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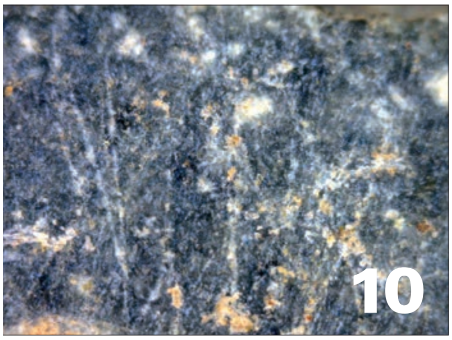

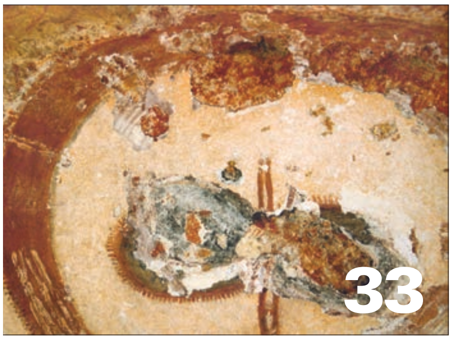

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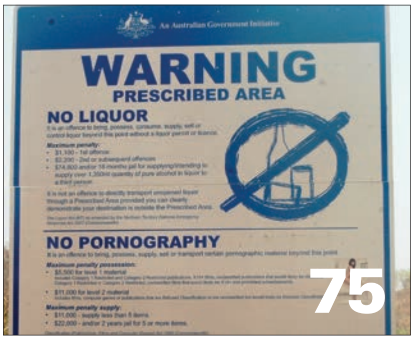

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The first Australian Synchrotron powder diffraction analysis of pigment from a Wandjina motif in the Kimberley, Western Australia

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Abstract

We report the identification of minerals in stratified paint layers from a Wandjina motif in the central Kimberley region, Western Australia, via synchrotron powder diffraction. Interpreting our findings with reference to previous pigment characterisations of Wandjina motifs, we outline the potential of this method for rock art investigations. We particularly highlight the implications of successful major and minor phase identification in very small (~3 μg) pigment samples. The results of this pilot study show that crystallographic data is critical in helping to separate environmental/cultural signatures from post-depositional processes within anthropogenically applied pigments. In Wandjina rock art, crystallography facilitates the examination of the cultural context of rock art production within an assemblage ethnographically known to have undergone regular, ritual repainting.

Introduction

The characterisation of crystal structure—the arrangement of atoms in inorganic materials (Jercher et al. 1998:385)—is a critical part of holistic investigations of mineral pigments used for rock art production. This was recognised by conservation scientists during the very early stages of the Australian Synchrotron project (Creagh et al. 2007; O'Neill et al. 2004) and, internationally, the vastly reduced sampling requirements of synchrotron powder diffraction have been noted as facilitating the examination of culturally significant materials (Hradilová and Žižak 2011). Mineralogical data can provide vital information for the archaeological study of rock art by describing the physical properties, and likely geomorphic procurement contexts, of pigments (Rapp and Hill 2006:196; Švarcová et al. 2011). In addition, crystallography of rock art pigments has been used to differentiate and define post-depositional processes (Ford et al. 1994) and as a proxy record for palaeoclimatic conditions (Goodall et al. 2009). This article reports the results of a pilot investigation of stratified paints from a Wandjina[1] motif in Ngarinyin Country in the central Kimberley using synchrotron powder diffraction. Our results demonstrate that major and minor phase identification of discrete painting episodes through time is possible in a rock art tradition ethnographically known to have undergone regular ritual repainting (Blundell 1974; Crawford 1968).

