Waste container durability: monitoring of a stainless steel 4 metre box over 12 years

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ABSTRACT

In the UK, most intermediate level radioactive waste is packaged in stainless steel containers. It is important that containers should maintain their integrity for extended periods of storage prior to final disposal. A prototype 4 metre box fabricated from 304S11 stainless steel was monitored at an inland rural building for over twelve years to gain experience in monitoring environmental conditions and corrosion during storage. A wide range of different types of aerosol particles were observed on the surfaces and in the air. They contained corrosive ions such as chloride, as well as other anions such as sulfate and nitrate. The chloride deposition rate was between 0.1 and 4 µg cm^{-2} yr^{-1} depending on the orientation and location of the surface, with a chloride/sulfate ratio ranging from ~0.3−2. The temperature and relative humidity typically ranged from 0−30ºC and 40−90%, respectively. The greatest degree of corrosion developed on the base of the box, which had elevated chloride concentrations (10−140 µg cm^{-2}). However, there was little evidence of corrosion propagation on the surface of the box and no evidence of stress corrosion cracking. The average pit propagation rates on a 316L coupon mounted next to the 4 m box were <1 µm yr^{-1}.

KEYWORDS: radioactive waste container, storage, atmospheric, corrosion, environment, monitoring.

Introduction

In the United Kingdom the Nuclear Decommissioning Authority (NDA) is responsible for ensuring that intermediate level waste (ILW) is packaged by waste producers in a form that is suitable for safe storage, transport, handling and potential disposal in a geological disposal facility (GDF). After retrieval from waste storage facilities and processing, NDA's current plans are that most ILW will be placed in stainless steel containers and encapsulated in a cementitious grout, although other possible encapsulants (e.g. polymers) and container materials (e.g. cast iron) are under consideration. After encapsulation in the stainless steel containers the packages will be transported to above-ground interim storage facilities, where it is likely that they will remain for several decades before eventual transport and placement in a GDF. During the period of interim storage, and subsequently during the operational phase of a GDF, it is important to have confidence that the containers will maintain their integrity so that they can fulfil their key generic waste package safety functions (Nuclear Decommissioning Authority, 2010). One of the key processes that could affect the performance of the waste containers is corrosion, which is primarily governed by the environmental conditions during storage. The aim of the study described in this paper was to monitor the corrosion behaviour of a prototype waste container in a representative environment, whilst characterizing the environmental conditions and developing corrosion monitoring techniques. This paper summarizes the experimental approach that was adopted and presents a selection of the results

obtained. Further detail can be found in (Winsley *et al*., 2011).

Experimental Methods

Materials

The NDA have designed a number of standard radioactive waste packages, ranging in increasing size from the most commonly used container, the 500 litre drum, through the 2 m box, the 3 m^3 drum to the 4 m box, which will be used predominantly for packaging large items that cannot easily be reduced in size. The corrosion monitoring programme described in this paper was carried out using a prototype 4 m box, which has nominal external dimensions of $4 \times 2.4 \times 2.2$ m, with a wall thickness of 3 mm and a base thickness of 6 mm. It has a filtered vent in the lid, with a polymeric lid gasket (EPDM) around the lid flange. The prototype container used for the monitoring programme was fabricated from 304L (equivalent to 304S11 and 1.4306) stainless steel, although 316L stainless steel would be the material of choice for operational waste containers. The 4 m box has a 200 mm thick stainless steel reinforced concrete lining, but there will be some areas of stainless steel exposed to atmospheric conditions inside the box (i.e. the underside of the lid). The surface finish was 2B on panels and 1D on structural members (e.g. corner posts). The welds were pickle paste treated (except the welds on the base of the box) and some trials of glass bead peening and chemical pickling were carried out on selected areas during the initial stages of the project.

Environmental monitoring

For most of the duration of the project (1998−2008), the 4 m box was located at an inland rural location in an environmentally uncontrolled building at Culham Science Centre, Oxfordshire, UK. A short duration road trip was made to Dounreay in early 1999 after which the 4 m box was returned to Culham. During the trip the base of the box accumulated some road salt contamination. In 2008 the 4 m box was transferred to another environmentally uncontrolled store at Harwell, Oxfordshire, where it was monitored up to 2010. Therefore, for the whole of the project, the surfaces of the box were exposed to a range of atmospheric conditions. The following key environmental parameters were measured and recorded using automatic data logging equipment, both inside and outside the box: air temperature and steel surface temperatures; relative humidity; time of wetness (using various designs of sensor). In addition, secondary parameters measured were atmospheric pressure and the rate of air movement in the stores at various locations.

