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End Point Determination for Spent Nuclear Fuel Drying Operations

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Abstract

When spent nuclear fuel is being dried ahead of either interim storage or long term disposal it is necessary to be able to confirm that the required level of dryness has been achieved. This has typically involved a vacuum rebound test however this method has certain limitations in terms of both reliability of the result and also by introducing an additional step that is time consuming and depending on the drying system used requires additional equipment at high system cost. It would be preferable if the end point and requisite dryness could be confirmed from online data readings recorded during the drying process.

This paper presents the results of a number of vacuum drying tests using a benchtop drying rig in which online dew point, temperature, pressure and mass flow rate readings were compared to the results of vacuum rebound tests.

Mass flow rate, pressure and dew point readings all showed cliff edge behaviour as the drying process progressed. Flow rate provided a good indicator of progress however it was clear from the behaviour that the resolution of the instrument was not sufficient to confirm dryness. Neither pressure nor dew point readings alone were capable of indicating whether a test would be passed successfully however it was found that in combination an envelope existed in which a vacuum rebound test was always passed.

Testing of the same techniques for flowed gas drying methods was limited due to the lack of a suitable way of confirming whether dryness was achieved however there was an indication that dew point measurements would be capable of confirming that a set level of dryness had been achieved.

Keywords: `nuclear fuel, dry storage, stainless steel, AGR, pinholes, cracks`

1. Introduction

The UK has traditionally reprocessed its spent nuclear fuel however the decision has been made that this will cease in favour of direct disposal to a geological disposal facility (GDF). The majority of spent fuel in the UK comes from its Advanced Gas-cooled Reactors (AGR) which is clad in stainless steel (SS) unlike the zirconium alloys used for cladding fuel in the majority of reactors worldwide.

Pond storage of spent AGR fuel in demineralised water initially led to failures due to stress corrosion cracking however experiment and subsequent experience (since the 1980's) has shown that AGR fuel can be safely stored in water dosed to pH 11.4 with sodium hydroxide for periods of up to 25 years[1], however with a GDF not being expected to be available for disposal of fuel until 2075[2] it is necessary to ensure that a viable alternative to extended pond storage is available. Consequently there is a growing interest in the UK in interim dry storage as a contingency option and work is required to establish whether dry storage is viable for previously pond stored AGR fuel.

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Abbreviations

AGR	Advanced Gas-cooled Reactor
CH	Circulation heater
CSS	Cracked stainless steel
DPM	Dew point meter
GDF	Geological disposal facility
HX	Heat exchanger
S1	Sieve 1
S2	Sieve 2
SS	Stainless steel
SSP	Stainless steel pin-holed
ST	Surface temperature
TC	Thermocouple
TP	Test piece
VH	Vessel heater
Vout	Vessel outlet
VT	Vessel temperature

Dryness for dry storage systems is typically confirmed through the use of a vacuum rebound test which is recommended in both ASTM C-1553-Standard Guide for Drying Behaviour of Spent Nuclear Fuel[3] and NUREG 1536-Standard Review Plan for Dry Cask Storage Systems [4]. A vacuum rebound test involves evacuating the drying vessel to below 4 mBarA before isolating the vessel and holding it there for at least 30 minutes. This is based on calculations by Knoll and Gilbert[5] which indicated that the at this level the quantity of oxidising gas that would remain in a storage cask after drying would be insufficient to lead to significant failure of Zircaloy clad fuel in the expected 40 year lifetime of a dry storage canister.

Flowed gas methods such as Holtec's Forced Helium Dehydration[6] were developed such that dryness could be determined by a combination of vapour pressure and measured number of canister air changes. However, since the vacuum rebound test is recommended in documents such as ASTM C-1553 and NUREG 1536 it has become the standard and recognised technique and is also used to confirm dryness when carrying out flowed gas drying.

While a properly implemented vacuum drying test is a reasonable method of confirming dryness it is possible to effectively cheat the test since the requirement is based on staying below a certain pressure for a given time limit rather than specifying a maximum rate of pressure rise. For instance by evacuating the canister to a final pressure of <1 mBarA, then even a pressure rise of 6 mBarA hr^{-1} (1.7 x 10^{-3} mBar s^{-1}) would lead to a passed test. A pressure rise such as this would almost certainly only be possible by the vaporisation of a significant quantity of water and would consequently lead to the limits on oxidising gas concentration laid calculated by Knoll and Gilbert[5], upon which the vacuum rebound test criteria is based, being broken. Note that the Cold Vacuum Drying Method used to dry uranium metal fuel at Hanford specified that a vacuum rebound test should be commenced once the canister pressure had dropped below 0.7 mBarA[7].