Synchrotron Radiation

The application of high resolution synchrotron radiation heralds a significant breakthrough in terms of the opportunity to gain more refined structural data by overcoming the limitations of previous laboratory-based powder x-ray diffraction (XRD) analyses. Conventional XRD analysis has been fundamentally constrained in rock art

1 We have retained the spelling as reported in the academic literature at the time when the sample analysed was collected (ca 1996). We intend no offence by retaining this historical spelling, and acknowledge that the present nomenclature of Aboriginal traditional owners may vary.

research applications by initial sample sizes in the order of several grams (Crawford and Clarke 1976; Ford et al. 1994; Ward et al. 2001; Watchman et al. 1997) compared to the synchrotron powder diffraction beamline, which typically requires a sample in the order of ~3 mg. In addition, data collection times are greatly reduced at a synchrotron, in the order of 5 minutes per sample, compared to many hours for a conventional laboratory XRD machine. The exceptional high resolution of the synchrotron also allows for easy differentiation between phases compared to the peak overlap often experienced in conventional XRD spectra.

Mineral pigments, often termed 'earth' pigments, are generally coarsely described in relation to their physical structure as ochres and/or clays. The crystallography of mineral pigments, specifically minor phases, is important, as there is significant diversity in formation processes and, therefore, the geomorphic contexts in which deposits are found (Hradil 2012:86). In Aboriginal Australia, where landscape is so innately a part of culture, knowing the geographic origin of minerals offers researchers insights into past cultural landscapes (Head 1993; McBryde 1997), as well as continuing cultural traditions (Clarke 1976; Crawford and Clarke 1976; Mosby 1993; O'Connor et al. 2008; Randolph and Clarke 1987). The low backgrounds and orders of magnitude increase in signal-to-noise ratios available at synchrotron beamlines make this technique sensitive to small fractions (<1%) of minor mineral phases, which may be crucial to determining the geomorphic context of a particular pigment.

Previous laboratory XRD analyses of Wandjina rock art motifs have produced composite diffraction spectra derived from homogenised powder analytes rather that discrete, stratified painting episodes (Figure 1) (Crawford 1968, 1977; Crawford and Clarke 1976; Ward et al. 2001). In contrast, synchrotron powder diffraction offers opportunities to collect high resolution data sets with small sample requirements, facilitating the examination of discrete stratified paint (and accretion) layers. An examination of the mineralogy of the stratified layers observed in exfoliated rock art paint flakes has significant implications for geochronological, archaeological and material science (conservation) investigations and may yield data relating to:

- The highly complex geomorphic environment of rockshelters in subtropical (palaeo) climates (Bowdler 2005; Huntley et al. in press; MacLeod and Haydock 2008; MacLeod et al. 1997; Wyrwoll et al. 2012);
- The cultural context of rock art production via changes in paint sources and recipes through time (Ford et al. 1994; Huntley et al. in press; Thomas 1998); and,
- The relationship between stratified paint layers and observed post-depositional mineral phase transitions (Ford et al. 1994).

Foundation studies into the physical properties and durability of distinctive huntite pigments from Wandjina rock art have shown that the mineral is highly alkaline, resulting in chemical reactions with the rock substrate and other mineral pigments (Clarke 1977:61). In addition, the small (1–2 µm), uniform particle size of huntite means it is easily dispersed in water, allowing thick suspensions of the pigment to be applied as poorly coherent layers of paint. The porosity of these layers has been observed to create a network of capillaries within pigment layers that can draw water into the rock art with enough force to cause disruption of paintings (Clarke 1977:61). These attributes, combined

Figure 1 Wandjina site at the King Edward River crossing showing flake exfoliation typical of that described in the text (photograph by Mike Donaldson, reproduced from Donaldson and Kenneally 2007:112 with permission).

with differential movement of mineral paint layers with different properties (such as less hydrous, acidic to neutral iron oxide, clay and/or mica), over a variety of changing microclimatic conditions, have been posited as the cause of interstrata failures, or flaking, in Wandjina rock art (Clarke 1977:61). In our opinion, the points of greatest weakness, and the microstrata therefore most susceptible to interlayer failures, would be those between discrete painting episodes. Discrete painting episodes of Wandjina rock art have been observed as often starting with a layer of white paint that obscures the underlying motif (Crawford 1968; Randolph and Clarke 1987), though this is not always the case (O'Connor et al. 2008). Where motifs, or entire panel compositions, are superimposed over older existing rock art it follows that stronger cohesion, with some admixture or bleeding of coevally applied paints, will be created between damp paint layers of the freshly executed painting, when compared to the pre-existing, weathered rock art underlying it. The strong adhesion of a small number of paint layers observed in the two subsamples analysed here is therefore thought to represent discrete, stratified painting episodes.