The surface contamination on the surface of the 4 m box was also characterized by (1) using swabs (low chloride filter papers) that had been wetted using demineralized water to wipe the surfaces of interest and collect samples for chemical analysis, and (2) sticky carbon tape to collect samples for scanning electron microscopy (SEM) and energy-dispersive X-ray (EDX) analysis. Anions were analysed using ion chromatography (IC) of the leachate from the swabs; the main anion of interest was chloride, but some swabs were analysed for other anions as well (such as sulfate and nitrate, which are potential corrosion inhibitors). Trials using multiple swabs on the same sampling area (Winsley *et al*., 2011) showed that the chloride present on the second swab was ~10% of the chloride detected on the first swab and so three swabs from each area were normally used to maximize the amount of water-soluble material removed during the sampling procedure. This technique makes the assumption that all soluble chlorides and other ions are absorbed equally readily on the filter papers.

Airborne species were collected by pumping air through a filter (0.2 μm) mounted on top of a flask containing demineralized water. In this way coarser solid particulates were collected on the filter paper whereas finer particulates and gases were collected in the demineralized water. Both the particulates on the filter paper and the demineralized water were analysed using (1) IC to determine the concentration of water soluble species and (2) SEM/EDX analysis to characterize the morphology and elemental composition of solid materials. The aim of the analyses was to characterize airborne particulates and soluble gases, including organic species such as organic acids. Air samples were also analysed for volatile organic carbon species (VOCs). A basic characterization of microbial activity (total viable aerobic count) was obtained by taking swabs from a number of representative surfaces on the 4 m box and other containers in the same store and preparing cultures from which most probable numbers (MPN) could be calculated.

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Corrosion monitoring

Corrosion monitoring used a range of techniques, including visual inspection, photography and microscopy of key areas (particularly the base of the 4 m box and areas of embedded iron), including metallographic analysis of samples extracted from specific areas; atmospheric corrosion probes (based on measuring the electrical resistance of thin 304L stainless steel foils exposed to the atmosphere); half-cell potential measurements on stainless steel reinforcement embedded in the inner concrete liner (the corrosion potential, measured against a copper–copper sulfate reference electrode, which gives an indication of whether the reinforcement was undergoing active corrosion) and dye penetrant testing of welds. In addition a set of 316L, carbon steel and galvanized steel corrosion coupons was set up on a support rack situated next to the 4 m box, together with 316L and 304L stainless steel U-bend specimens (with and without silicone rubber crevice formers), which were used to test susceptibility to atmospheric stress corrosion cracking (ASCC). Strain gauges were applied on a number of different locations on the 4 m box to evaluate mechanical deformation during storage.

Results

A large amount of data was accumulated during the course of the twelve-year monitoring period all of which are available in Winsley *et al.* (2011). Selected examples of the results follow.

Environmental monitoring

The air temperature and box surface temperature were found to follow a daily and seasonal cycle and the daily fluctuations on the surface temperature of the box were smaller than the fluctuations in the air temperature. The external relative humidity fluctuated according to the prevailing atmospheric conditions. It was found that the temperature and relative humidity fluctuations inside the box were much smaller than outside the box, with the internal relative humidity equilibrating at a steady state value of approximately 72%, probably as a result of equilibration with the moisture present in the porewater of the concrete liner.

The data that were collected for a number of years were analysed statistically to determine maximum and minimum values, and the standard deviation. For example, the temperature during 2007−2010 ranged between −7ºC and 32ºC and the relative humidity ranged from 30% to 91%. In addition, the cumulative probability for temperature and relative humidity lying within certain ranges were determined, as shown in Fig. 1 for air temperature and relative humidity. Similar plots were made for surface temperatures at various locations (Winsley *et al.*, 2011).

