6 Assessment of materials used for anoxic microenvironments

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Abstract For various reasons, museum objects may require to be stored in environments with low oxygen levels and/or low levels of fluctuation in relative humidity (RH). Geological specimens that are susceptible to pyrite decay (oxidation of the mineral pyrite) or specimens that have undergone treatment for pyrite decay are in this category. For this purpose, low-oxygen enclosures can be used. This study has assessed the effectiveness of various materials that can be used in the manufacture of anoxic microenvironments. The results indicate that Escal barrier film is more effective than BDF 200 barrier film, and is also a better moisture barrier. A double-skin enclosure is more effective as an oxygen barrier. Commercially available oxygen scavengers are very effective in removing oxygen within barrier film enclosures, while purging with oxygen-free nitrogen has no apparent benefit. Oxygen indicator eyes do not give entirely consistent results and so should be used with care.

Keywords: storage of material containing pyrite, oxygen scavengers, oxygen indicator eyes and low-oxygen enclosures

Introduction

When considering geological collections it is common to assume that objects are quite robust. However, it has been known for a considerable time that geological specimens are subject to various types of deterioration (Howie 1979). One such problem associated with geological material is the breakdown of iron sulphide – a reaction commonly referred to as pyrite decay.

Iron sulphide (FeS_2), present as the minerals pyrite and/or marcasite, is found as an accessory phase in many rock types. It follows that many rock, mineral and fossil specimens held in museums, as well as stone objects in archaeological and ethnological collections, contain significant quantities of pyrite. Increasing and/or fluctuating relative humidity (RH) can cause breakdown in susceptible pyrite material (Howie 1977a,b, 1992). Pyrite breakdown involves the release of sulphuric acid vapours. If the reaction is not halted, the specimen may completely disintegrate. The sulphuric acid can burn specimen labels making them brittle and difficult to read; it can also burn cardboard storage boxes or wooden cases that are in close contact with the specimens. However, most damage to specimens results from the volume change incurred in the hydration of the iron(II) sulphate from the 1 hydrate to the 7 hydrate form which could involved a volume change of over 100% (Waller 1987, 1992). The oxidation to iron sulphate (a white/yellow residue or powder) and the release of sulphuric acid vapours is often accompanied by the loss of surface shine on pyrite specimens. The powder produced has a strong acidic /sulphurous smell – a common indicator of pyrite decay. In the absence of this characteristic smell, a simple surface pH measurement with an indicator strip showing a pH below 5 is usually sufficient to confirm the presence of pyrite decay (Howie 1977a).

Although pyrite can be present in many rock types, not all specimens containing pyrite are vulnerable to decomposition. Some localities are known to be the source of vulnerable pyrite that may decompose quickly. Pyrite from other localities may remain stable even if stored under the same RH and temperature conditions. A list of some localities containing vulnerable pyrite can be found in Bannister (1937).

Fluctuating temperature and RH together encourage pyrite to oxidise (Howie 1979) therefore controlling fluctuations in temperature and humidity are important factors in the care of fossils and minerals, as well as other stone museum objects (Collins 1988). Generally, geological material at risk should be stored at RH less than 40% (Howie 1992). However, the museum environment is often a compromise of the most suitable conditions for a range of object types (as well as the comfort of staff!). Once pyrite decay has started in unprotected specimens, it is irreversible, but controlling the environment – i.e. maintaining a low RH, providing an oxygen-free atmosphere, ensuring only small fluctuations in temperature and using acid-free storage and packaging material – can prevent further deterioration.

Within the National Museums of Scotland (NMS) Geology Section, specimens considered to be at risk of pyrite decay or specimens that have been treated for pyrite decay are stored in laminated low-oxygen enclosures. Oxygen levels are kept at 0.1% (maintained by the use of oxygen scavengers within the enclosures). RH within the enclosures is maintained at 30–35%, thereby significantly decreasing the probability of pyrite decay.

The NMS currently applies a pyrite decay treatment during which minerals and fossils are exposed to an ammonium hydroxide and polyethylene glycol (PEG 400) solution in a sealed container (Waller 1987). The length of this treatment varies depending on the size of specimen. Vapour from the solution neutralises the acidic products of pyrite decay, precipitates iron as an iron(III) oxide and arrests further crystal growth. Any iron oxide precipitated on the surface is removed manually.

