

# Primary red bed magnetization revealed by fluvial intraclasts

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## <sup>1</sup> ABSTRACT

The magnetization of hematite-bearing sedimentary rocks provides critical records of geomagnetic reversals and paleogeography. However, the timing of hematite remanent magnetization acquisition is typically difficult to constrain and has led to much controversy in the interpretation of such data. This so-called “red bed controversy” stems from the reality that while detrital hematite in sediment can lead to a primary depositional remanent magnetization, alteration of minerals through interaction with oxygen can lead to the post-depositional formation of hematite. Growth of hematite crystals within sediments could occur in a geologically short time period immediately following deposition or could occur thousands to millions of years later due to the passage of oxygenated fluids following burial. Given that many paleomagnetic field tests such as the reversal test and fold test could still “pass” in scenarios with secondary post-depositional hematite growth, this problem has been particularly intractable in many sedimentary successions. In this study, we use an exceptionally well-preserved fluvial sediments within the 1.1 billion-year-old Freda Formation to gain insight into the timing of hematite remanence acquisition. This deposit contains siltstone intraclasts that were eroded from a coexisting lithofacies and redeposited within channel sandstone. Thermal demagnetization and petrographic data from these clasts reveal that they contain two generations of hematite. One population of hematite demagnetized at the highest unblocking temperatures and records directions that

19 rotated along with the rip-up clasts. This component is a primary detrital remanent  
20 magnetization that formed prior to the reorientation of the clasts within the river. The other  
21 component is removed at lower unblocking temperatures and has a consistent direction  
22 throughout the intraclasts. This component is held by a population of finer-grained hematite that  
23 grew and acquired a chemical remanent magnetization following deposition. The data support the  
24 interpretation that the magnetization of hematite-bearing sedimentary rocks held by >400 nm  
25 grains is more likely to record magnetization from the time of deposition and can be successfully  
26 isolated from co-occurring authigenic hematite.

## 27 INTRODUCTION

28 The magnetizations of hematite-bearing sedimentary rocks known as “red beds” have provided  
29 ample opportunities for Earth scientists to gain insight into the ancient geomagnetic field and the  
30 paleogeographic positions of sedimentary basins. However, with these opportunities has come  
31 much scientific debate, leading to what has been referred to as the “red bed controversy” (Butler,  
32 1992; Beck et al., 2003; Van Der Voo and Torsvik, 2012). This controversy stems from the reality  
33 that hematite within sedimentary rocks can have two sources: 1) detrital grains that are within  
34 the sediment at the time of deposition; 2) grains that grow *in situ* after the sediments have been  
35 deposited.

36 How does one constrain the relative age of hematite within sedimentary rocks? Many of the  
37 traditional paleomagnetic field tests are unable to differentiate between primary versus diagenetic  
38 remanence. For example, a structural fold test can constrain that a remanence direction was  
39 obtained prior to folding, but millions of years have typically passed between the deposition of a  
40 sediment, its burial in a sedimentary basin, and such tectonic tilting. Dual polarity directions  
41 through a sedimentary succession are commonly interpreted as providing assurance that the  
42 remanence records primary or near-primary magnetization; however, hematite growth could occur  
43 significantly after deposition during a period when the geomagnetic field was reversing.

44 Petrographic investigations are valuable, but it can be difficult to ascertain how much the  
45 petrographically observed hematite contributes to the magnetization and to unambiguously  
46 interpret whether observed grains are detrital or not (e.g. Elmore and Van der Voo, 1982). A  
47 common approach to classify hematite grains within red beds is into a fine-grained pigmentary  
48 population, typically interpreted to have formed within the sediment, and a coarser-grained  
49 population that has been referred to in the literature as “specularite” (Butler, 1992; Van Der Voo  
50 and Torsvik, 2012). Tauxe et al. (1980) showed that sediments with abundant red pigmentary  
51 hematite in the Miocene Siwalik Group had lower thermal unblocking temperatures than grey  
52 samples dominated by a coarser-grained phase of specular hematite. Observations such as these  
53 have led to the practice of defining the characteristic remanent magnetization from  
54 hematite-bearing sediments as that held by the highest unblocking temperatures (Van Der Voo  
55 and Torsvik, 2012). The primary versus secondary nature of micron-scale “specularite” grains  
56 that likely carry this remanence has been one of the largest sources of contention in the “red bed  
57 controversy” (Van Houten, 1968; Tauxe et al., 1980; Butler, 1992; Van Der Voo and Torsvik,  
58 2012).

59 What is needed to address the timing of remanence acquisition is a process that reorients the  
60 sediment before it has been lithified. Two such processes that can occur within a siliciclastic  
61 depositional environment and be preserved in the rock record are: 1) syn-sedimentary slumping  
62 wherein coherent sediment is reoriented through soft-sediment folding in the surface environment  
63 and 2) intraclasts comprised of the lithology of interest that have been redeposited within the  
64 depositional environment. Sediments that have undergone reorienting sedimentary processes can  
65 provide significant insight into whether magnetization was acquired before or after reorientation.

