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Weep no more: conservation of an iron-nickel meteorite from Canyon Diablo, Arizona

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Abstract

This article documents the treatment of a fragment of the asteroid that created the Barringer meteor crater, officially known as the Canyon Diablo Meteorite. This includes investigations into the condition of the specimen, evaluation of techniques used in meteorite and archaeological iron conservation, and the eventual treatment with tannic acid of an NHMUK specimen.

Keywords: meteorite; conservation; iron; tannic acid; ochtahedrite; storage

Introduction

Meteorites are an invaluable source of information on the early history of the solar system, the composition of planets, the proportions of elements present in the solar system, and how impacts of large meteorites have altered Earth's history and could affect our future. Current research focus includes the study of pre-solar grains to understand our parent stars, how the physics of flight in our atmosphere shapes meteorites, detecting the presence of biological compounds, and the use of non-destructive microcomputed tomography (3D imaging) in conjunction with scanning electron microscopy. Some meteorites remain unaltered for millions, if not billions, of years but, despite an estimated fall of 2900-7300 kg per year of meteorites within the 10g – 1kg range (and 8.7 events weighing over 1kg per year) (Bland et al., 1996), many land in the oceans or climates within which deterioration is extremely rapid (Bevan, 1992). Undeteriorated specimens are very rare and are mostly discovered in the Antarctic, where the dry cold climate allows good preservation and low levels of contamination (Bland et al., 2006). Meteorites are divided into

three main groups: irons, stones and stony-irons, but there are many subclasses. The largest group of meteorites is the stones (mainly silicate minerals), once forming part of the outer crust of a planet or asteroid. Some stone meteorites (chondrites) contain tiny grains pre-dating the formation of our solar system. Achondrites include material from the moon, mars and asteroids (Lotzof, 2018). Iron meteorites form the second most common type and were once part of the core of a planet or large asteroid. The majority of iron meteorites contain 90-95% iron, plus nickel and trace elements. Iron meteorites are subdivided into classes both by chemical composition and structure. Structural classes are determined by studying their two component iron-nickel alloys: kamacite and taenite (Notkin, 2019). The stony-irons, account for less than 2% of all known meteorites. They are comprised of roughly equal amounts of nickel-iron and stone and are divided into two groups: pallasites and mesosiderites. The pallasites are thought to have formed at the core/mantle boundary of their parent bodies, revealing details about the structure of planets, whilst mesosiderites are



© by the author, 2020, except where otherwise attributed. Published by the Natural Sciences Collections Association. This wok is licenced under the Creative Commons Attribution 4.0 International Licence. To view a copy of this licence, visit: http://creativecommons.org/licences/by/4.0/ believed to form when debris from a collision between two asteroids are fused together (Lotzof, 2018).

As a meteor travels through the atmosphere the frictional heating causes its surface to melt and vaporise. The melted material is stripped away, creating the characteristic indents (regmaglypts). As the meteor cools, the surface solidifies to form a thin shiny fusion crust (Bevan, 1992). When a meteorite hits the Earth, a crater may be formed but the bulk of material can be destroyed by vaporisation. At Meteor Crater in Arizona, 30 tonnes of meteorite fragments were discovered at the crater rim and in the surrounding plains, including the large Canyon Diablo specimen, currently at the Natural History Museum (NHM) in London (UK) (NHM BM. 1959.1052) which is the subject of this article.

Storage of meteorites

Different types of meteorites require different storage environments. Iron-nickel meteorites are better preserved in dry environments, which would be unsuitable for carbonaceous chondrites which contain up to 20 wt% water, mainly in phyllosilicates and hydrous sulphates such as epsomite, which will readily effloresce and cause delamination of the fusion crust if allowed to dry out (Bevan, 1992).

