	0.19	-
132-Te	78.2h	41.6	-	0.04	-
134-Cs	2.04y	2.3	1.7	0.023	0.014
136-Cs	13.0d	38.3	-	0.038	-
137m-Ba*	135.5s	5175	5170	5.17	5.1
140-La	40.2h	117	-	117	-
141-Ce	32.0d	5.52	0.003	5.52	0.003
144-Pr	17.28m	54.1	22.9	54.1	22.9
147-Nd	10.95d	1.91	-	1.91	-
Total		6225	5215	332	47.3

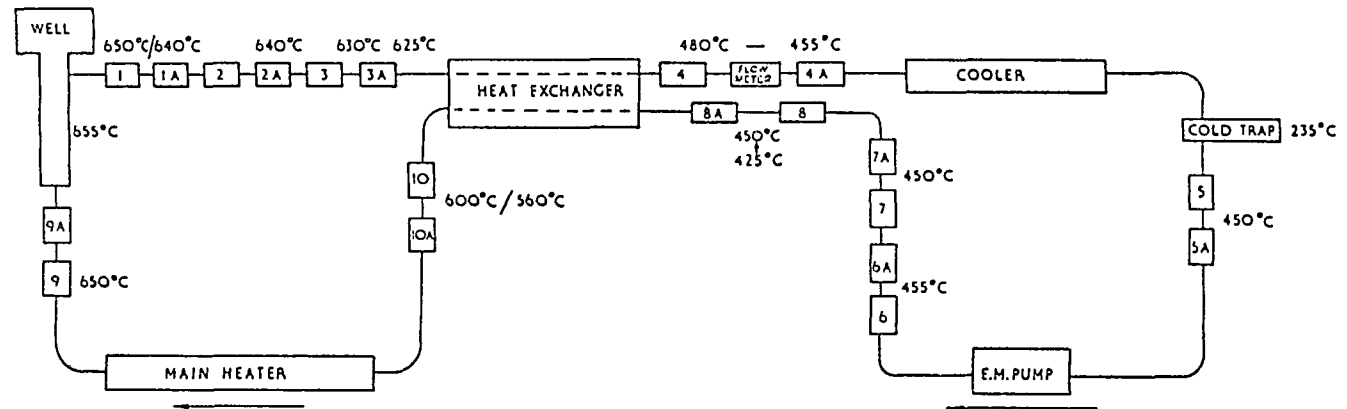* 10 year build-up of 137-Cs activity obtained from 40 failed pins.

FIG. 1.   SPECIMEN LAYOUT AND TEMPERATURE DISTRIBUTION FOR 99-125 WEEKS AT 13 ft/sec.
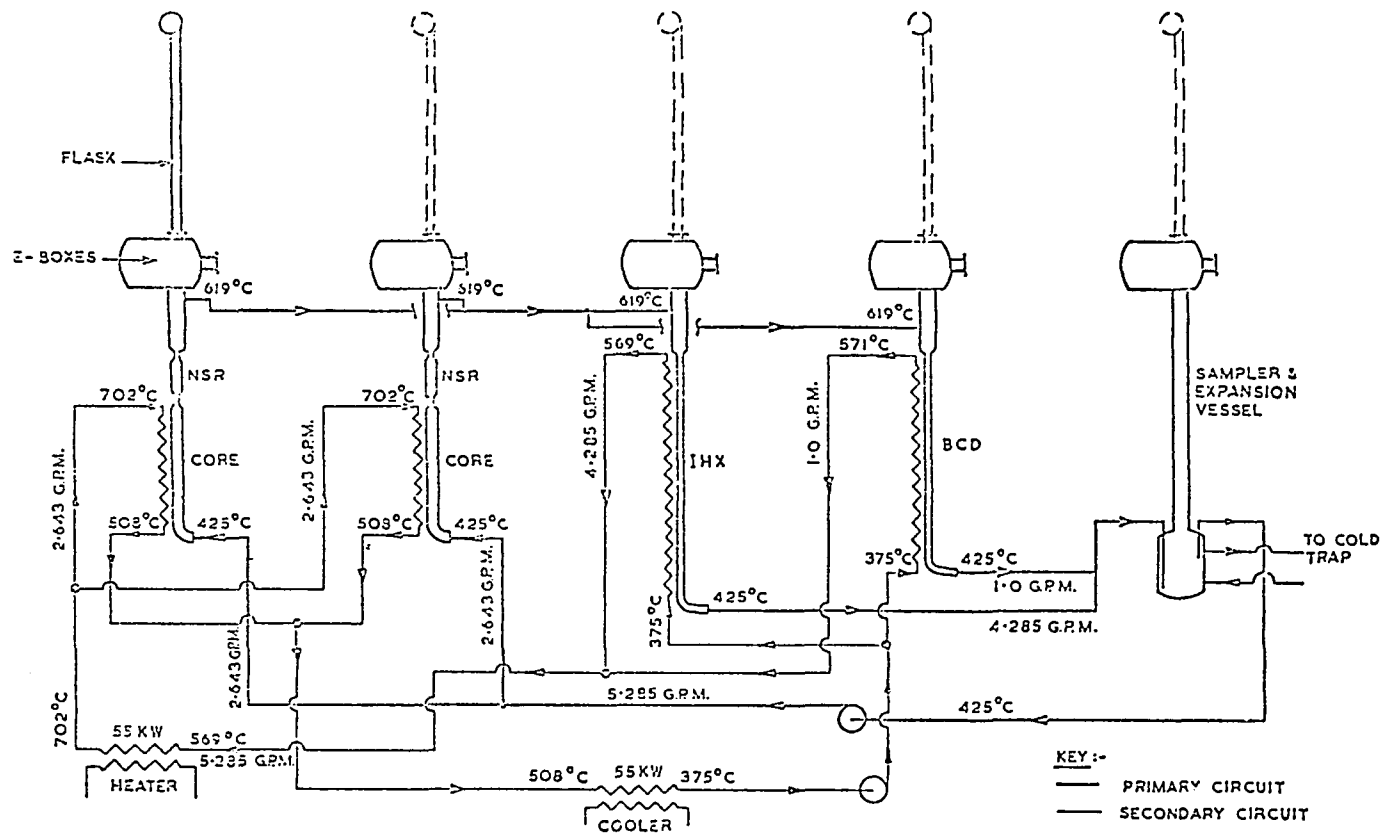