Once treatment has been carried out, in order to avoid further deterioration it is necessary to store the specimen below 40% RH or in an oxygen-free environment with RH control. The former is both expensive and requires additional space for specimen storage since the specimens very often cannot be stored in their original location within the collections. Oxidation of iron sulphides in the presence of water vapour can occur at levels as low as 30% RH. Although the generally accepted range for material vulnerable to pyrite decay is 30–40% RH, bone material should be stored at about 45–50% RH (Howie 1979). Therefore, conditions that would be desirable for pyrite decay-treated specimens would not be suitable for bone material. (For a list of the environmental requirements see the chapter by Waller in Howie 1992).

The latter storage option – that of maintaining the specimens in oxygen-free micro-environments – not only discourages oxidation of the pyrite through lack of oxygen but, with careful selection of barrier film, can also provide RH-controlled microenvironments, making this a more practical solution in terms of medium- to long-term storage.

In order to achieve such conditions, the specimens are placed in low-oxygen enclosures following treatment for pyrite decay. The specimen is placed in an envelope of oxygen barrier film that is purged with nitrogen and sealed. Any remaining oxygen within the envelope is removed using an oxygen scavenger such as Ageless (Lambert *et al.* 1992; Daniel and Lambert 1993). Oxygen levels and RH within the envelopes are monitored using commercially available oxygen indicator 'eyes' and humidity indicator strips respectively. The specimens are returned to storage within the collections where they are monitored. However, over the course of three years of this systematic programme of treatment and storage, it was observed that the medium-term stability of the low-oxygen enclosures was not uniformly reliable. Since there is a variety of materials available and slightly differing procedures for manufacture of the enclosures, an experiment was designed to test the effectiveness of resulting enclosures from the possible combinations of materials and processes. We have assessed various types of oxygen barrier film and oxygen scavengers for effectiveness over a range of RH values. The study is also investigating the longevity of the low-oxygen enclosure in order to determine how often the envelopes require to be replaced, and the effectiveness of these anoxic microenvironments in the treatment of pyrite decay.

It should also be noted that low-oxygen enclosures are also used in other areas of conservation such as pest control (Daniel and Lambert 1993) or storage of materials such as plastics that are susceptible to deterioration in normal atmospheric conditions. Clearly, points relating to the general effectiveness of such enclosures are relevant to all areas that use these materials and processes.

Materials and equipment for making low-oxygen enclosures

- Cardboard photographic film boxes 12.5 cm × 10 cm with pH 7.
- Escal, a thick, ceramic-deposited gas and moisture barrier polymer film. The film is supplied in rolls allowing different sizes of enclosures to be made. Sealing was done either with a heat sealer or with an Escal clip.
- BDF 200 is a 25 μm thick, heat-sealable, transparent, oxygen and moisture barrier film.
- Ageless products are packaged oxygen absorbers. Ageless chemically absorbs oxygen and generates some heat and moisture in initial reactions. Therefore it should not be put in direct contact with the specimens. The selection of the appropriate type and number of Ageless sachets is important. A method for calculating the oxygen volume in an enclosure and therefore the number of oxygen absorber packets needed is available (Mitsubishi Gas Chemical Company, leaflet 96.4). The absorbers are supplied in a vacuum-sealed pouch. Oxygen absorption begins immediately after the master pouch is opened so the sachets should be used as quickly as possible.
- Ageless Z removes oxygen and one sachet will absorb the oxygen from a litre of air. The deoxygenation time at room temperature is one to four days depending on the size of enclosure and the ambient temperature. The maximum exposure time to open air is four hours. Escal combined with Ageless Z will retain the moisture given off during the active period of the Ageless Z (Conservation by Design, February 1999).
- Ageless RPA-5 removes oxygen, moisture and corrosive gases. Escal combined with Ageless RPA-5 will maintain low RH. It can therefore be used where high humidity adversely affects the specimens and where a low RH is required. It is also used where oxygen and moisture are primary sources of deterioration.
- The Ageless Eye is an in-package colour change monitor for oxygen levels. A pink colour indicates 0.1% or less oxygen inside the enclosure. A bluish colour indicates the presence of 0.5% or more oxygen in the enclosure. The lower the temperature, the more slowly the colour of the eye will change. The 'eyes' are stored under refrigeration in oxygen-free vacuum packs prior to use (Mitsubishi Gas Chemical Company 1994).
- Audion Sealmaster 620 electric heat sealer.
- Escal clip: a clip designed to seal an open bag effectively.
- Humidity indicator cards provide a simple indication of RH within 10% bands.