Contamination is a primary concern for meteorites, handling procedures and storage media must be strictly controlled, and a record of materials which have been in contact with the specimen should be made, for elimination of elements when research is undertaken. Meteorites can be identified by weight, avoiding the need to adhere labels to them (Bevan, 1992). Even if contamination is not a concern, contact with bare skin must be avoided because chlorides from sweat residues will accelerate iron corrosion (Walker, 1982) and finger-prints can often be observed on inappropriately handled specimens. Pollution can also be a source of acids, such as sulphuric acid, which are hygroscopic and will contribute to iron corrosion (Selwyn, 2004) and deterioration of calcium-based minerals. Materials that are considered appropriate for storage of meteorites at the NHM include glass, aluminium foil cleaned with isopropanol, and un-coloured platinum-cured silicone, polytetrafluoroethylene, polyester, polyethylene and polypropylene. Only unused plastic should be used to store meteorites, since plastic molecules are easily transferred between materials, leading to potential cross-contamination (Smith, 2017). Kebukawa et al. (2009) recommend that glass and polystyrene are the only suitable

storage media for certain types of meteorite.

Meteorites should be stored at a stable relative humidity (0-10% for iron meteorites and 35-45% for carbonaceous chondrites (Almeida, 2019)). Double polythene bags are a protection from contamination, but they will only protect against changes in humidity if appropriately conditioned silica gel is included. Relative humidity can fluctuate dramatically in a sealed environment with no controls, when temperature changes. AMNH (2008) recommend a triple ziplock bag system to maintain low humidity but avoid contamination from silica gel dessicants: "The bag containing the sample (and is still open) is placed inside the bag containing desiccant. The desiccant bag is sealed, minimizing the amount of air in both bags, and left for 20 minutes. Once the allotted time has passed, the interior sample bag is sealed without opening the outer desiccant bag. The sample should no longer be in contact with the desiccant. Finally, the desiccant bag is placed inside a third Ziploc, which is then sealed, to ensure that there is no longer any air exchange."

At the NHM, a variety of storage techniques are used to safeguard the specimens. These range from a display case, constantly replenished with positive pressure nitrogen, to small microenvironments in the collections storage area with oxygen and/or humidity control where appropriate. Iron meteorites should be stored in anoxic environments similar to those constructed by Trafford and Allington-lones (2017), these can be made dry by using oxygen scavengers such as RP-System A by Mitsubishi Gas Chemicals. Oxygen levels must be less than 3% to prevent corrosion (Walker, 1982). Certain specimens subjected to many years in the field, inappropriate storage or open display, however, have suffered from corrosion.

Deterioration of iron meteorites

The most damaging corrosion of iron meteorites is caused by moisture and air, accelerated by terrestrially derived chloride ions (Bevan, 1992). In a burial environment, at the interface between the iron and the corrosion products, iron (II) ions dissolve, accumulate and hydrolysis occurs, in turn leading to local acidification which increases the solubility of iron ions (Selwyn, 2004). The major corrosion products are akaganéite and goethite (Bevan, 1992). The former decomposes to form maghemite and goethite, releasing chloride ions to the corrosion front to re-initiate corrosion. In addition, the small crystal size of akaganéite means its water absorption capacity is very high (Bevan, 1992). Active corrosion can sometimes be identified because akaganéite is orange and goethite is brown (Knight, 1982).

Corrosion of iron is uneven due to the creation of cathodic and anodic areas (in electrochemical reactions positive ions flow from the cathode and oxidation occurs at the anode). At the cathode hydroxide ions are produced, increasing pH, and at the anode ferrous ion hydrolysis causes a reduction in pH. The acid increases the solubility, and causes dissolution of iron oxide-hydroxides (Turgoose, 1982). In addition, chloride ions will concentrate at the anodes, contributing to corrosion reactions by increasing the conductivity of the aqueous phase of electrochemical corrosion (Turgoose 1982). Chloride ions are not necessary for all iron corrosion, but they are the main accelerator (Turgoose, 1982; Watkinson, 1996).