FIG. 2.   GENERAL ARRANGEMENT OF LARGE MASS TRANSFER LOOP

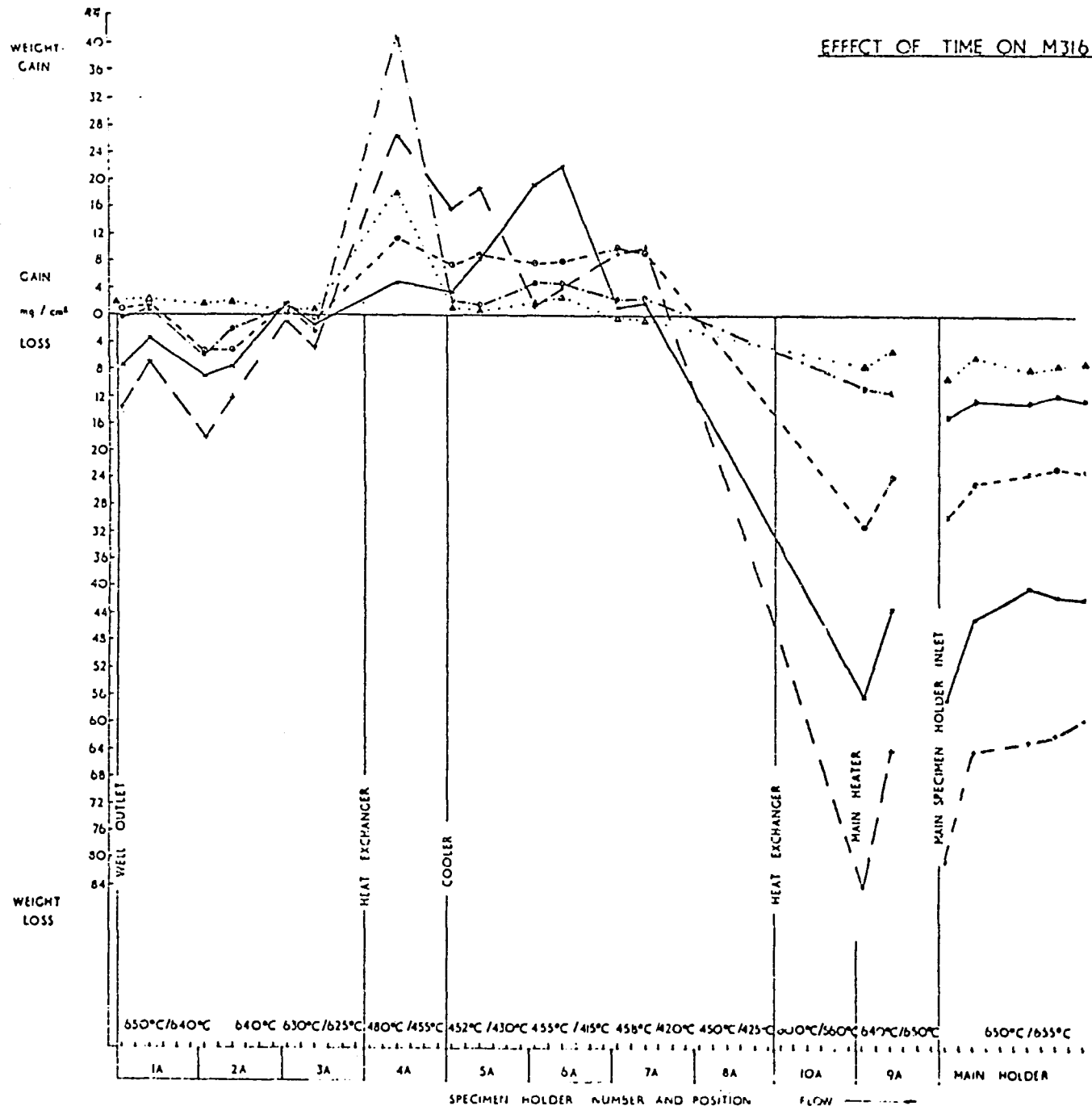EFFECT OF TIME ON M316 SPECIMENS 0.23" BORE ASSESSED AT