Method for making low-oxygen enclosures

- Select a container (cardboard box with or without a mineral/fossil specimen) and estimate its dimensions to calculate the volume of oxygen.
- Select the oxygen barrier film, cut and make a bag to enclose the container.
- Heat-seal three sides of the bag.
- Place the container in the bag.
- Choose the type of oxygen scavenger required.
- Calculate the number of oxygen scavengers required. Place them inside the bag.
- Put the oxygen indicator eye(s) and the humidity indicator card inside the bag.
- Heat-seal the bag, leaving a small gap for possible flushing with oxygen-free nitrogen.
- Purge oxygen from the bag using oxygen-free nitrogen for around 30 seconds.
- Seal the bag using heat or clip as required.

The following points were considered:

- Estimation of the colour shown by humidity indicators can vary from one observer to another, hence a single observer was chosen for all colour demarcation estimations.
- The colour of the Ageless Eye was similarly assessed.
- Exposure of the oxygen scavenger Ageless to oxygen prior to use could make the scavengers ineffective.
- Inappropriate quantities of the oxygen scavengers could have been used.
- Escal and BDF 200 need to be completely flat for perfect sealing.
- Enclosures may not be well sealed. Creases or foreign substances on the seal area could render the seal faulty.
- Barrier film used could be faulty or damaged.
- Oxygen indicator eyes could be faulty or inaccurate.
- Oxygen scavenger sachets could be faulty.
- Boxes could have higher or lower initial humidity levels depending on where they were stored prior to the experiment (boxes were taken from different stores).
- UV could affect the barrier film since sealed containers were stored in an area open to diffused natural light.
- Escal and BDF 200 film could deteriorate after a period of time.
- Ambient humidity and temperature varied throughout the experiment: 30–40% RH and 15–30 °C.

The experiment involved preparing and monitoring 13 different combinations of these variable factors (see Table 6.1). In order to test variability due only to materials and procedures, no specimens were included in the enclosures.

Experimental results

The different combinations and results for the whole experiment are set out in Table 6.2. They can be subdivided into three groups:

- G.1 Samples 5, 8, 9, 10 and 13
  These enclosures showed little fluctuation in RH over the course of the experiment (Table 6.3). Enclosures 8, 10 and 13 indicated low to no oxygen presence. Enclosures 5 and 9 indicated low initial oxygen levels but a sudden increase in oxygen between readings suggesting a possible breach in the seals/barrier films during the course of the experiment.

- G.2 Similar patterns of fluctuation in RH levels were exhibited by enclosures 1, 2, 3, 4 and 12 with RH showing an initial decrease of 10% to 20% within a few weeks and then increasing to an acceptable 30–40% (Table 6.4). The oxygen levels in each of these bags however showed variable patterns.

- G.3 Enclosures 6, 7 and 11 showed RH decreasing to zero over the course of the experiment. The oxygen levels are generally low (Table 6.5).

Table 6.1 All combinations of materials and procedures used.

Starting date and sample numbers: 28 June 2000	Presence (1) or absence (0) of Escal ceramic barrier film	Presence (1) or absence (0) of Bdf 200 barrier film	O_2 scavenger Ageless Z: Number of sachets used	O_2 scavenger Ageless RPA-5: Number of sachets used	Presence (1) or absence (0) of N_2	Presence (1) or absence (0) of Escal clip	Heat sealer setting	Ageless Eye: colour when started	RH (%) at start
1	0	1	2	1	0	0	5	Pink	30–40%
2	0	1	2	1	1	1	5	Pink (eye broken)	30–40%
3	0	1	2	1	1	0	5	Pink	30–40%
4	0	1	0	1	0	0	5	Pink	30–40%
5	0	1	2	0	1	1	5	Pink	30–40%
6	1	0	0	1	1	1	5	Pink	30–40%
7	1	0	0	1	1	1	5	Pink	30–40%
8	1	1	2	1	1	1	5	Pink (eye broken)	30–40%
9	1	0	2	0	1	1	5	Pink	30–40%
10	0	1	4	0	0	0	5	Pink	30–40%
11	1	0	0	2	0	0	5	Pink	30–40%
12	0	1	0	2	0	0	5	Pink	30–40%
13	1	0	4	0	0	0	5	Pink	30–40%

Table 6.2 All results.