A further disadvantage of the use of a vacuum rebound test to confirm dryness is that the drying process itself must be interrupted while a rebound test is held. This is an even greater inconvenience for flowed gas drying when the delay is greater than 30 minutes, as the vessel itself must be evacuated and additional equipments is required which adds to the capital cost. For these reasons it would be preferable to be able to confirm that dryness has been achieved through the use of online readings during the drying process which may remove the need for the time consuming extra step which could potentially require a repeated drying cycle if the test is failed with further time costs.

In scientific literature there is relatively little work related to the drying of spent fuel and of that which does exist virtually none touches upon attempts at detecting and confirming end point. One exception is the work of Rodrigo et al [8] who unsuccessfully attempted to correlate vacuum rebound rate to dryness, however this work used fuel containing significant quantities of corrosion product which are not a concern for AGR fuel or LWR fuel. It would however seem likely that some research in this area has been carried out commercially but not published or remains proprietary.

This paper presents the results of a number of

tests which investigate whether online data measurements such as temperature, pressure, dew point and mass flow rate could be used to confirm end point and dryness and remove the need for vacuum rebound tests. The testing uses a benchtop scale drying rig which has been used in earlier research into the drying behaviour of spent nuclear fuel[9, 10]. This work focusses on vacuum drying although reference is made to several tests using using the flowed gas drying method and is concerned with the drying behaviour through physical cladding defects such as pinholes and cracks and does not consider other cladding properties or the influence of factors such as material, irradiation and service temperature.

2. Methodology

2.1. Drying Rig

The drying rig (fig. 1) was designed as a multi-purpose system capable of both vacuum and flowed gas drying and a simplified schematic is shown in fig. 2. Full details of the rig can and subsequent testing can be found in Goode et al[9]. The rig consisted of a SS drying vessel with a volume of ∼500 ml. The lid of the vessel had two main process lines one of which was fitted to a dip tube which ran to the bottom of the vessel and acted as the inlet line and the second of which entered at the top of the vessel and was the outlet. A thermocouple and pressure transducer were fitted to the vessel lid (vessel temperature-VT) and a second flexible thermocouple (TC) was fed into the vessel itself and was able to measure the temperature of objects inside the vessel (surface temperature-ST). Band heaters with integral TC's were fitted to the vessel walls (vessel heater-VH). A smaller bore line was fitted to the vessel with a valve and blanking plug.

From the outlet line a hose led to a dew point meter (DPM) and further thermocouple (Vout), then to a mass flow meter, a water cooled heat exchanger with a thermocouple at the inlet (HX-heat exchanger) to condense out bulk water and finally a 1.5" inside diameter SS tube packed with molecular sieve as a final drying step. Two thermocouples were fitted to the surface of the tube, approximately one third (sieve 1-S1) and two thirds (sieve 2-S2) from the start of the tube. From here a small line was fed to an Edwards E2M5 vacuum pump while a larger line was fed back to the vessel via a TCS Micropumps D10K gas pump and a CastX-1000 circulation heater (CH) for flowed drying operations. All temperature, pressure, dew point and mass flow measurements were logged digitally at a rate of at least 1 Hz.

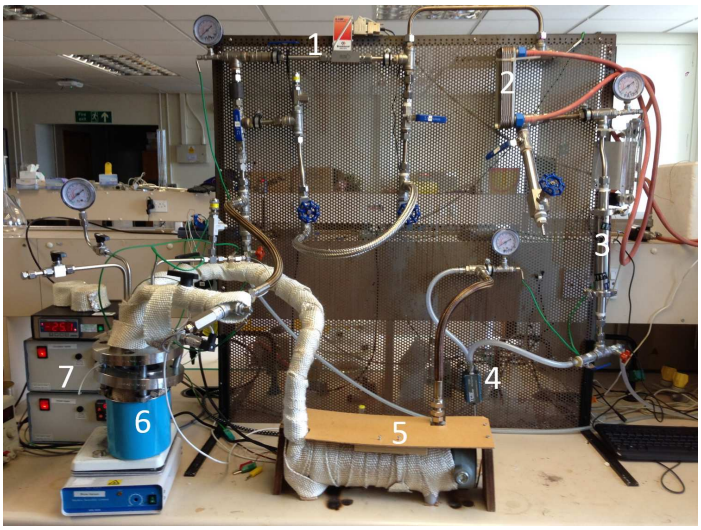


Figure 1: The drying rig used in these tests. 1-Flowmeter, 2-condenser, 3-molecular sieve, 4-circulation pump, 5-circulation heater, 6- drying vessel and 7-heater controller.