Areas of active oxidation are porous and allow oxygen and moisture to react with the layers below and continue to oxidise (Logan and Selwyn, 2007). The chloride ion reacts with the iron to form a hydrous ferric chloride which is deliquescent and will then react with oxygen to continue a cycle of deterioration in the meteorite (Pickard, 2005). Small actively corroding pits form, containing acidic solution which promotes pit growth (Selwyn, 2004). This pitting corrosion is promoted by the presence of chloride ions (Selwyn, 2004). Chlorides are hygroscopic so they encourage electrochemical corrosion and also increase the conductivity of the solution (Walker, 1982). Chlorides are present in seawater, soil and groundwater so any meteorite find is likely to be contaminated. The presence of nickel can in some cases reduce the rate of corrosion reactions but it can also cause concentrated corrosion at weak points (due to an imbalance between anodes and



Figure.1 The Canyon Diablo specimen (NHM BM. 1959.1052) in its old wooden crate.

cathodes) leading to local intense attack (Walker, 1982).

The NHMUK Canyon Diablo specimen

The Canyon Diablo specimen is a coarse ochtahedrite meteorite IAB Og 2 (mostly iron and nickel) weighing almost 100 kg, from the Barringer Crater in Arizona (Figure 1). The asteroid is believed to have fallen 50,000 years ago and this fragment was collected in 1891 and acquired by the NHM in 1959. It was initially stored in a wooden crate in a dark storage area, lacking environmental control.

Condition

Outline sketches of the specimen were created and used to form condition maps of each surface, a useful technique on objects for which pinpoint locations are difficult to describe (Figure 2). Approximately 60% of the surface is covered with a black compact and adherent layer of inactive oxidised iron, but 30% by a thin porous orange-brown corrosion layer, which is most prevalent around areas which show abrasion. 10% of the surface is composed of patches of orange akaganéite which has caused spalling as the crystals grow at the metal-rust interface (Selwyn et al., 1999) (Fig. 3a). In the case of Canyon Diablo, environmentally derived chlorides may have been exacerbated by the presence of acids and salts derived from rodent urine at some point in the specimen's long history: the greatest concentration of spalling is located on the upper surfaces of the specimen and analysis using LEO 1455 VP SEM (variable pressure scanning electron microscope) revealed the presence of elevated levels of chlorides in these areas. Discrete patches of weeping iron were also observed (Fig. 3b). Weeping or sweating is caused by high humidity and high concentrations of chloride salts. When humidity decreases, the liquid precipitates as iron hydroxide oxide droplets and forms shiny crusts or orange blisters (Selwyn, 2004; Logan and Selwyn, 2007). In its liquid state, this is acidic and will eat away at the iron (Logan and Selwyn, 2007) so stabilisation or storage at low relative humidity is essential for weeping iron meteorites. The patches on the specimen correspond to areas which had been in contact with the wooden crate. These were presumably caused by the localised higher humidity and concentration of formic and acetic acids generated by the deterioration of the wood (Selwyn, 2004).

Possible stabilisation techniques

The corrosion patches could be removed using airbrasive, a glass bristle brush, Waller sodium salts



Figure 2. (a) The Canyon Diablo specimen after removal from the crate; (b) A condition map of a similar elevation. Object maps are an excellent way of recording the condition of specimens which are difficult to describe verbally. They are quick and easy to refer to when checking future deterioration, and they can be invaluable in aiding pattern recognition which can lead to the identification of the cause of deterioration.

solution (Waller, 1980), commercially available corrosion removers (such as Biox Gel) or with abrasive polishing compounds. Logan and Selwyn (2007) recommend that corrosion layers on archaeological iron are removed from objects by rubbing gently with fine steel wool (000 or 0000 grade) and a few drops of light oil (e.g. sewing machine oil). Clean, lint-free cloths wetted with mineral spirits are then used to wipe off the resulting oil/rust slurry. This is followed with a thin fresh coat of oil, applied with a clean cloth. Oil enhances the surface appearance of the object and leaves a film, which may act as a thin vapour barrier that temporarily protects the underlying iron against further corrosion.