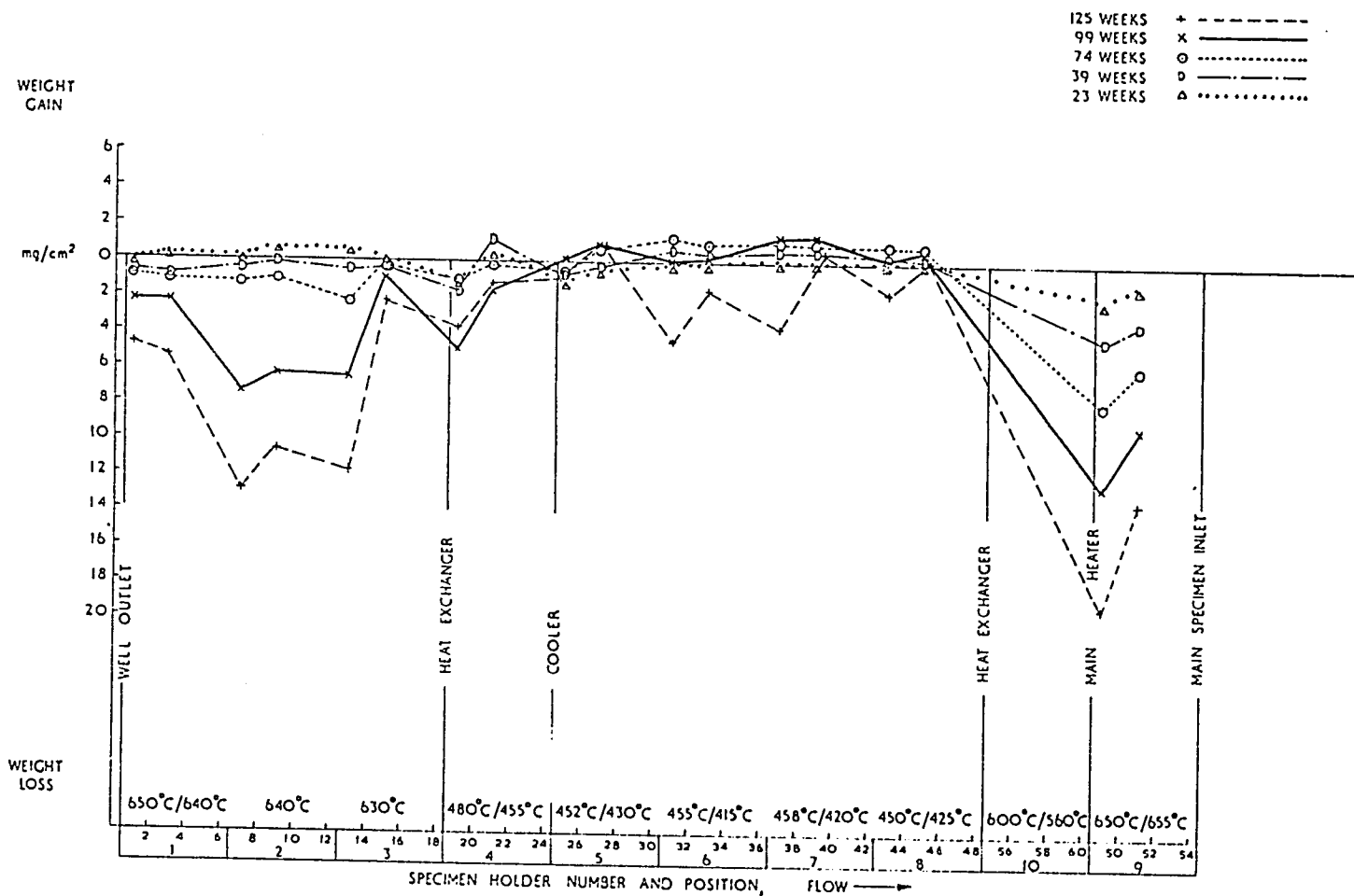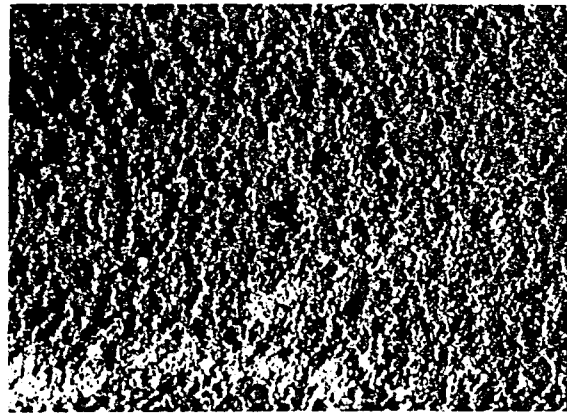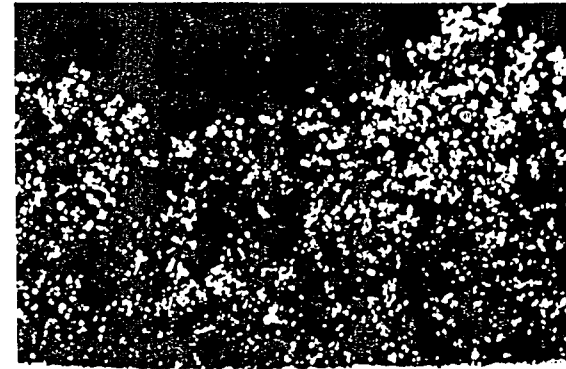23, 39, 74, 99 AND 125 WEEKS

FIG. 3.

FIG 4 EFFECT OF TIME ON TYPE 316 S.S. SPECIMENS OF 0·718" BORE. ASSESSED AT 23, 39, 74, 99 AND 125 WEEKS

FIG. 4.