The Sample

The analysed flake of exfoliated stratified paint was collected from the eyes of a Wandjina motif in a rockshelter in the central Kimberley. This specimen was one of a number of samples collected during a chronologically focused research programme in the mid-1990s (Morwood et al. 1994, 2010; Roberts et al. 1997; Watchman et al. 1997). Through direct ^{14}C dating, the production of Wandjina motifs has been established as having begun at least 4000 years ago (2457–2033 BCE), with evidence for 'classic' stratified Wandjina motif production appearing more recently, from 1634 CE to the present (Morwood et al. 2010:5).

Two very small 'flecks' of pigment, each >1 mm in greatest dimension (Figure 2), were acquired from the aluminium foil in which the sample had been wrapped since its original collection. The paint layers within the flecks were probed with a scalpel under magnification and could not be further separated. The flecks contained an estimated 2–4 layers of pigment, though the precise stratification was difficult to establish even under high magnification, owing to the adhesion and admixture of pigment layers. As we have argued above, we believe each pigment fleck represents a

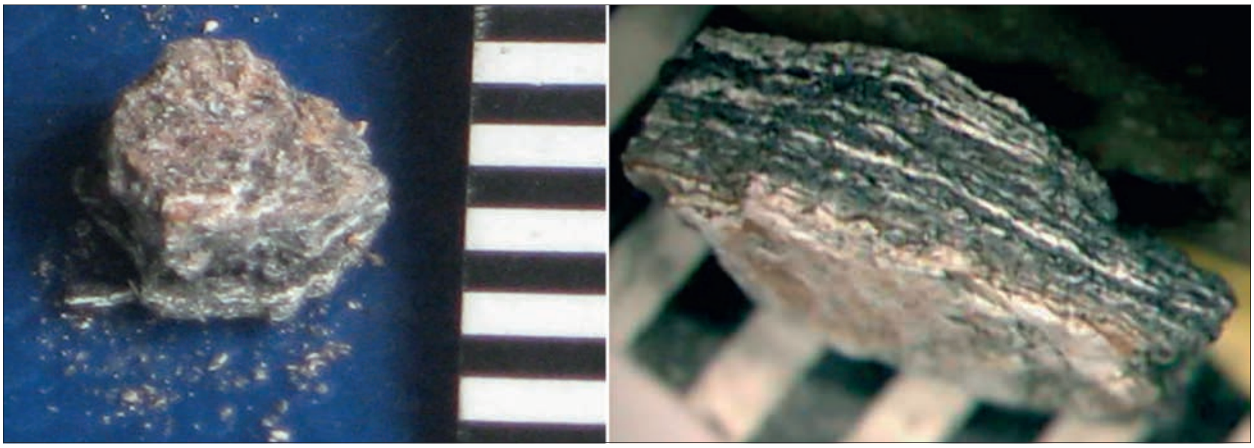

Figure 2 Wandjina paint flake analysed in the pilot. Left: overview; Right: profile. The small detached flecks in the left image include the subsamples analysed. Note the scale is in mm increments.

discrete, stratified painting episode on the rock art panel. The exact stratigraphic context of the subsamples could not be established as the flecks were already separated from the main paint flake. Rather, the examination of minerals contained within the discrete painting episodes was undertaken as a proof of concept, designed to inform subsequent archaeometric analyses of Wandjina rock art.

Each fleck was designated with a subsample number. Subsample 1 was a black fleck that, on closer inspection, had at least one, possibly more, layers of white pigment adhered to one side (either under- or overlying the black pigment) that, when ground during sample preparation, produced a dark grey hue. Subsample 2 had an overall pink hue containing a layer of red paint and at least one, possibly two, layers of white pigment (one possibly overlying and one underlying the red paint). When ground during sample preparation, Subsample 2 produced a light grey hue, indicating that a layer of black pigment may also have been incorporated into the fleck, though this was not visible prior to grinding.