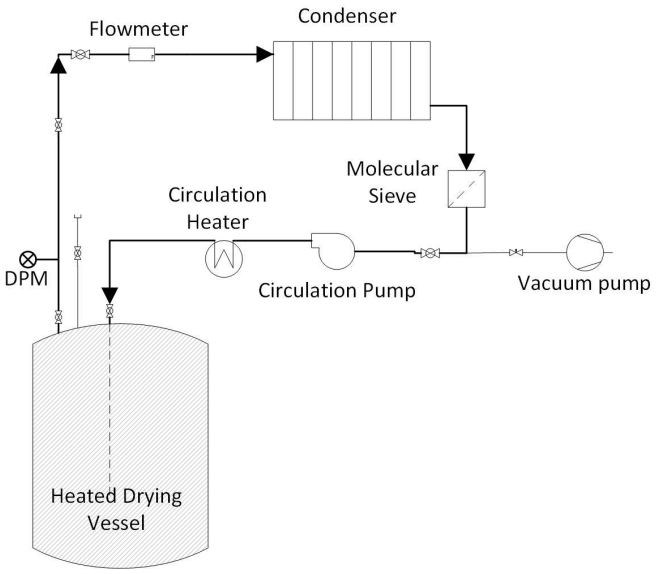


Figure 2: Simplified schematic of the drying rig.

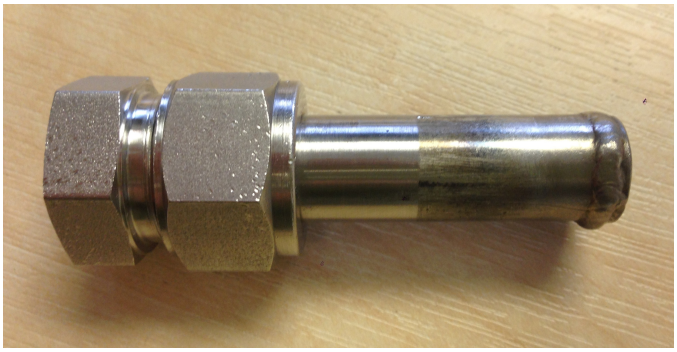
2.2. Test Pieces

Drying tests were carried out with two stainless steel test pieces (TP) as seen in fig. 3. The first TP was constructed from a small length of AGR cladding with a SS plug welded into one end and a short length of SS tube welded into the other. A 300 μm hole was drilled into the cladding section to replicate a pinhole and this TP is known as SSP TP (Stainless steel pinholed TP, fig. 3a). The second TP was produced from a length of 304 SS which had been sensitised and then compressed lengthwise leading to stress at the inner surface at the 12 and 6 o'clock position and at the outer surface at the 3 and 9 o'clock position. It was then held in boiling 42% magnesium chloride solution for 28 days leading to stress corrosion cracks running axially in the stressed regions. This is known as CSS TP (Cracked stainless steel TP, fig. 3b). The volume inside each TP was ∼7 ml. During the weeks of testing the TP was held in a glassware drying oven at 50°C with the lid removed when not in use and reweighed before use so an accurate dry mass was available.

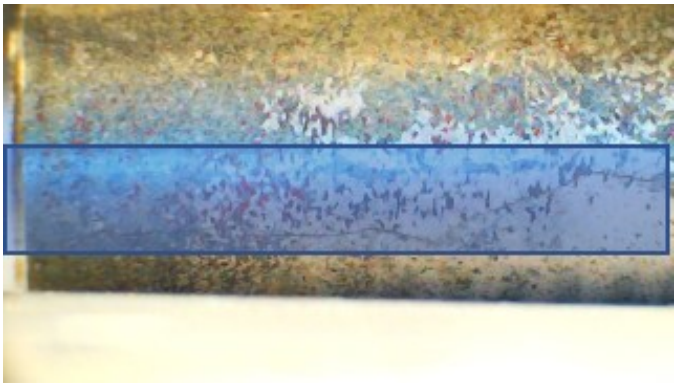
Ahead of each test a small quantity (∼1 ml) of water was placed into the TP and the TP was shaken to wet all of the the surfaces. The remaining water was then tipped out and the lid replaced. The mass of the TP was then recorded to 0.1 mg. The TP was placed inside the drying vessel with a thermocouple attached to its surface. A small number of tests were carried out using both the SSP and CSS TP's.