DEPOSITION STUDIES – 316ss — 125 Weeks in holder 4A
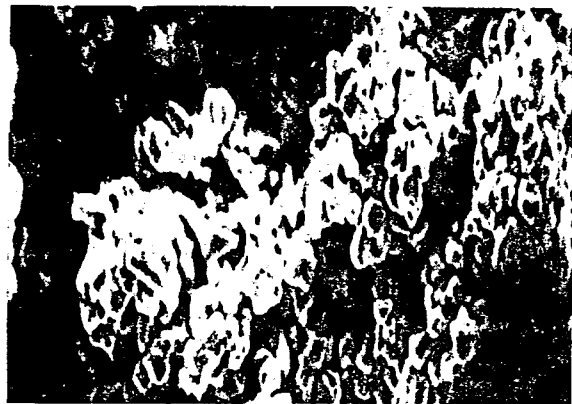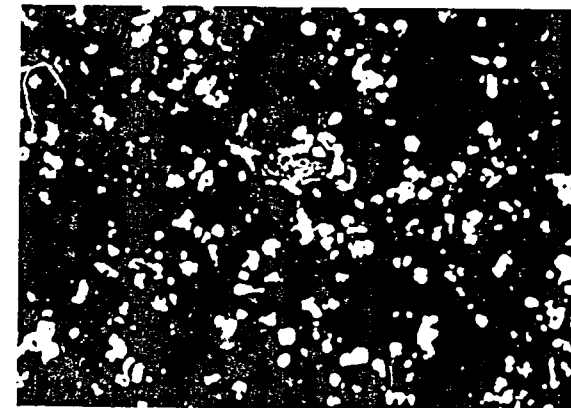

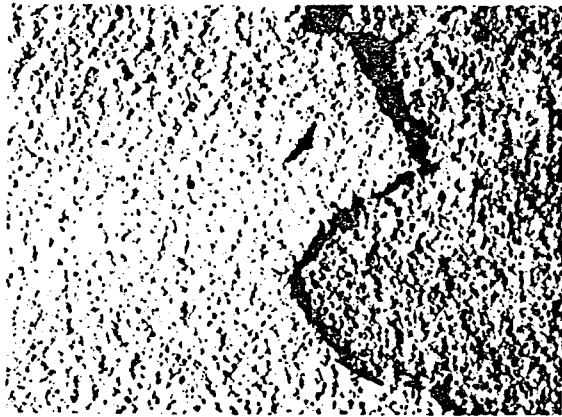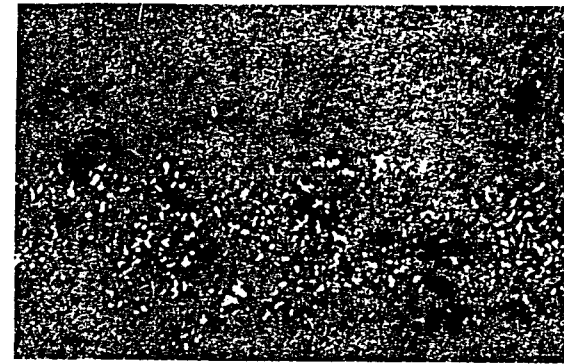Showing surface topography   x20