Method

Each subsample was ground to a fine powder in a mortar and pestle, loaded into a 0.3 mm diameter borosilicate capillary and mounted onto the diffractometer at the Australian Synchrotron powder diffraction beamline (Wallwork et al. 2007). The samples were positioned in the diffractometer centre and spun at ~1 Hz during data collection. Datasets were collected at a refined wavelength of 0.95356 Å, from 5–85° 2Theta, using the MYTHEN microstrip detector (Bergamaschi et al. 2010), a position sensitive detector which allows for the collection of 80° of 2Theta simultaneously with a step size of 0.002°. Data were collected for 5 minutes per detector position, a total of 10 minutes per sample.

Phase identification was undertaken using Panalytical's Highscore Plus equipped with the ICDD PDF4 database (Fawcett et al. 2009). Owing to the high clay and amorphous content of the subsamples, full quantification was not possible; however, indicative relative amounts are reported for each crystalline constituent to serve as semi-quantitative indices of the composition of these multi-phase pigments.

Results

The minerals identified and their approximate weight percentages are reported in Table 1. Approximate weight percentages are useful for understanding the relative composition of the rock art pigment and prove that it is possible to obtain high resolution, semi-quantitative mineral datasets using very small samples. This proof of concept experiment demonstrates that mineralogical identification of discrete, stratified Wandjina pigment applications (painting episodes) can be readily achieved using synchrotron powder diffraction.

Comparison with Previous Kimberley Pigment Studies and Preliminary Interpretations

The mineral identifications reported here are consistent with the findings of previous conventional XRD investigations of rock art and white pigment source locations in the Kimberley. That is, they are mixed minerals dominated by huntite and clay (kaolinite) (Clarke 1976 1977; Ford et al. 1994; Ward et al. 2001; Watchman 1997). Previously, Thomas (1998) performed XRD analyses on two geographically discrete white pigment 'quarries', demonstrating that multiple mineral phases were present in 'raw' white pigment sources. The quantification of composite mineral constituents within stratified paint layers may therefore provide information regarding changes in pigment sources over time (as suggested by Thomas 1998).

Mineralogical substitutions have important environmental, chronological and conservation implications (Clarke 1976, 1977; Ford et al. 1994; Goodall et al. 2009). Based on XRD spectra from amalgamated stratifications within Wandjina paint flakes (i.e. combining several painting episodes), Ford et al. (1994) suggested that post-depositional mineral alterations were occurring in rock art panels in situ. Our analysis demonstrates that the semiquantitative composition of white pigments is achievable with very small samples and therefore within discrete layers of stratified paint thought to represent individual painting episodes. Our identification of whewellite and dolomite in the two minute subsamples analysed in this pilot study shows that the minerals indicative of a proposed post-depositional transition of calcite rock art paints to calcium oxalate minerals described by Ford et al. (1994) will be visible using synchrotron powder diffraction (if occurring).

The proportion of different minerals within mixed phases may have significant applications regarding rock art conservation (the treatment of paint layers with different properties). As outlined previously, Clarke (1977:61) concluded that the properties of huntite (alkalinity, small grain size and