Vacuum drying tests were carried out at 60°C with the vessel preheated before the test. At the beginning of each test the data loggers were started followed by the vacuum pump and valves were opened where necessary. Each test would be ended at different points depending on live data readings. A test was ended by isolating the vessel (closing the vessel isolation valves), shutting down the pump. When vacuum drying individual test pieces the data was then logged for at least 30 minutes further so a pressure rise could be detected and online readings compared to the results of a pressure rebound test.

(a) Pinholed stainless steel test piece (SSP TP) with pin hole highlighted.



(b) The cracked stainless steel test piece (CSS TP).



(c) The tube used to produce the CSS TP showing one of the four cracks.

Figure 3: The test pieces used in the drying tests [10].

2.3. Test Criteria

A test was deemed a pass (all water had been removed) if the rebound rate was sufficiently low. The value of this rate was determined from leak testing of the vessel to ascertain the system leak rate, and this is presented below. The final mass of the TP was also recorded for comparison to the dry mass of the test piece and when the start pressure was low enough the results of a standard vacuum rebound were recorded as a pass or fail.

3. Results

3.1. Vacuum Drying

3.1.1. System Vacuum & Leak Rate

Before carrying out full drying tests the system was evacuated without a TP present to establish the system ultimate vacuum and ensure that a sufficiently low pressure could be obtained reliably. Once the vacuum had stabilised the vessel was isolated from the vacuum pump and held for at least 30 minutes to establish what the systems background leak rate was. An example of one of these tests is shown in fig. 4. After multiple tests it was found that a pressure in the range 2.4-3.6 mBarA could be obtained reliably. This was low enough to allow a vacuum rebound test to be performed as dictated by ASTM C-1553. The leak rate in the system was also found to be consistent being 1.8-6.3 x 10^{-4} mBar s^{-1}. Based on this a rebound rate of less than 1 x 10^{-3} mBar s^{-1} was, albeit somewhat arbitrarily, designated as being a pass. This value is a little more than half the value that is effectively allowable with a standard vacuum rebound test.

3.1.2. Initial Data Plot-SSP TP

Figure 5 shows a data plot produced for the first end point test using the SSP TP. This test was extended to ensure that this test was a pass and that any visible changes in the data were observed although the behaviour was typical of all tests. Figure 5a shows the pressure, dew point and flow rate. The pressure initially drops sharply to ~24 mBarA and then ~22 mBarA when the vessel is evacuated and the flow rate shows similar behaviour. The dew point drops sharply and

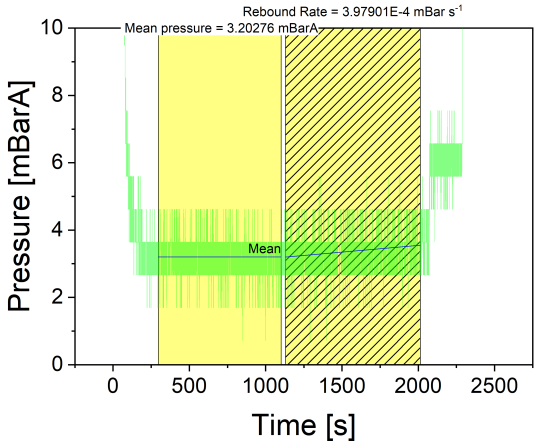


Figure 4: Plot showing the behaviour of an empty vessel during a rebound test.

rises before stabilising around 15°C with fluctuations matching the steps in flow and pressure. These three values all show clear cliff edge behaviour after around 1000 s. The pressure drops rapidly to the ultimate vacuum achievable, while the flow rate reduces to zero. The dew point drops rapidly and continues dropping until the vessel is isolated.

Figure 5b shows the thermocouple readings in which some none linear behaviour is observed along with the pressure plot. All of the thermocouple readings show an initial drop due to adiabatic cooling as the vessel is evacuated. Vout and HX then rise sharply as warm air from the vessel is drawn past. HX slowly warms during the test levelling out at the cooling water temperature after around 2000s, before starting to rise again around 4000 s. After the sharp rise Vout shows a steady downward trend levelling out around 4000 s however a small but noticeable drop in Vout takes place as the cliff edge occurs. VT increases in three steps with the steps coinciding with the fluctuations in pressure, mass flow and dew point. S1 rises steadily due to the exothermic adsorption of water by the molecular sieve. After the cliff edge behaviour S1 begins to drop until it levels out around 4000 s. The change in behaviour for Vout, HX and S1 at ~4000 s is due to the cooling water to HX being shut off rather than an event within the system and causes temperatures to level out at close to ambient.