Meteorite dealers have been known to use commercial products such as RustGuardlt, Rig, Rig2, Sheath, and WD40 to treat specimens (Twelker, 2018). Bathurst Observatory in Australia have traditionally used light oil, which requires removal and a reapplication every six months or a coating of a protective polyurethane (Pickard, 2005). They later adopted an alkaline treatment in which meteorites were wrapped in aluminium foil and immersed in hot water and sodium carbonate for 2-4 hours.

If left too long, formation of the mineral limonite (iron hydroxide) occurs, which can be rubbed off with a cloth. Several treatments were sometimes



Figure 3. (a) Spalling and (b) Weeping, on the Canyon Diablo specimen.

found to be necessary to stop weeping and some re-treatment was found to be necessary in subsequent years (Pickard, 2005). Results have been mixed, depending on the type of meteorite treated - Pallasite slices have suffered recurrent weeping, but some Campo del Cielo specimens did not suffer a recurrence of corrosion for 12 years, and even one open-air specimen has not suffered significant deterioration (pers. comm. R. Pickard, Bathurst Observatory, 3 February 2018).

San Diego Natural History Museum (California) only use anoxic storage to prevent corrosion after discovering that shellac and other coatings are ineffective (Shelton, 1995). Harvard University Mineralogical and Geological Museum and Museum National D'Histoire Naturelle (Paris) store selected specimens at 0% RH (Alonso-Perez, 2019; Gounelle, 2019), whilst The Centre for Meteorite Studies (Arizona State University) store iron meteorites at 10-15% RH and use nitrogen cabinets for carbonaceous chondrites (Garvie, 2019). Most institutions, however, do not have the financial resource or facilities to store specimens at specific environmental conditions. At the New England Meteororitical Services, all new acquisitions and any specimens which undergo sampling, are instead immersed in 99.9% alcohol warmed to 35°C for 10-15 minutes before air-drying, to remove contaminants such as lubricants from cutting blades. They have found that this treatment is effective to prevent most types of deterioration (Kempton, 2019).

Many treatments of archaeological iron are concerned with the removal of chloride ions since they are present in the majority of corrosion products on artefacts (1.6-14.0% of artefact content from marine sites, and 0.5-1.0% of artefact content from soil-based sites consist of chloride ions). The chloride content of archaeological artefacts far exceeds that of their burial environment, because the chloride ions carry the current to the anode during corrosion (Turgoose, 1982). In archaeological conservation, soluble salts were traditionally removed by immersion in water (Johnson, 1998). Other aqueous treatments include pressurised water and repeated boiling but both methods can cause physical deterioration (Watkinson, 1982) and lead to further severe corrosion (Blackshaw, 1982). Watkinson (1996) found non-aqueous extraction methods (ethanol and ethanoic lithium hydroxide) to be ineffective. Watkinson (1996) recommends alkaline sulphite treatment followed by Soxlet hot wash (immersion at 60°C over 60 days) to extract chlorides from archaeological iron. Watkinson (1996), however, warns of residual chemicals, physical damage and

that no guarantee can be made that corrosion will not continue to occur.

The presence of chloride ions interfere with some treatment reactions, and a higher pH is necessary for passivation (Selwyn, 2004). Bevan (1992), however, warns that caustic immersion treatments pose great risks of leaching, reaction with mineral assemblages, and the production of more corrosion than had previously existed. Coating the surface of iron increases resistance in the corrosion circuit and slows the rate of reaction (Walker, 1982). Protective coatings like waxes and lacquers do not stop the transmission of water vapour and oxygen, however, they only reduce them. Crystalline waxes have good barrier properties and are more effective than films made from polymer solutions or polymer powder coatings, but they have low resistance to strain from thermal and mechanical shock, whilst solvent -free coatings delivered by spraying will not penetrate pores in corrosion products (Pascoe, 1982). In addition, when applied to porous corrosion layers, the coatings may be very difficult to remove if the object continues to actively corrode (Logan et al., 2013). Waxes are particularly difficult to remove from heavily corroded iron surfaces, so are not normally recommended for use on rusted iron.