The surface chloride concentrations were calculated and plotted as a function of exposure time, as shown in Fig. 2. Typical chloride deposition rates were measured as 1−4 µg cm^{-2} yr^{-1} on horizontal surfaces and 0.1−2 µg cm^{-2} yr^{-1} on vertical surfaces. Much
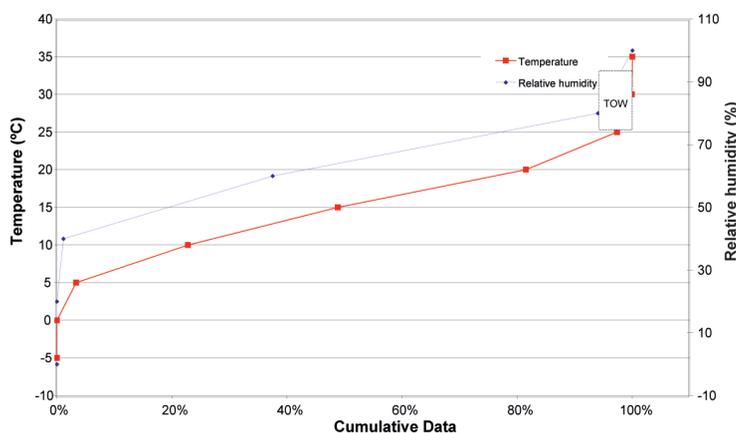

FIG. 1. Results of the statistical analysis (cumulative probability) of the air temperature and relative humidity recorded in the 4 m box storage building (B2 Culham and B404 Harwell) for the period from 2007−2010 and illustrating the area of the data which is defined as time of wetness (TOW) by ISO 9233.
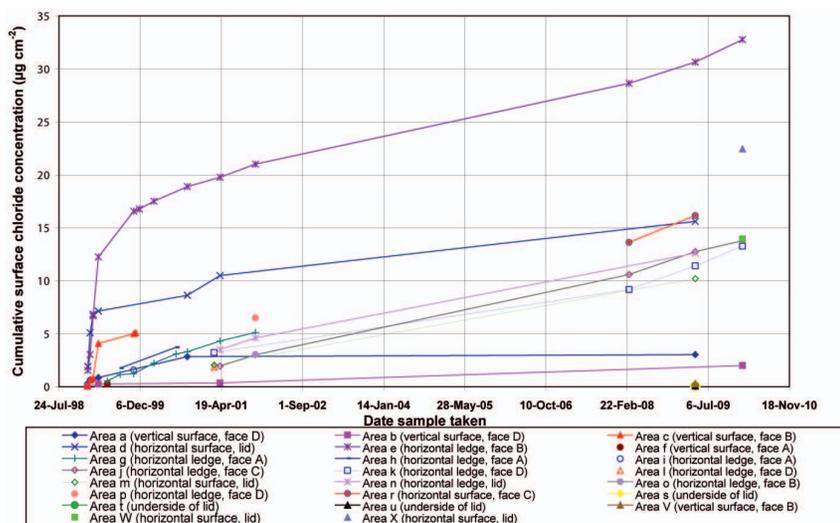
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FIG. 2. Results of surface chloride analyses as a function of time, up to February 2010. The key shows the various locations on the 4 m box where the measurements were made.

higher concentrations of chloride were measured on the base of the box, with concentrations up to 140 μg cm^{-2} being recorded. It was also found that the ratio of chloride to sulfate varied between different locations on the box as shown in Table 1. The concentration distribution of cations in the particulates collected on the swabs is shown schematically in Fig. 3.

Samples of particulates collected on sticky carbon pads were examined and analysed using SEM/EDX analysis. This analysis found a large number of different types of particles and a range of compositions, for example NaCl, Na/CaCl$_2$ and

CaSO$_4$. Particles were typically in the 1−100 μm size range. The particles on the base were found to be predominantly sodium chloride.

When the samples collected by pumping air through the filter paper and bubbler arrangement were analysed it was found that the majority of deposited chloride was present in the larger aerosol particles (above 0.2 μm in size). In particular, the chloride:sulfate concentration ratio in the particles collected on the filter paper was 3.75, whereas it was only 0.49 in the demineralized water (corresponding to particulates finer than 0.2 μm).

TABLE 1. Results of anion analysis from swabs taken from the base and sides of the 4 m box (μg cm^{-2}) in February−March 2008 at Culham. The chloride:sulfate concentration ratio is also reported.