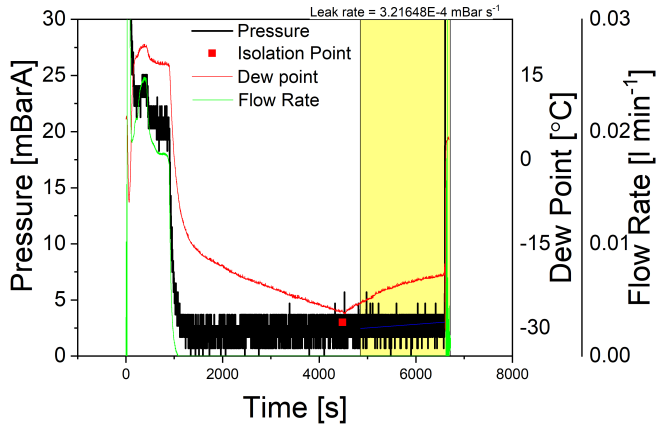
The point at which the vessel was isolated was noted. The the rebound rate was found to be within the acceptable range for the test to be a pass and the pressure also stayed below 4 mBarA for in excess of 30 minutes therefore the requirements for a vacuum rebound test in ASTM C-1553 were achieved. The final mass of the TP indicated that the TP had returned to close to its dry mass.

The important points from these plots are the cliff edge behaviour exhibited by the pressure, flow and dew point which are likely to be indicating the point at which the final quantities of liquid water are removed, particularly since the exothermic warming of the molecular sieve ceased at the same time. These parameters may therefore provide a suitable means to detect the experiment end point i.e. that the fuel is dry. Furthermore there is some evidence that some thermocouple readings showed changes at the same time and may therefore provide some information. It should be noted that the shape of the mass flow curve is not that of an exponential decay curve as would be expected since the flow meter reached its limit of detection (which was approximately 2.91 x 10^{-3} l min^{-1}).

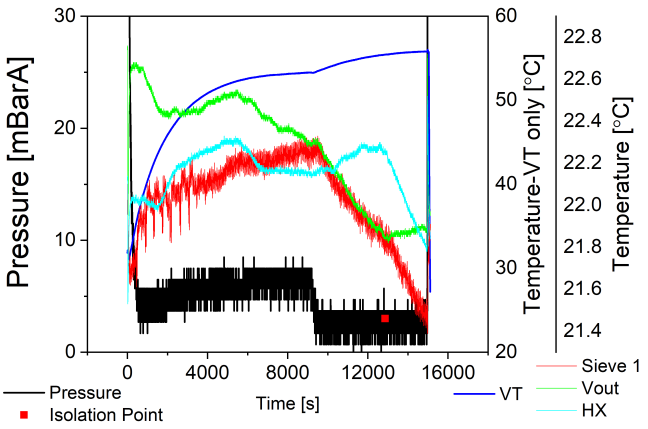
Based on this, future tests were carried out in which the vessel was isolated as close to the start of the cliff edge, but after the flow had reached zero (since mass flow is a clear indication of water being present), as possible.

3.1.3. Initial Data Plot-CSS TP

Figure 6 shows a data plot for the first test vacuum drying end point test using the CSS TP. The overall behaviour of the cracked TP is similar to the pinholed TP in that there is an initial drop in pressure and flow and a rise in dew point followed by a broadly steady period before all values drop rapidly. There are however significant differences. Firstly the pressure initially drops to ~4 mBarA before rising slowly with the steady period being around 6 mBarA. The flow behaviour is also similar, dropping briefly to zero before rising again. The most likely reason is that the pressure and flow initially drop as the vessel is evacuated. Due to the size and shape of the defect in the TP there is a delay before water is able to vaporise and pass

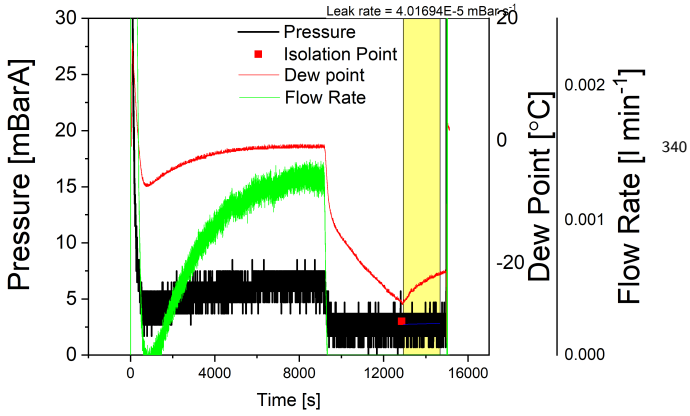


(a) Pressure, dew point and flow rate.