The use of iron-specific corrosion inhibitors would be risky on meteorites, since they can react adversely with other metals (Walker, 1982) but tannic acid has been used on archaeological iron for over 50 years. The treatment can produce a blue-black coating resembling uncorroded iron, and is suitable for iron stored indoors. The acid reacts with the corrosion layers to form ferric tannate, which will prevent the most susceptible areas from re-rusting in the short term (Logan and Selwyn, 2007). Tannic acid treatment allows iron to be stored at much higher relative humidity - up to 50% (Logan et al., 2013) but will not protect specimens from exposure to even higher relative humidity levels (Selwyn, 2004) and may need repeat applications. Treated specimens therefore require periodic visual monitoring. The advantage of tannic acid is that it can be used to treat areas which are actively spalling, but where the flake is still attached, otherwise making akaganéite crystals inaccessible. If the object starts to re-corrode tannic acid can be re-applied easily and without the need to remove the previous treatment layer (Logan and Selwyn, 2007).

The hydrogen reduction technique (Barker *et al.*, 1982) also creates a blackened effect but was rejected as an option because oxidation occurs

extremely rapidly after treatment if the iron is not coated with resin.

Treatment

The specimen was removed from its wooden crate and dry-cleaned using latex-free additive-free polyurethane cosmetic sponge to remove particulate contaminants from the surface. This was followed by ethanol flooding and swabbing to remove rodent urine and mobile chlorides. Techniques were then trialled on small fragments which had previously become detached due to spalling.

Initial trials on spalled fragments

Air-abrasive techniques and steel wool removed the akaganéite from spalled fragments but left a shiny fresh surface behind, which would be sensitive to further corrosion (as exemplified by the corrosion haloes around abraded areas of the meteorite). The glass bristle brush failed to remove the akaganéite. Liquid abrasive polishing compounds were rejected because they would leave chemicals on the porous surface and sodium salts were rejected because they would remove the corrosion products completely, leaving a fresh surface exposed to corrosion. The tannic acid treatment described by Logan et al., (2013) was trialled. This was adapted because the recommended technique was ineffective on the meteorite, presumably due to its higher nickel content or lower porosity than archaeological iron. The treatment solution was found to be more effective with a higher percentage of ethanol (the final addition of 100 ml water in the recipe was replaced with 100 ml ethanol), which acts as a wetting agent and aids penetration. The solution was used at 10% concentration (higher than the recommended dilution), heated to 50°C and applied by local flooding of the surface and agitated with a stiff brush. During heating a watch glass was placed on the beaker to prevent a disproportionate evaporation of the ethanol. SEM analysis showed the presence of phosphor in areas treated with tannic acid, which derives from the phosphoric acid used to adjust the pH and increase the amount of dissolved iron ions available for reaction with tannic acid. Phosphoric acid reacts with iron ions to form ferric phosphate, which also protects the iron (Logan et al., 2013).

Treatment of the specimen

Curatorial staff were consulted following the initial trials and tannic acid was chosen for treatment of the specimen. One coat of tannic acid was applied to the entire surface of the specimen using a stiff brush and then allowed to dry, to stabilise the thin layer of oxidation covering 30% of the surface. The spalled craters were then treated with 2 or 3 additional tannic acid treatments, using a fine brush, until the orange akaganéite crystals had turned black.

The spalling areas (where slivers of metal had begun to peel away but were still firmly attached to the main specimen) were treated using a pipette and the solution was introduced to cracks using capillary action. The uncorroded areas of the meteorite were unaffected by the treatment but the areas of corrosion assumed a darker brownblack colour and an increased lustre (Figure 4). This resembled the fusion crust of fresher meteorites, a positive by-product of the stabilisation treatment (although care must be taken to record all treatments to avoid unethical deception). Two patches assumed a purplish-blue appearance, which was not acceptable to curatorial staff. These areas were treated with a thin film of Renaissance microcrystalline wax polish (a mixture of Cosmolloid 80 hard and BASFA microcrystalline wax), pre-tinted with raw umber and mineral black earth pigments (pers comm. JP Brown 11 September 2017) (Figure 5). The ferric tannate passivation layer was considered by conservation staff to provide a sufficient barrier between the wax and the meteorite in this instance.