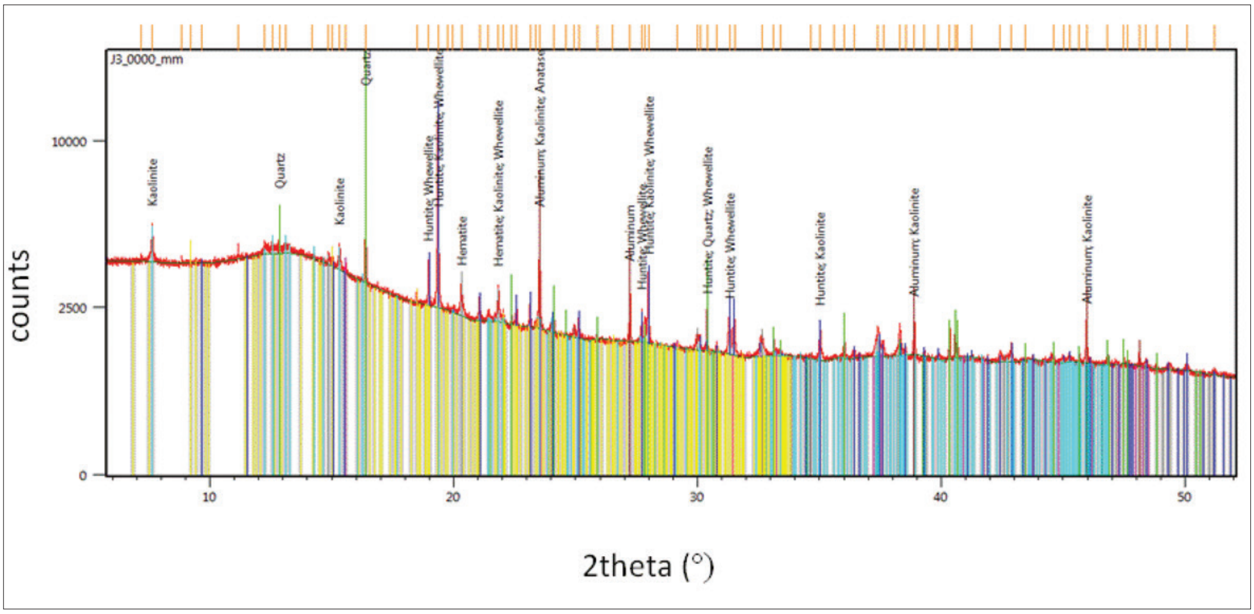

Figure 3a Synchrotron powder diffraction phase diagram for Subsample 1.

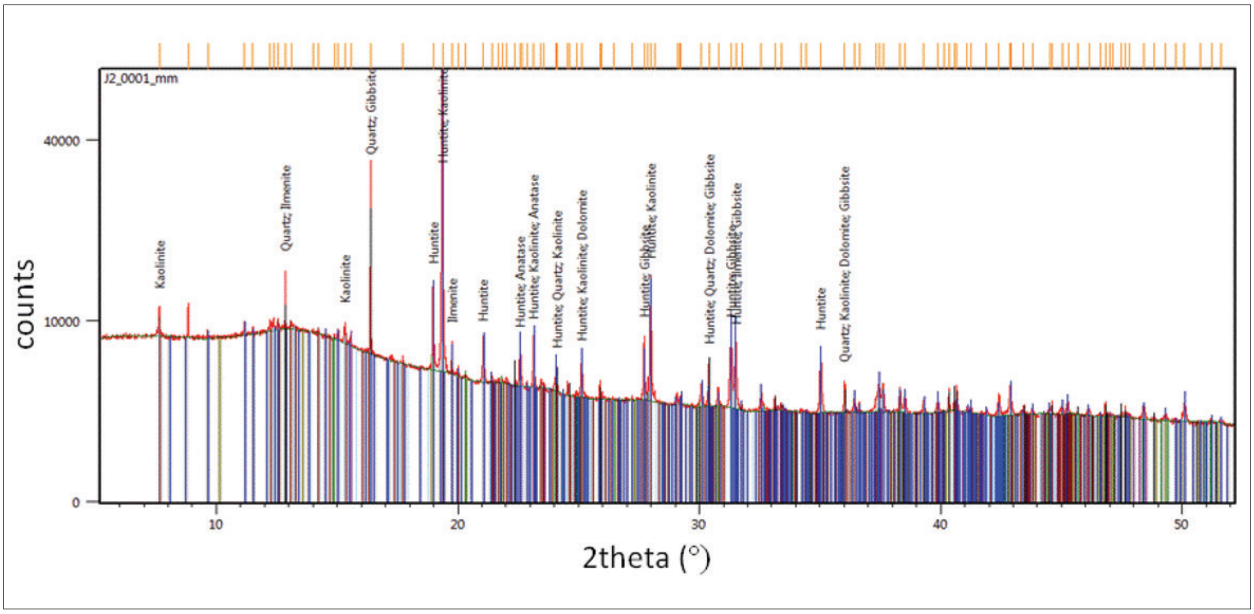

Figure 3b Synchrotron powder diffraction phase diagram for Subsample 2.