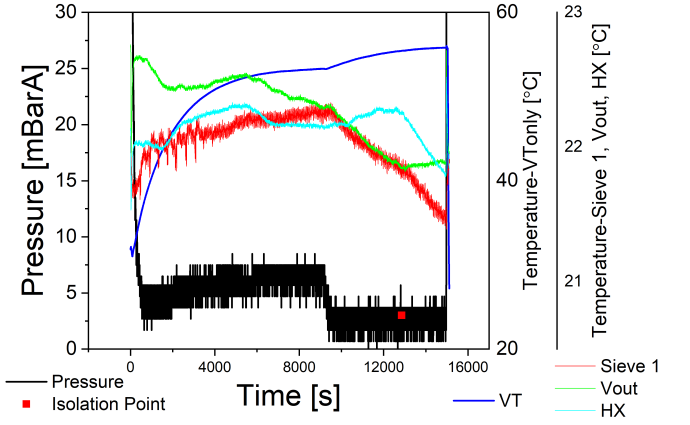


(b) Temperature (and pressure).

Figure 5: Data plot of the initial extended vacuum drying end point test using the pinholed TP.



(a) Pressure, dew point and flow rate.



(b) Temperature (and pressure).

Figure 6: Data plot of a vacuum drying end point test using the cracked TP.

through the crack in sufficient quantity to impact upon the pressure and flow rate. Since the rate at which water is able to pass across the defect is reduced the steady period is significantly longer than with the pinholed TP with the cliff edge behaviour now occurring after ∼9000 s rather than ∼1000 s.

3.1.4. Multiple Test Pieces

A small number of tests were carried out using both the cracked and pinholed test pieces simultaneously. An example of one of these tests is shown in fig. 7. The observed behaviour is a combination of the two separate test pieces. After around 1700 s there is a cliff edge drop in pressure, flow and dew point. Importantly the flow rate does not drop to zero. The pressure drops to a little over 4 mBarA but the dew point stays above -10°C. All three parameters rise again before a further cliff edge after ∼8000 s. On this occasion the flow rate drops to zero, the dew point drops rapidly to well below -10°C and the pressure drops below 4 mBarA indicating that both test pieces are dry.

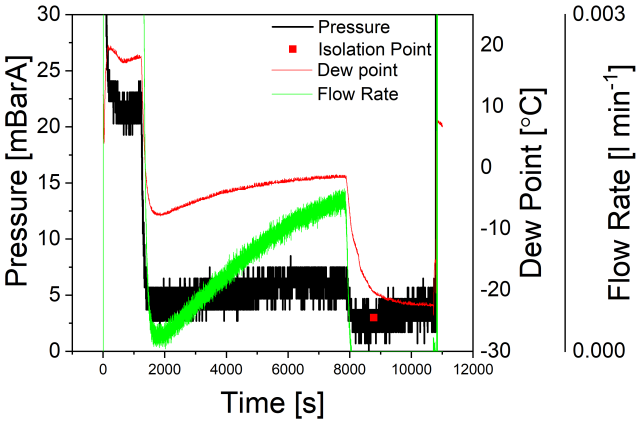


Figure 7: Data plot showing drying behaviour when drying the cracked and pinholed test pieces. Two distinct cliff edges are apparent.

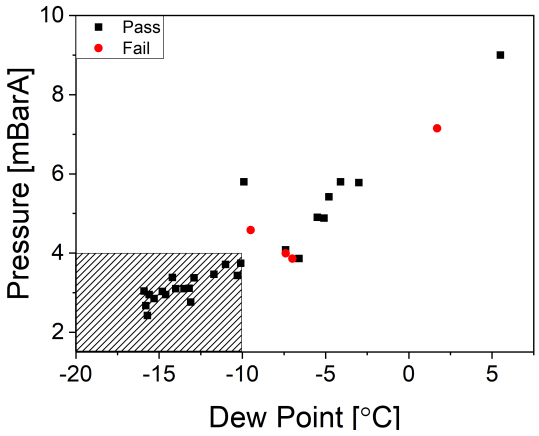
3.2. Overall Results

In fig. 8a the dew point at the time of isolation is plotted against the pressure at the time of isolation for in excess of 30 different tests using the pinholed TP. Passed tests are shown in black and failed tests in red. All tests in which a pressure of ≤4 mBarA and a dew point of ≤-10°C was achieved was a pass.