An additional specimen, a portion of the Henbury meteorite IIIAB OM 0.9 (first found in 1931, Northern Territory, Australia) was also treated with tannic acid, with similar success (Figure 6).

Results

After 12 months and 18 months respectively, the Henbury meteorite showed no active corrosion but the Canyon Diablo specimen showed fresh corrosion inside one especially deep regmaglypt. This may be because the original tannic acid treatment was not thorough enough in this area, or because vapour pressure is lower at concave meniscus so water can be trapped (Pascoe, 1982), or due to solubilisation of oxychlorides over time (which can occur at high RH) making them available for reactions (Rinny and Schweizer, 1982). The most likely explanation is, however, that the RH in the temporary storage environment went up to 72% for a short period of time, and over 60% RH for extended periods, far above recommended levels for objects treated with tannic acid. This regmaglypt was treated locally with tannic acid as recommended by Pelikán (1966) and Logan and Selwyn (2007) and no visible active corrosion has recurred after a further 12 months in storage.



Figure 4. The specimen (a) before treatment; (b) after treatment with tannic acid.



Figure 5. (a) area on one face of the treated specimen, showing a bluish lustre (right-hand side of the image); (b) the same area after application of the tinted wax.



Figure 6. Left: Henbury meteorite before treatment. Right: after treatment.

Conclusion

A suitable treatment must be chosen based on the research value held by a specimen and how analysis would be undertaken. In the case of this Canyon Diablo meteorite, preserving the core and improving overall appearance for exhibition was chosen at expense of preserving the crust chemistry. The crust had already become heavily contaminated and corroded through years of inappropriate storage and handling. Any areas of corrosion are in themselves already altered and likely to contain a suite of different minerals caused by oxidation, hydration, dehydration, acidic and alkaline regions, alternating structural layers, migration of ions (Tamura, 2008) and pollution. There are ethical concerns within the field of conservation regarding the removal of corrosion layers, since they are composed of original (although altered) material from the object. Curators and conservator were in agreement in this case, however, that the patches of active corrosion are detrimental to the stability and visual authenticity of the meteorite, and therefore should be removed or chemically stabilised. The conversion of iron corrosion to stable iron compounds such as magnetite is a widely accepted practice in the conservation of archaeological iron (Argyropoulos et al., 2017). Iron meteorites which have suffered years of varied contamination and deterioration, due to improper storage conditions, may be stabilised using tannic acid, as long as conservation and curation staff are in agreement. If undesirable blue tints are created, these may be masked using tinted microcrystalline wax.

Chemical treatment should, however, never replace environmental control as a method of preservation and non-interventive options such as 3D surface scanning should be considered to capture physical properties in a digital format. Preventive conservation methods should be used to preserve the specimen in the long term. This type of meteorite should normally be stored below 35% relative humidity, and ideally below 12% (Watkinson and Lewis, 2004), but the tannic acid treatment should allow storage up to 50% RH. The specimen should be monitored regularly for further evidence of crystal growth and spalling.

Further work

The treated meteorite must be displayed and stored in a stable relative humidity up to 50%. Its condition must be monitored at regular intervals and any deterioration compared with the posttreatment images and the condition maps, to identify the cause of any continued oxidation. The weeping areas were not porous so it is uncertain whether the tannic acid treatment will be effective in the long term. If continued oxidation is observed in these areas, a gel (perhaps thickened with Laponite RD containing sodium salts (Waller, 1980) or a poly(vinyl) acetate borate gel with chelators (Duncan *et al.* 2017)), will be trialled to remove the oxidised iron and then the exposed surface will be treated again with tannic acid.

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