X-section through pipe
showing particle layer   x250

Showing particle detail   x2000
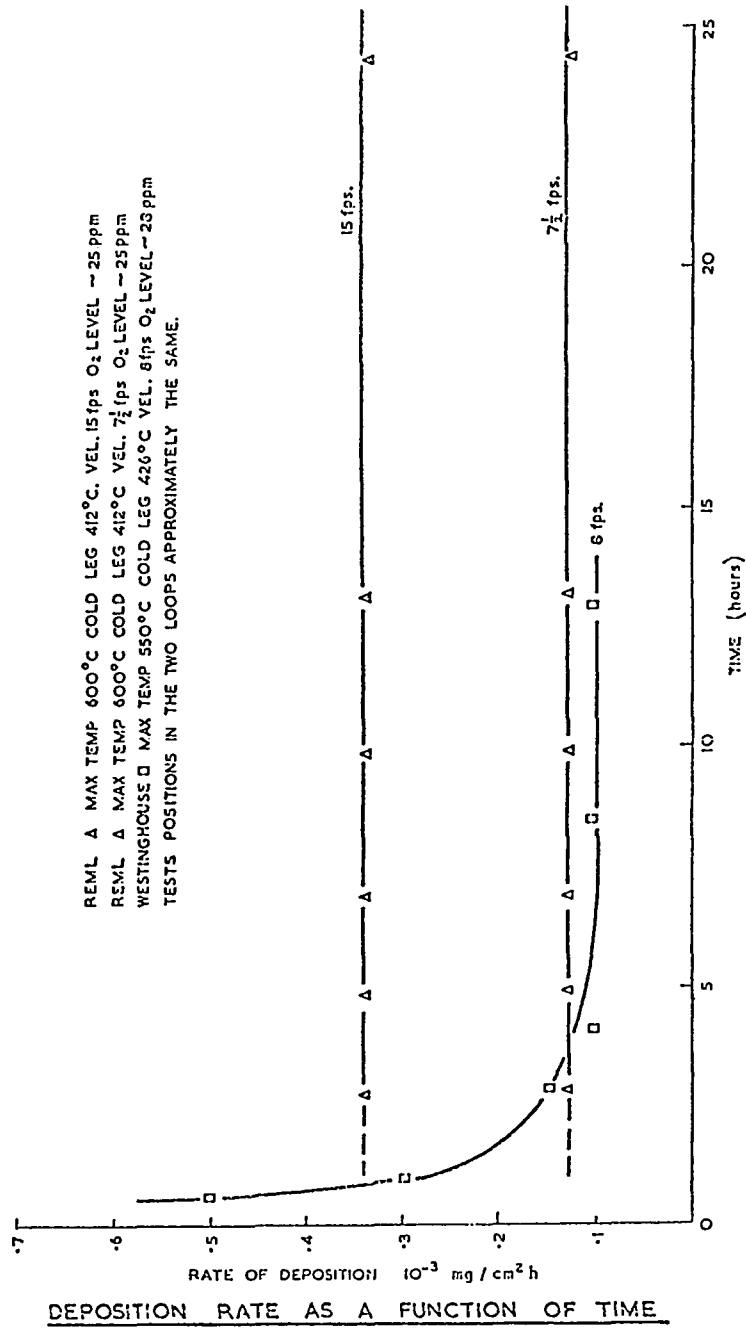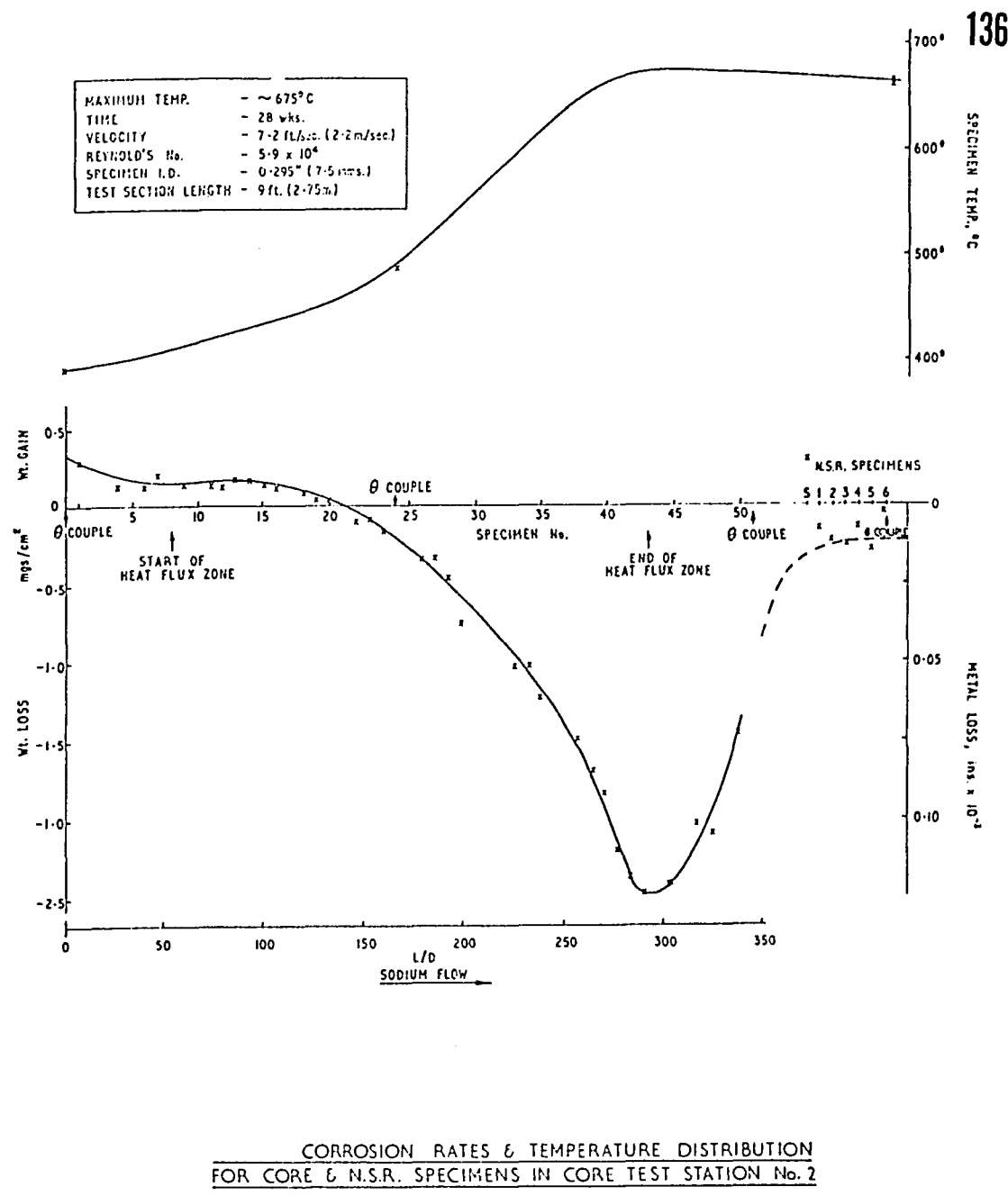

X-section through particle   x1000

FIG. 5.