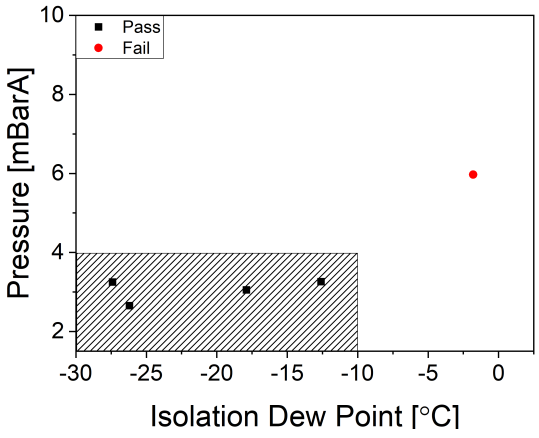
While far fewer tests were conducted using the cracked test piece any test within this region was also a pass (fig. 8b).

Figure 9 shows the mass of water remaining in the TP at the end of each of the SSP TP vacuum drying test. It was noted early on that the dry mass varied by ∼2 mg with on occasions negative masses being observed, making the use of mass alone a poor measure of final dryness. Nevertheless it can be seen that the mass recorded for the failed tests was significantly higher than for the passed tests.

The vacuum test rebound rate is shown in fig. 10. While two of the failed tests had rebound



(a) Pinholed TP resuts.



(b) Cracked TP results.

Figure 8: Plots showing the results of multiple drying tests.

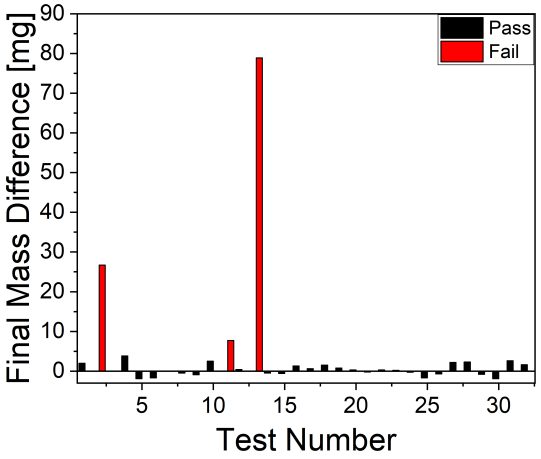


Figure 9: The mass of water remaining after each of the vacuum drying tests with the pinholed test piece.

rates well in excess of 1.7 x 10^{-3} mBar s^{-1}, which can be thought of as almost the limiting rate in standard vacuum rebound tests, one failed test had rebound rate below this level despite. Note that test number 30 was isolated using an alternative, downstream valve and the higher leak rate obtained compared to other failed tests is almost certainly due to a higher system leak rate.

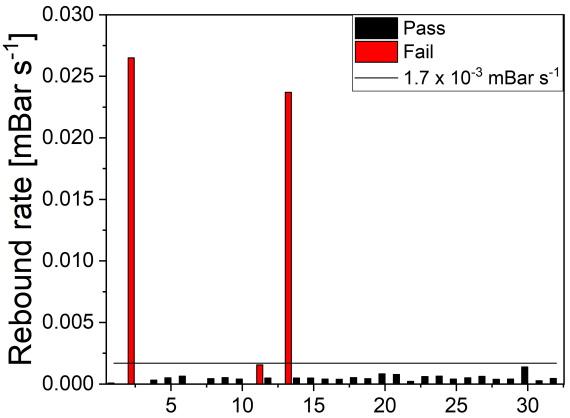


Figure 10: The rebound rate observed with the pinholed test piece.

3.3. Flowed Gas Drying Tests

For flowed gas drying a vacuum rebound test could not be used to confirm the instrument readings since the action of evacuating the vessel would

remove any water that had remained. The initial intention was to record dryness with mass measurements however when these were found to fluctuate this was felt to be unreliable. Nevertheless a number of flowed gas drying tests were carried out an example of which is seen in fig. 11.

While the system pressure and mass flow rate would technically alter as water in the system vaporised this change would be too small to be detected. The same is not however true for dew point. As with the vacuum drying tests the dew point shows a clear cliff edge behaviour. The only logical explanation for this is the complete removal of unbound water from the system.