Face	Area	NO$_3^{2-}$	SO$_4^{2-}$	Cl$^-$	[Cl$^-$]:[SO$_4^{2-}$]
Base	*2a*	0.27	5.77	18.87	3.27
Base	*2b*	12.86	6.88	21.57	3.14
Base	*5a*	0.27	3.99	15.31	3.84
Base	*5b*	4.08	4.97	75.22	15.14
Base	*8a*	1.16	5.27	7.43	1.41
Base	*10a*	5.71	10.01	20.24	2.02
C	*j*	7.07	14.99	7.60	0.51
D	*k*	9.11	16.91	5.98	0.35
B	*e*	7.75	15.86	7.66	0.48
C	*r*	11.34	40.82	13.65	0.33

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WASTE CONTAINER DURABILITY

16.4, 0%
950, 22%
286, 7%
226, 5%
2890, 66%
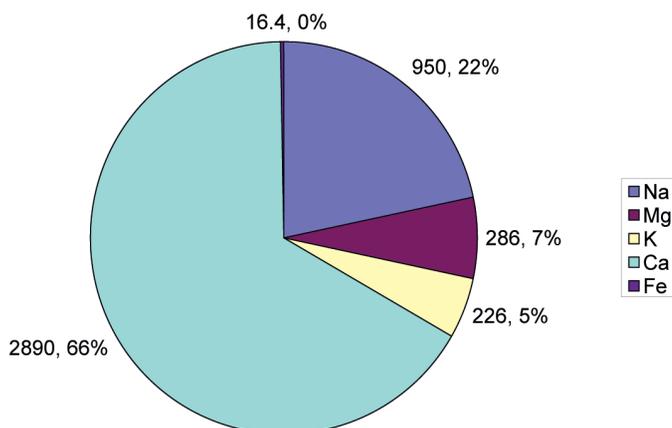
Legend:
- Na
- Mg
- K
- Ca
- Fe

FIG. 3. Relative distribution of cation concentrations in deposits collected from surfaces on the 4 m box, with results expressed as total μg of each cation measured in four samples taken from two ledges and the lid (all 116 cm² sampling areas) and a stainless steel corrosion coupon (75 cm² sampling area). The relative percentage of each cation is also shown.

Indicative measurements of microbial activity (not shown) gave very low counts for the stainless steels surfaces compared to other surfaces in the building (e.g. wood, carbon steel).

Corrosion monitoring: photographic and microscopic analysis of key areas

A number of areas of corrosion were monitored on the surface of the 4 m box over the course of the 12-year period; the areas can be divided into three main categories, as follows: areas on the base of the box where significant corrosion was visible, general areas on the surface of the box where superficial pitting was present and areas where embedded iron present on the surface of the container had led to some superficial rusting (embedded iron was present in areas where carbon steel tools had come into contact with the surface of the box during manufacture and handling). The general appearance of these areas is shown in Fig. 4 and described in further detail below.

The attack on the base of the box, where high chloride concentrations were measured, was predominantly in the form of general discoloration of the butt and stitch/tack welds, which had not been pickle paste treated after manufacture. With respect to the other surfaces of the container, a greater density of pits had also developed in this area (although neither the pit density nor the pit depth were measured). By comparing photographs taken in 2003 with photographs taken in 2008 (not shown) it was found that the degree of

corrosion was similar (i.e. there had not been any significant increase in the extent of corrosion). A cross-section through a weld taken from the base of the box was mounted and polished; no evidence of pitting or SCC of the weld area was observed. Dye penetrant testing of the panels and welds of the 4 m box, including the base of the box where high concentrations of chloride were measured, also showed no indications of SCC.

The rest of the container was generally in very good condition, with very few signs of corrosion. A few areas where black spots were present were noted on the horizontal ledges of the 4 m box (Fig. 4). These spots had the appearance of micropits, although an evaluation of the depth of attack was not carried out. One ledge (Ledge j) with these black spots was shown to have a relatively high coverage of chloride (7.57 μg cm² in March 2008). This type of attack was one of a few isolated regions and not representative of all the ledges of the 4 m box.