DEPOSITION STUDIES - 316ss —125 Weeks in holder 7A

Showing surface topography x20

X-section through pipe showing particle layer x250
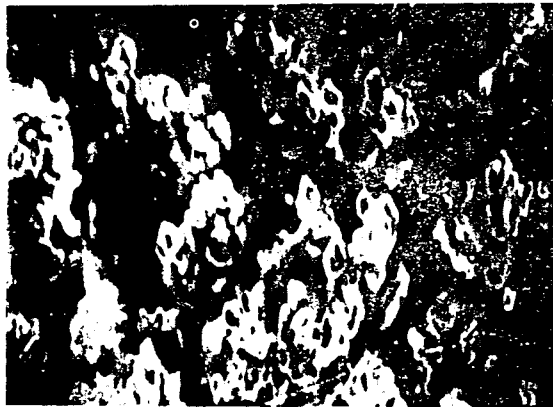
Showing particle detail x2000
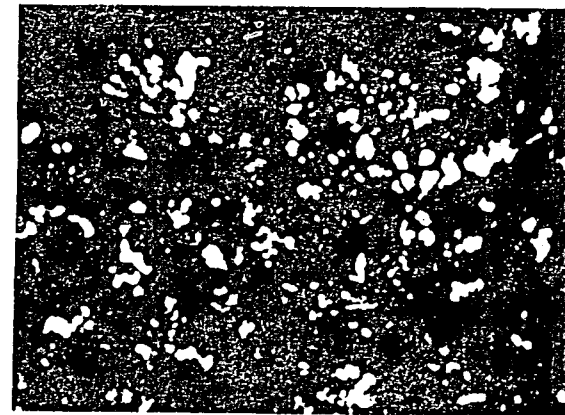
X-section through particles x1000

FIG. 6.

REML △ MAX TEMP 600°C COLD LEG 412°C, VEL. 15 fps O₂ LEVEL ~ 25 ppm
REML △ MAX TEMP 600°C COLD LEG 412°C VEL. 7½ fps O₂ LEVEL ~ 25 ppm
WESTINGHOUSE □ MAX TEMP 550°C COLD LEG 426°C VEL. 6 fps O₂ LEVEL ~ 23 ppm
TESTS POSITIONS IN THE TWO LOOPS APPROXIMATELY THE SAME.

15 fps.

7½ fps.

6 fps.

TIME (hours)

RATE OF DEPOSITION   10⁻³ mg / cm² h

DEPOSITION   RATE   AS   A   FUNCTION   OF   TIME

FIG. 7.

MAXIMUM TEMP.	— ~675°C
TIME	— 28 wks.
VELOCITY	— 7·2 ft/sec. (2·2 m/sec)
REYNOLD'S No.	— 5·9 × 10⁴
SPECIMEN I.D.	— 0·295" (7·5 mms.)
TEST SECTION LENGTH	— 9 ft. (2·75 m)

SPECIMEN TEMP., °C

Wt. GAIN

mps/cm²

Wt. LOSS

METAL LOSS, ins. × 10⁻³

θ COUPLE

START OF
HEAT FLUX ZONE

θ COUPLE

SPECIMEN No.

END OF
HEAT FLUX ZONE

θ COUPLE

N.S.R. SPECIMENS
S 1 2 3 4 5 6

θ COUPLE

L/D
SODIUM FLOW

CORROSION   RATES   &   TEMPERATURE   DISTRIBUTION
FOR CORE & N.S.R. SPECIMENS IN CORE TEST STATION No. 2
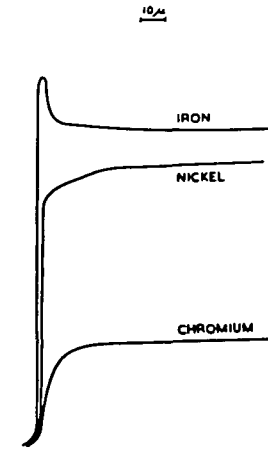
FIG. 8.