Starting date and sample numbers:	Presence (1) or absence (0) of Escal ceramic barrier film	Presence (1) or absence (0) of Bdf 200 barrier film	O_2 scavenger Ageless Z: Number of sachets used	O_2 scavenger Ageless RPA-5: Number of sachets used	Presence (1) or absence (0) of N_2	Presence (1) or absence (0) of Escal clip	Heat sealer setting	Ageless Eye: colour when started	Final RH (%) and Ageless Eye Colour								
28 Jun 2000									3 Jul 2000	10 Jul 2000	17 Jul 2000	27 Jul 2000	23 Aug 2000	19 Sep 2000	2 Mar 2001	28 Jun 2001	18 Mar 2002
1	0	1	2	1	0	0	5	Pink	30 P/B	20 B	10 B	10 B	10 B	20 B	20 B	30 B	30 B
2	0	1	2	1	1	1	5	Pink (eye broken)	30 P	20 P	20 P	20 B/P	20 B	20 B	20 B	30 B	30 B
3	0	1	2	1	1	0	5	Pink	30 P	10 P	10 P	10 P	10 P	30 P+	20 P+	30 P+	30 P
4	0	1	0	1	0	0	5	Pink	20 B	10 B	10 B	10 B	10 B	20 B	20 B	30 B	30 B
5	0	1	2	0	1	1	5	Pink	40 P	40 P/B	40 B	30 B	20 B	30 B	30 B	30 B	30 B
6	1	0	0	1	1	1	5	Pink	20 P	10 P	10 P	10 P	10 P	10 P+	0 P/B	0 P/B	0 P/B
7	1	0	0	1	1	1	5	Pink	20 P	10 P	10 P	10 P	10 P	10 P+	0 P/B	0 P/B	0 B
8	1	1	2	1	1	1	5	Pink (eye broken)	30 P	30 P	30 P	30 P	30 P+	30 P+	30 P+	30 P+	30 P+
9	1	0	2	0	1	1	5	Pink	50 P	50 P	50 P+	50 P+	50 P	50 P+	50 B	50 B	50 B
10	0	1	4	0	0	0	5	Pink	50 P+	50 P+	50 P+	50 P	40 P+	40 P+	30 P+	40 P+	40 P+
11	1	0	0	2	0	0	5	Pink	10 P/B	10 P/B	10 P/B	10 P	10 P/B	10 P+	0 P+	0 P+	0 P
12	0	1	0	2	0	0	5	Pink	10 P/B	10 B/P	10 B/P	10 B/P	10 P/B	10 P+	10 P+	40 P+	40 P+
13	1	0	4	0	0	0	5	Pink	50 P+	50 P+	50 P+	50 P+	50 P+	50 P+	50 P+	50 P+	50 P+

P+ = 1, completely oxygen-free; P = 0, oxygen-free; P/B = –1, slight oxygen presence; B/P = –2, intermediate oxygen presence; B = –3, full oxygen presence

Table 6.3 Group 1 results for samples 5, 8, 9, 10 and 13. Sample 9 chosen as representative result.

Starting date and sample numbers: 28 June 2000	Presence (1) or absence (0) of Escal ceramic barrier film	Presence (1) or absence (0) of Bdf 200 barrier film	O_2 scavenger Ageless Z: Number of sachets used	O_2 scavenger Ageless RPA-5: Number of sachets used	Presence (1) or absence (0) of N_2	Heat sealer setting	Presence (1) or absence (0) of Escal clip
5	0	1	2	0	1	5	1
8	1	1	2	1	1	5	1
9	1	0	2	0	1	5	1
10	0	1	4	0	0	5	0
13	1	0	4	0	0	5	0