Apart from its cosmetic impact, corroded embedded iron is commonly cited as a possible initiation point for localized corrosion of stainless steel (due to its potential ability to act as a crevice former, to enhance the cathodic reactivity of the surface and to form corrosive ferric chloride) and so a sample from one of the main areas showing rusting of embedded iron was cut and analysed metallographically (Fig. 5). This showed the remains of a particle of metallic iron embedded in a mass of iron oxide (rust), but there were no indications of localized corrosion propagating
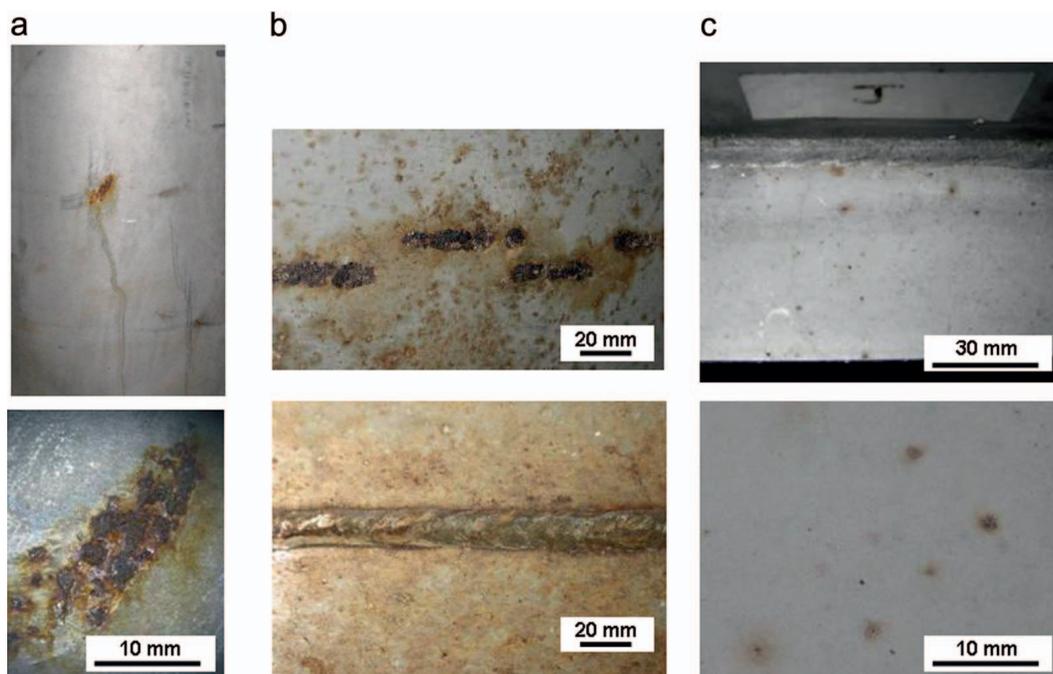
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FIG. 4. Examples of areas of corrosion found on 4 m box. (*a*) Embedded iron (top), higher magnification of same area (bottom); (*b*) areas of corrosion on base of 4 m box; (*c*) superficial pits on 4 m box ledge.
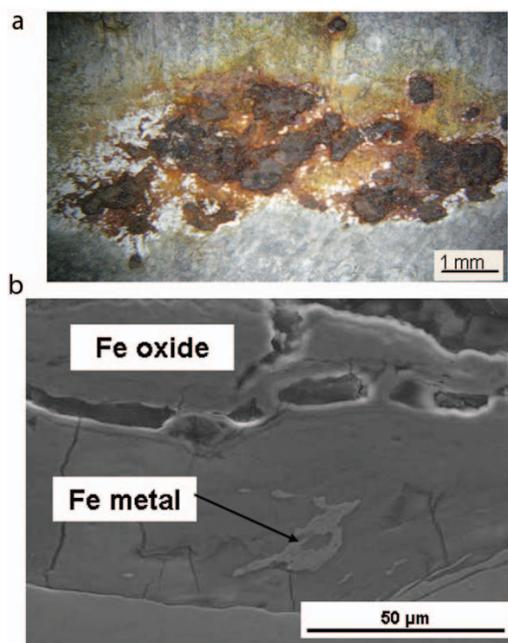

FIG. 5. Embedded iron on the surface of the 4 m box; (*b*) cross-section through the embedded iron, showing the remains of metallic iron in a mass of iron oxide.

beneath the embedded iron into the parent material nor of chromium in the iron oxide (which may have indicated corrosion of the stainless steel).

Corrosion monitoring: photographic and microscopic analysis of corrosion coupons

A coupon of 316L that had been exposed to the same storage atmosphere as the 4 m box from 1999 to 2010 was removed and cleaned in 10% nitric acid to remove any corrosion product in pits on the surface. A number of 10×10 mm areas were mapped using confocal microscopy to establish the maximum pit depth and pit density and the results from the examination are given in Table 2. In carrying out the analysis, it was assumed that any depth measurements above 1.5 μm corresponded to a pit, but depressions <1.5 μm deep were probably due to the inherent surface roughness of 316L (2B finish). The maximum pit depth measured was always less than 7 μm. Optical microscopic examination of the U-bend specimens showed that there was no atmospheric SCC on the U-bend samples.