WEIGHT GAIN AND TEMPERATURE PROFILES FOR I.H.X. SPECIMENS

TIME	— 28 wks.
VELOCITY	— 3·3 ft./sec (1m/sec.)
REYNOLD'S No.	— 6·2 x 10⁴
SPECIMEN I.D.	— 0·670" (17mms)
TEST SECTION LENGTH	— 14 ft. (4·27ms.)
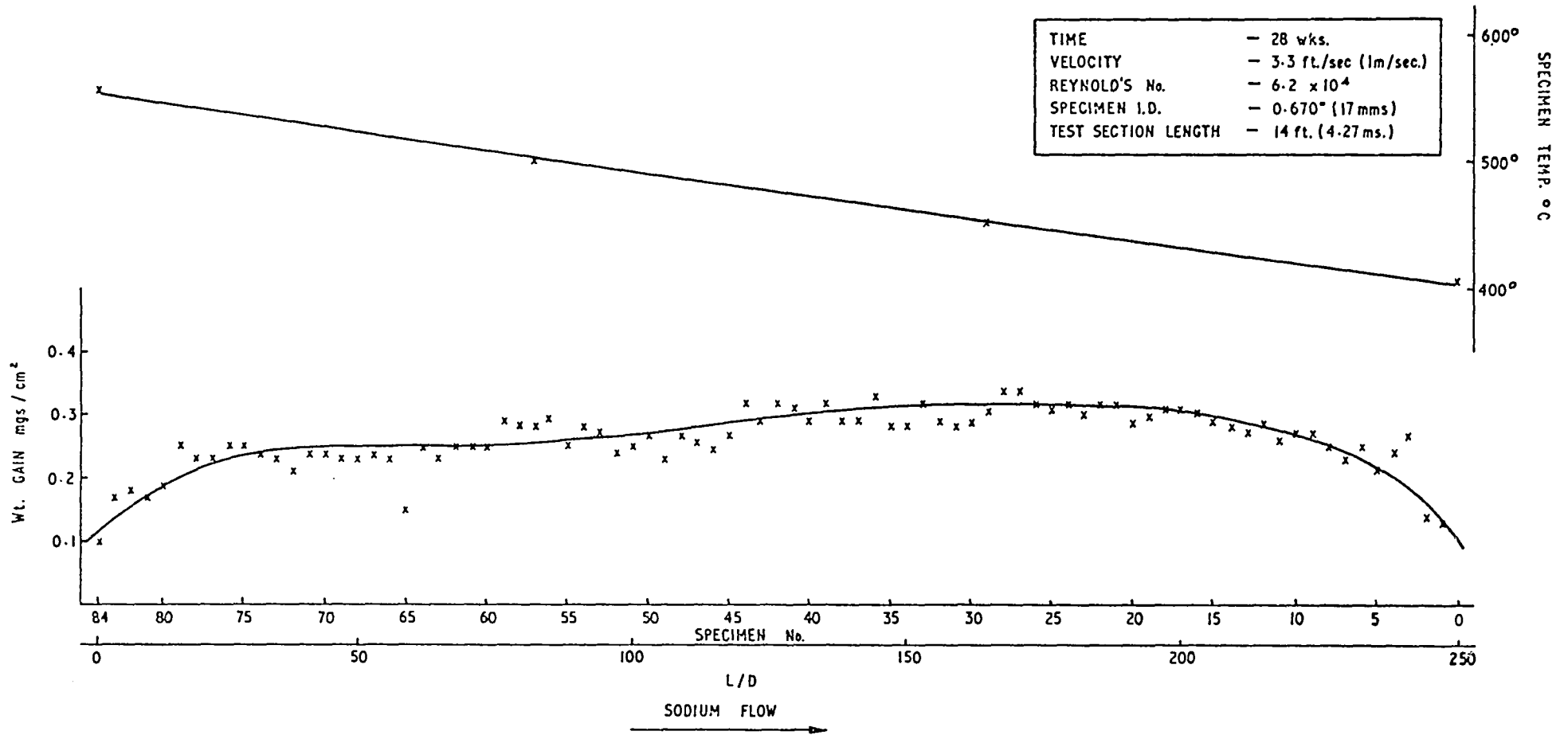
FIG. 9.
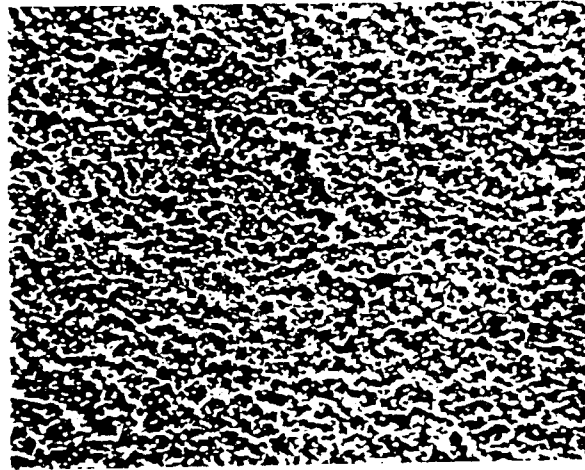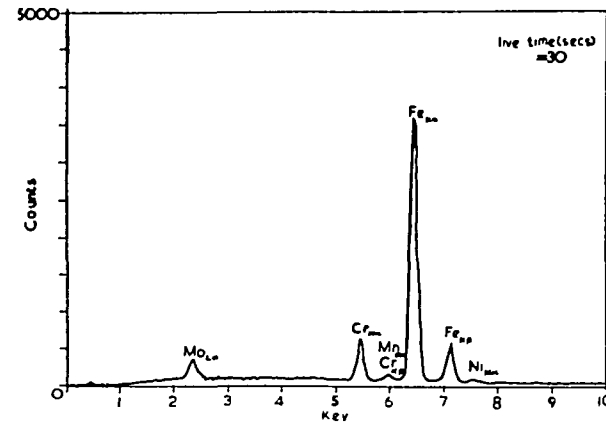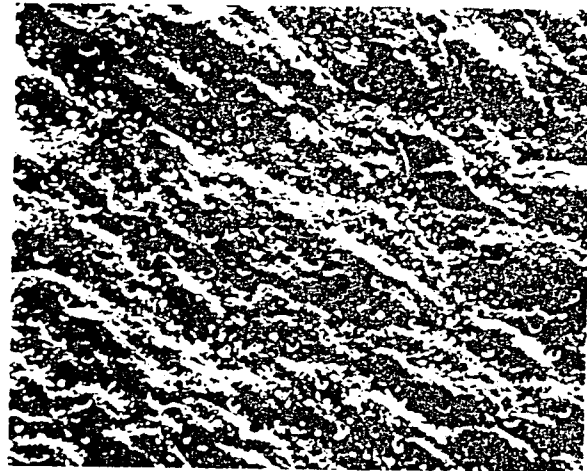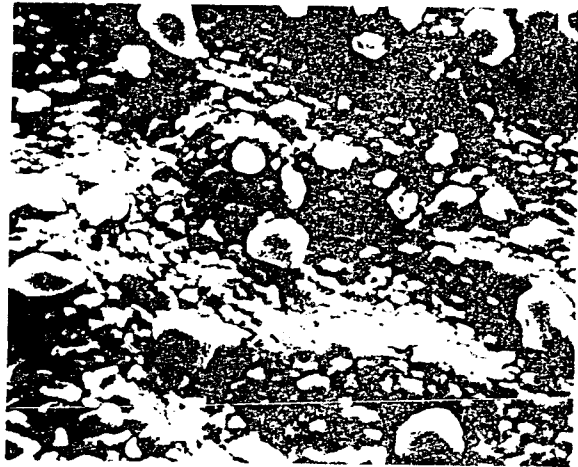
Structure and element concentration in core test section
No 2. Max temp position.