porosity) made this mineral inherently unstable. Our findings are consistent with this; however, we were able to define with much higher resolution the composite mineral properties of dominantly huntite, mixed mineral rock art pigment. Our identification of minor phases within discrete painting episodes may further explain the propensity to deterioration in Wandjina art noted by Clarke (1977).

Other phase identifications reported are also consistent with previous work (though much more precise). For instance, the major haematite phase (22%) present in Subsample 2 is consistent with the findings of Ford et al. (1994) and is thought to be cultural, associated with the red pigment present within the stratified Wandjina paint layers analysed (Figure 2). In contrast, the minor haematite (1%) and gibbsite (2%) in Subsample 1 are common phases in lateritic environments with high rainfall, and it is therefore not surprising that they would be present as precipitates in a central Kimberley rockshelter environment. Though aluminium phosphate has been described as a constituent in white rock art paint in the Kimberley (Watchman 1997:50–51), the aluminium reported in Subsample 2 (21%) is pure and therefore thought to derive from the visibly perishing aluminium foil in which the sample was stored post-1996.

Implications for Ongoing and Future Research

The results of this pilot project demonstrate that mineralogical identifications can be achieved on minute rock art pigment samples (3 µg), and therefore within discrete painting episodes, using synchrotron powder diffraction. High resolution, semi-quantitative, mineral data will be invaluable for exploring changes through time in stratified, laminar rock art paint samples. This is the first time stratified minerals have been examined from repainted Wandjina motifs and further research of this type will provide new insights into the environmental and cultural context of rock art production in the Kimberley. High resolution mineralogical data will

Subsample	Reference Code	Compound	Formula	Relative Amount of Crystalline Material (%)
Subsample 1	96-900-0985	Huntite	$CaMg_3(CO_3)_4$	69
	96-901-2601	Quartz	SiO_2	17
	96-900-9235	Kaolinite*	$Al_2Si_2O_5(OH)_4$	6
	96-101-1241	Haematite	Fe_2O_3	1
	96-900-3525	Dolomite	$CaMg(CO_3)_2$	2
	96-900-0911	Ilmenite	Fe_2TiO_3	3
	96-720-6076	Anatase	TiO_2	1
	96-900-3875	Gibbsite*	$Al(OH)_3$	2
Subsample 2	96-900-0985	Huntite	$CaMg_3(CO_3)_4$	43
	96-901-2601	Quartz	SiO_2	34
	96-900-0140	Haematite	Fe_2O_3	22
	96-900-8461	Aluminium~	Al	21
	96-900-9231	Kaolinite	$Al_2Si_2O_5(OH)_4$	15
	96-900-9087	Anatase	TiO_2	6
	96-900-0764	Whewellite	$Ca(C_2O_4)(H_2O)$	8

Table 1 Mineral phase identifications for Subsamples 1 and 2. * = No clay preparations (such as settling) were undertaken; ~ = Likely to be derived from the (visibly perishing) aluminium foil in which the sample was stored.

be critical in helping to separate environmental/cultural signatures from post-depositional processes within samples, thereby facilitating an examination of the cultural context of rock art production by observing change or continuity in the choice of pigments by artists over time, preserved within layers of paintings that are ethnographically known to have undergone regular, ritual repainting (Blundell 1974; Crawford 1968). The resolution achievable using synchrotron powder diffraction has also facilitated significantly greater sensitivity of minor phase identification, which will be instrumental in determining the geomorphic context of mineral pigment sources (Hradilová and Žižak 2011:334-5).

Acknowledgements

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