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TABLE 2. Results of pit depth analysis on a coupon of 316L stainless steel in 2010 after eleven years of exposure to a store atmosphere. The pit density in the range 1.5−2 μm is affected by large uncertainty due to the surface roughness.

Pit density (pits cm^{-2})	Pit depth (μm)
56	1.5−2
7	2−3
2	4−5
1	6−7

Discussion

Environmental monitoring

The data obtained for variations in temperature and relative humidity were analysed to make an approximate assessment of the time of wetness of surfaces in the storage buildings, based on standards available in the literature. For example, by applying ISO 92239 (International Organization for Standardization, 1992) and ASTM G 84-89 (ASTM, 2012) it was possible to calculate the time of wetness (TOW), based on the definition given in these standards. On the basis of these standards, the total time of wetness experienced by the 4 m box surface was only 5−6% of the overall monitoring period, which is equivalent to ~500 hours per year. This assessment does not take account of any deliquescence effects that might arise as a result of hygroscopic particles depositing on the surfaces of the box[1]. In the future, the data for the relative humidity and temperature collected in this programme could be used to evaluate a more realistic TOW considering the hygroscopic behaviour (e.g. deliquescence range) of the type of particulate identified on the surface.

[1] The TOW, defined as the period of time when the metal surface is sufficiently wetted for corrosion to occur, is a parameter which depends on both climatic conditions and on the nature of a surface. In these standards, wetness is assumed when the temperature is above 0ºC and the relative humidity above 80%. This however is a simplification as the conditions and degree of wetness are likely to depend on the nature of the hygroscopic particles deposited on the surfaces of the metal and their deliquescence behaviour.

The particulate material deposited on the surfaces of the 4 m box exhibited a wide range of compositions, sizes and morphologies. The observed size distribution of the particulates is consistent with the bimodal distribution discussed in the literature (e.g. Leygraf and Graedel, 2000), with the larger particulates originating from a number of mechanical proceseses, such as wind blowing of dust and sea spray, and smaller particulates arising from condensation and coalescence processes. Analysis of the chloride:sulfate concentration ratio in particulates that passed through a 0.2 μm filter indicated that the majority of the chloride measured on the surfaces was present in the larger particulates. The particulate deposition rate was significantly higher on the horizontal surfaces compared to the vertical surfaces and this effect is presumably related to gravitationally enhanced settling processes. The information obtained about particle size distribution may have ramifications for the design of store filtration systems.

The high concentration of chloride on the base of the 4 m box is due to the road salt that was deposited during transport of the box to Dounreay in 1999. It is noticeable that the chloride to sulfate ratio was considerably higher in the deposits on the base of the box compared to on the upper surfaces of the box. This is because the particulates predominantly originated from road salt, which has a high proportion of sodium chloride. The surface chloride concentration has been shown to have a significant influence on the atmospheric corrosion behaviour of stainless steels. For example, Nakata *et al*. (1997) investigated the corrosion of stainless steel roofs in Okinawa, approximately 1 km from the coast. A correlation was observed between the degree of rusting and the concentration of chloride in the surface deposits. Nakata *et al*. (1997) also measured surface chloride concentrations at 17 other sites across Japan. It was found that the surface concentration of chloride was affected in a complex way by the geometry of the roofs (e.g. the slope of the surface and the presence of sheltering by eaves). Underneath eaves the surface chloride concentration was higher (up to 160 μg cm^{-2}) than on surfaces exposed to the weather (up to 22 μg cm^{-2}), due to a lack of rainwater washing under eaves. In tests on simulated roofs, Tochihara *et al*. (1996) found that the chloride concentration was higher underneath the eaves and that the surface concentration was higher the steeper the angle of the eaves.

After 1 year at a coastal location, the surface chloride concentrations were in the range $2-12$ µg cm^{-2}, values which are comparable to those obtained in the present programme after the box was transported to Dounreay and back.