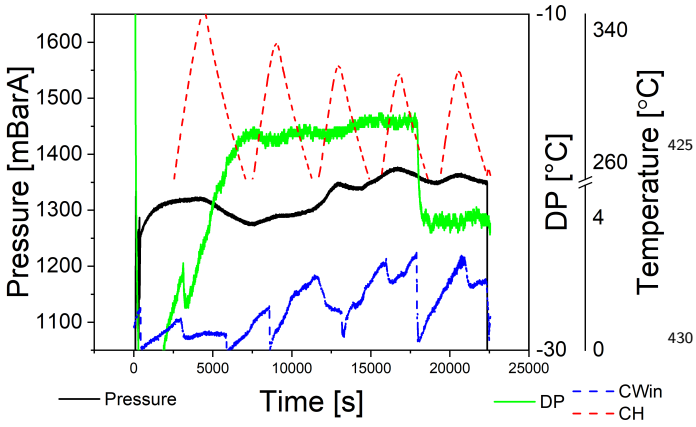


Figure 11: Example of a data plot when flowed gas drying the pinholed test piece.

4. Discussion

It is clear from the data that the majority of tests using a single test piece resulted in a pass. This is thought to be in part due to the fact that stopping tests just as the flow rate hit zero was exceptionally difficult. It does however indicate that cliff edge behaviour is indicative of the last drops of water being removed. The most interesting observation however is the presence of an envelope within which a test was always a pass. Neither dew point nor pressure alone were able to confirm dryness however in combination they could be used to guarantee dryness when vacuum drying. One of the concerns when vacuum drying is ice formation due to adiabatic cooling. If ice were to form it is possible for cliff edge behaviour to be observed (and theoretically a vacuum rebound test to be passed) however in all vacuum drying tests the surface temperature of the test piece was recorded and was found to remain well above the freezing point of water in the conditions observed. Since the value of 4 mBarA is below the triple point of water it is certain that only water vapour can possibly have been present after the cliff edge behaviour and that would have been at low levels.

Mass flow rate was highly sensitive however to be of use on this scale it would have to be able to detect the background leak rate of the system which is too low on a system of this size with the equipment available. It would seem likely that on the commercial scale mass flow rate could play a role by diverting the flow through narrow bore flow meters once a sufficiently low pressure has been reached, effectively amplifying any mass and increasing sensitivity.

Temperature data has been shown previously to be sensitive to changes in system behaviour[9] and this is shown once again. The behaviour of several thermocouple were shown to react to the end point. While the behaviour was not as clear as dew point, pressure and flow rate, it would seem likely that temperature measurements could be used informatively if applied in a well thought out manner.

Mass measurements were found to be somewhat disappointing due to the variation observed however there is a thought that this may have been related to thermal expansion. They did however provide some circumstantial corroborating evidence that dryness was being achieved.

Discounting test number 30, which is in this regard considered to be incomparable, the highest rebound rate observed, in a passed test was found to be ~8 x 10^{-4} mBar s^{-1}, less than half the "allowable" rate in a standard vacuum rebound test, with the average rate being ~4 x 10^{-4} mBar s^{-1}. In comparison the minimum rate observed in a failed test was ~1.6 x 10^{-4} mBar s^{-1} which is within the range in which a commercial vacuum rebound test could be passed. This indicates that a time based test as is standard has is flawed as

it allows in some conditions excessive water to be carried over.

When both the cracked and pinholed TP's were dried together two separate cliff edges were observed. Importantly the values recorded after the first cliff edge indicated that water remained within the system and that drying needed to be continued. After the second cliff edge the values indicated that the both TP's were dry. This is important since it means the readings were sensitive enough to discern between the two pieces.

Testing of online measurements when flowed gas drying was limited in part because no reliable method of recording dryness was available after mass readings were found to be inconsistent. Vacuum rebound tests were not practical since the action of evacuating the vessel would remove any remaining water thus invalidating the result. Nevertheless the clear cliff edge behaviour observed suggests that such measurements could be used to confirm dryness.

5. Conclusion

Based on the experiments carried out it would seem likely that online measurements can be used to determine the end point when vacuum drying spent nuclear fuel without the need for vacuum rebound tests. For the system presented here when a dew point of $\leq$-10°C and a pressure of $\leq$4 mBarA were achieved all free and trapped water had been removed. Mass flow and temperature readings could also potentially be of use. Values would however have to be developed for a final system. There is a strong suggestion that dew point could be used to confirm that a flowed gas drying process was complete however further work would be required to confirm this. Finally, it was found that a rebound rate which could theoretically be achieved during a passed commercial vacuum rebound test failed using the test conditions stipulated here.

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