FIG. 10.

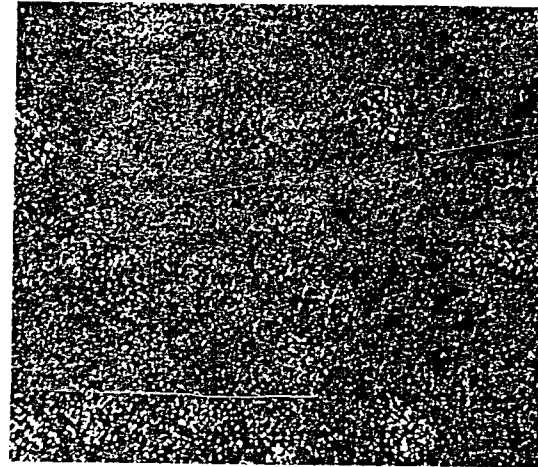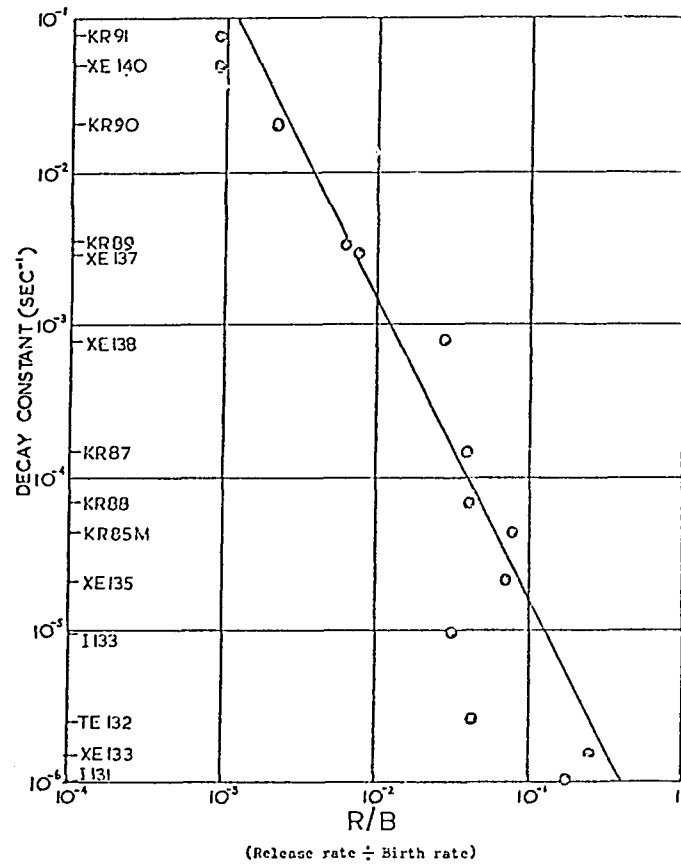×1000

×5000

dash____area adjacent to particle

line _____ particle
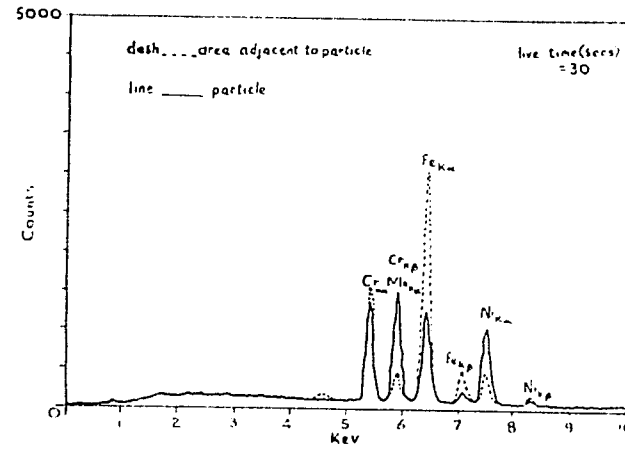
live time(secs)
=30

Mn. X-ray image

Surface structure and general distribution of particles in
THX test station.

FIG. 11.

Half-life Dependence of Short Lived Fission Product Emission from Oxide Fuel during Irradiation.

FIG. 12.

РАДИОАКТИВНОСТЬ ЗАЩИТНОГО ГАЗА БЫСТРОГО РЕАКТОРА (НА ОПЫТЕ БОР-60)

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АННОТАЦИЯ

Рассмотрены результаты исследований активности газовых продуктов деления (ГПД) в первом контуре реактора БОР-60. Полученные данные позволили оценить активность ГПД в газовой системе промышленной АЭС с быстрым реактором, необходимость и пути реализации системы очистки газа.

На реакторе БОР-60 был проведен комплекс исследований радиоактивности защитного газа в различных режимах работы реактора. С 1969 г. до февраля 1972 г. реактор работал с герметичными твэлами. В дальнейшем количество дефектных твэлов достигало 1% от общей загрузки.

I. РАБОТА С ГЕРМЕТИЧНЫМИ ТВЭЛАМИ

Радиоактивность защитного газа при работе с герметичными твэлами определяется активацией защитного газа (^{41}Ar) и теплоносителя (^{23}Ne), а перенос газа в I контуре может быть описан простыми уравнениями [I].

Для всех радиоактивных благородных газов с $T_{1/2} > 10$мин среднее время пребывания в теплоносителе одинаково и составляет ~ 8 часов. При этом доля выходящих из теплоносителя короткоживущих благородных газов мала (для $T_{1/2} < 10$мин не более 3%).