Corrosion monitoring

The analysis carried out in the monitoring programme (including results from atmospheric corrosion probes and potential measurements which are not shown in this paper) indicate that the rate of corrosion of the box was very low. Most surfaces of the container showed very little sign of corrosion at all. A limited amount of damage was detected on the base (most extensively on welds) and in association with embedded iron particles. Superficial micropitting was also observed on horizontal surfaces (some ledges) where the chloride concentration was relatively high. This type of attack however was one of a few isolated regions and not common. Analysis of pit depths in the 316L stainless steel coupon showed that the pit growth rates were negligible in the storage conditions studied (i.e. <1 µm yr^{-1}).

The results of the analysis of the pit depths and pit density on the 316L corrosion coupon analysed in 2010 (Table 2) can be compared with equivalent data set acquired in 2005. It was found that in 2005 the maximum pit depths were ~5 µm, with a pit density ranging from $11-43$ pits cm^{-2}. Consequently it can be seen that very little further pit propagation occurred between 2005 and 2010 (Winsley *et al.*, 2011).

No evidence of ASCC was observed on any of the 4 m box surfaces. Similarly, eddy current testing on other containers in the store also revealed no evidence of SCC (Raude and Rudlin, 2009). The conditions within the storage environment characterized in the current work do not appear to overlap with ranges of high SCC susceptibility identified within the literature (e.g. Shoji and Ohnaka, 1989; Phan *et al.*, 2008; Albores-Silva, 2011; Albores-Silva *et al.*, 2011), where there has been an emphasis on using pure MgCl$_2$ for the experimental studies rather than the mixed salts found in practice.

Conclusions

A large amount of data was acquired for (1) characterization of a typical inland storage environment, (2) characterization of the atmospheric corrosion behaviour of stainless steel in an environmentally uncontrolled storage environment. In general, there was little sign of degradation on most surfaces of the 4 m box after a twelve year monitoring period and this provides confidence that, if the environment is suitably controlled, stainless steel waste containers will provide the required resistance to external atmospheric corrosion for the long-term interim storage of ILW. Together with other work, the results from this programme have been used as the basis for developing NDA guidance on environmental control in interim stores (Nuclear Decommissioning Authority, 2011). However, further data are required to characterize different store locations and the possible conditions that could arise during GDF operations.

Future corrosion assessments and studies should take into account the surface chemistry likely to develop in underground environments and real ILW stores, including those in coastal locations (e.g. temperature, surface RH, wetting behaviour, particulate deposition rate and composition, deliquescence). The main conclusions from the work described in this paper are as follows:

(1) Typically, the temperature and relative humidity in the inland buildings characterized in this programme ranged from $0-30$ºC and $40-90$% respectively, but more extreme values were observed for short periods. By taking account of standard definitions of time of wetness, statistical analysis of temperature and relative humidity data suggests that wetting occurred rarely in the Culham building (~5% of the time on simple ISO/ASTM criteria), but this assessment ignores deliquescence effects. The data obtained could be used to assess the likelihood of deliquescence for different compositions of particles.

(2) A wide range of particulate types and compositions was found on the surface of the container. The particle size varies from <$0.1-100$ µm, with a range of anions present, of which chloride was the most significant in terms of corrosion behaviour, but sulfate and nitrate were also present. These may act as inhibitors of pitting but may also affect the deliquescence properties of the surface (e.g. they may widen the range of humidity in which wetting occurs).

(3) Chloride concentrations were higher on horizontal surfaces than vertical surfaces, with a deposition rate in the range $0.1-4$ µg cm^{-2} yr^{-1}. The chloride concentration in airborne and deposited particulates was related to particle

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size, with most being concentrated in large particles.

(4) Corrosion of embedded iron did not cause corrosion of the underlying stainless steel in the storage environment studied.

(5) The most severe corrosion occurred on the base of the box, which had high concentrations of chloride as the result of exposure to road salt ($>100\ \mu g\ cm^{-2}$). However, there was little change in the extent of corrosion over the last five years, probably due to the relatively benign environmental conditions in the building.

(6) A few areas on the ledges of the box showed indications of the initiation of apparently superficial micropits in regions of relatively high chloride contamination (approaching $10\ \mu g\ cm^{-2}$), but their depth has not been characterized.

(7) There were no indications of SCC on the base of the 4 m box or the U-bend specimens, despite the high chloride concentrations that were measured. Flat 316L corrosion coupons exhibited very low pit propagation rates ($<1\ \mu m\ yr^{-1}$).

Acknowledgements

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