Sample 9

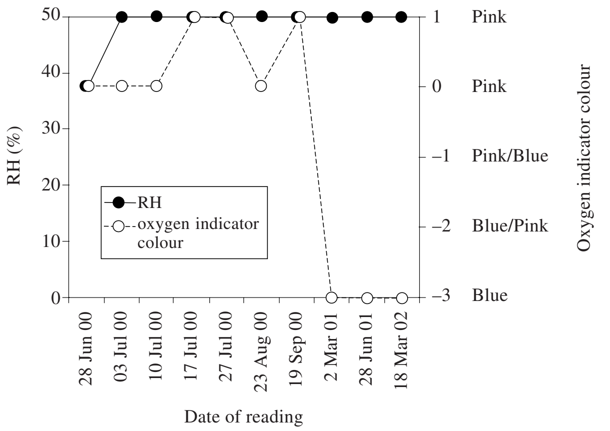


Table 6.4 Group 2 results for samples 1, 2, 3, 4 and 12. Sample 3 chosen as representative result.

Starting date and sample numbers: 28 June 2000	Presence (1) or absence (0) of Escal ceramic barrier film	Presence (1) or absence (0) of Bdf 200 barrier film	O_2 scavenger Ageless Z: Number of sachets used	O_2 scavenger Ageless RPA-5: Number of sachets used	Presence (1) or absence (0) of N_2	Heat sealer setting	Presence (1) or absence (0) of Escal clip
1	0	1	2	1	0	5	0
2	0	1	2	1	1	5	1
3	0	1	2	1	1	5	0
4	0	1	0	1	0	5	0
12	0	1	0	2	0	5	0

Sample 3

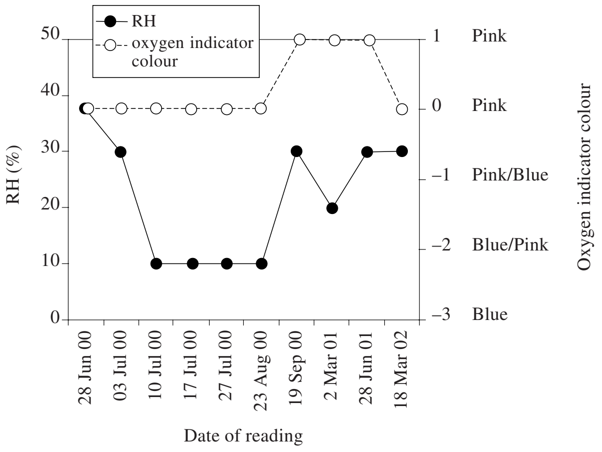
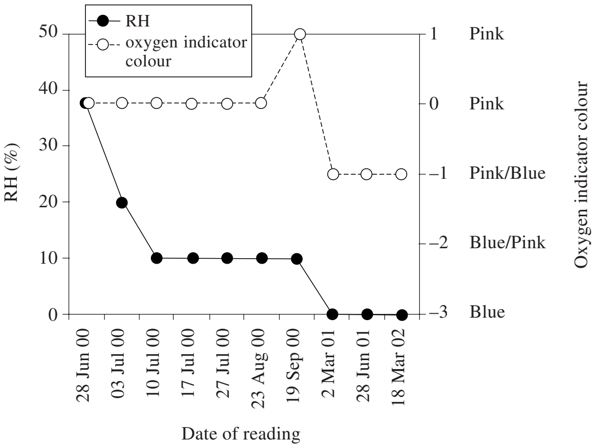


Table 6.5 Group 3 results for samples 6, 7 and 11. Sample 6 chosen as representative result.

Starting date and sample numbers: 28 June 2000	Presence (1) or absence (0) of Escal ceramic barrier film	Presence (1) or absence (0) of Bdf 200 barrier film	O_2 scavenger Ageless Z: Number of sachets used	O_2 scavenger Ageless RPA-5: Number of sachets used	Presence (1) or absence (0) of N_2	Heat sealer setting	Presence (1) or absence (0) of Escal clip
6	1	0	0	1	1	5	1
7	1	0	0	1	1	5	1
11	1	0	0	2	0	5	0

Sample 6



Discussion

From the work carried out here, it is clear that low-oxygen enclosures can be an effective way to maintain low oxygen levels and relatively stable RH conditions.

Using BDF 200 as an internal film barrier and Escal as the external film produced the best results. However, where specimens are enclosed in anoxic bags there may be a risk of boxes and sharp specimens breaking the thin Bdf 200 film thereby reducing the effectiveness of the process.

Sample 8 shows the results nearest to the combination that was deemed optimal for pyrite decay-treated specimens.

The experiment suggests that it can be difficult to achieve consistent results with respect to low-oxygen conditions. This would appear to be more associated with human error in the sealing of the bags and/or possible breach of the barrier films, as noted by previous studies (Burke 1992, 1996). The risk of this can be partially offset by double-bagging the enclosures. It is therefore very important to monitor the conditions within the enclosures on a regular basis. The reliability of oxygen indicator eyes is perhaps not quite as high as the manufacturer's literature would suggest, and it is important to include more than one 'eye' in any enclosure.

The experiments carried out in this study used empty enclosures. Obviously the nature of the objects within enclosures in collections will have a significant effect on the speed and effectiveness of oxygen removal.

With respect to RH, the experiment suggests that the low-oxygen enclosures are reasonable in terms of maintaining stable RH conditions. For all objects placed in low-oxygen enclosures, it is important that they are maintained at the appropriate RH for that object prior to being placed in the enclosures. This will ensure that the 'target' RH is not compromised. Alternatively, conditioned silica gel could be used within an enclosure for objects requiring very specific RH conditions (King 1982, 1983). The humidity indicator cards are a simple, relatively inexpensive method of monitoring RH variation, though they do not give absolute RH values. Again, it is necessary to monitor the enclosures on a regular basis for deterioration in the materials and/or breach of the enclosure.

An environment without oxygen is more important than lower levels of humidity. Fluctuation in humidity will be potentially more harmful than a high or low humidity. Of course, specific conditions will apply depending on the nature of the specimens/objects to be enclosed in the microenvironments. For pyrite decay treatments, storage will be more effective in an environment with no oxygen but levels of humidity at 30–40%.

Conclusions

- Both Escal barrier film and BDF 200 barrier film perform similarly.
- Escal appears to be better suited to maintain the RH levels suitable for pyrite decay-treated specimens.
- BDF 200 does not appear to have the ability to keep humidity stable for long periods and in fact, humidity increases over time. After periods of one to three

months, the moisture barrier becomes less effective and the environment reverts to atmospheric humidity.

- Using Bdf 200 as an internal film barrier and Escal as the external film produced the best results. The use of a double barrier film gives a more robust anoxic bag.
- Escal clips are efficient in sealing anoxic bags.
- The experiment has been carried out without specimens in the enclosures. Clearly, specimens in the microenvironments can vary the results in terms of length of time taken to stabilise the anoxic environment and the danger of abrasion to the bags themselves.
- Available manufacturer's information about Ageless Eye suggests that the colour first turns blue when in contact with oxygen, and then returns to pink after the container becomes oxygen-free. The oxygen indicator eyes in the majority of our experiments did not conform to this. From the 13 samples, only two of the indicator eyes (samples 11 and 12) turned blue before returning to pink. During the experiment we worked quickly transferring the indicator eye from its oxygen-free package to the enclosure. The enclosure was immediately sealed. Perhaps these procedures affected the behaviour of the indicator eye colour.
- Oxygen indicator eyes that were broken but remained sealed within their packet gave results that were consistent with the unbroken 'eyes'.
- Oxygen scavenger sachets worked well. The addition of oxygen scavenger sachets above the recommended numbers produced faster results.
- Surprisingly, purging with oxygen-free nitrogen appeared to have no significant benefit to either the speed or efficiency of oxygen absorption.
- The use of nitrogen in anoxic bags may decrease the risk of abrasion by ballooning the bags away from internal sharp edges.

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Suppliers

Ageless products, Escal, Escal clip, Bdf 200 barrier film and heat sealer: Conservation by Design Ltd, Timecare Works, 60 Park Road West, Bedford MK41 7SL, UK

Mitsubishi Gas Chemical Company, Inc, New Business Planning and Development Division RP Team, 5-2, Marunouchi 2-chome, Chiyoda-ku, Tokyo 100, Japan

Nitrogen gas: BOC, PO Box 12, 12 Priestley Road, Worsley, Manchester M28 2UT, UK

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