66 Tauxe et al. (1980) studied 7 cobble-sized clasts within the Siwalik Group that were  
67 interpreted to have formed by cut-bank collapse and discovered that their magnetic remanence  
68 was acquired prior to clast reorientation. An investigation by Purucker et al. (1980) on red beds  
69 of the Triassic Moenkopi Formation of Arizona used multiple such processes to gain insight into

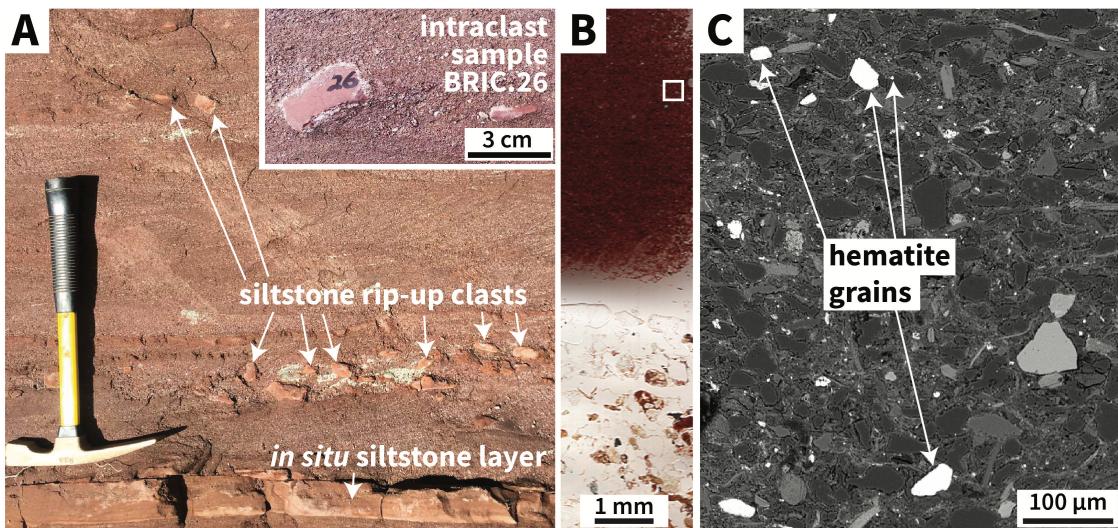
70 hematite acquisition. In their study, an intraformational landslide deposit with isoclinal folds of  
71 hematite-bearing claystone revealed non-uniform directions upon blanket demagnetization to  
72 650°C that cluster better when corrected for their tilt leading to a primary interpretation. Scatter  
73 was also observed in intraformational conglomerate clasts weathered out of an underlying unit  
74 upon blanket thermal demagnetization to 630°C. However, the lack of principal component  
75 analysis makes it difficult to evaluate the coherency of the directions. Complicating matters,  
76 Larson and Walker (1982) analyzed shale rip-up clasts in the same Moenkopi Formation and used  
77 the fact that similar remanence directions were removed between clasts during thermal  
78 demagnetization up to 645°C as support for the hypothesis that red beds rarely reflect the  
79 geomagnetic field at the time of deposition. Evaluating the robustness of this result is hindered by  
80 the cessation of thermal demagnetization before the Néel temperature of hematite and the lack of  
81 principal component analysis. These limitations are found in many studies from this era of  
82 research, when the red bed controversy was particularly fervent, as the work predates the  
83 widespread application of principal component analysis in conjunction with systematic  
84 progressive thermal demagnetization (Kirschvink, 1980; Van Der Voo and Torsvik, 2012).

85 In this study, we investigate cm-scale siltstone intraclasts within the Freda Formation that  
86 were eroded by fluvial processes and redeposited amongst cross-stratified sandstones (Fig. 1).  
87 High-resolution thermal demagnetization data on these clasts constrain the timing of hematite  
88 acquisition by revealing a primary component that formed prior to the erosion of the clasts within  
89 the depositional environment and a secondary component that formed following their redeposition.

## 90 GEOLOGICAL SETTING

91 The ~4 km thick Freda Formation was deposited in the Midcontinent Rift as it was thermally  
92 subsiding following the cessation of widespread magmatic activity (Cannon and Hinze, 1992). The  
93 fluvial sediments of the Freda Formation are part of the Oronto Group and were deposited  
94 following the deposition of the alluvial Copper Harbor Conglomerate and the lacustrine Nonesuch

95 Formation (Ojakangas et al., 2001). A maximum age constraint on the Freda Formation of  
 96  $1085.57 \pm 0.25/1.3$  Ma ( $2\sigma$  analytical/analytical+tracer+decay constant uncertainty; Fairchild  
 97 et al., 2017) is provided by an U-Pb date of a lava flow within the underlying Copper Harbor  
 98 Conglomerate. Abundant fine-grained red siltstones within the formation have a well-behaved  
 99 magnetic remanence dominated by hematite (Henry et al., 1977).



**Figure 1.** A: Siltstone intraclasts within the Freda Formation. The field photo shows an intact layer of siltstone below the hammer head which is topped by a bed of trough cross-stratified coarse sandstone with horizons of siltstone intraclasts. The hammer is 40 cm long. The inset photo is of an individual intraclast that was sampled as BRIC.26. B: A scan of a thin section of the BRIC.26 intraclast (upper half of image) and the coarse sand matrix (lower half of image). The red color of the intraclast is due to pigmentary hematite. C: Backscatter electron image of the siltstone clast from the region of the white box in B. The light-colored detrital grains (light due to iron's high atomic number) labeled with arrows were confirmed to be hematite through electron backscatter diffraction<sup>1</sup>.

100 The studied outcrop is located along the Bad River (northern Wisconsin) in the lower portion  
 101 of the Freda Formation – approximately 400 meters above its conformable base with the  
 102 Nonesuch Formation.<sup>1</sup> The two main lithofacies in the studied outcrop are: (1) siltstone to very  
 103 fine sandstone with planar lamination and horizons of ripple cross-stratification and (2) coarse to  
 104 very coarse subarkosic sandstone with dune-scale trough cross-stratification (Fig. 1). These  
 105 lithofacies are consistent with a fluvial depositional environment where the coarse sandstone facies

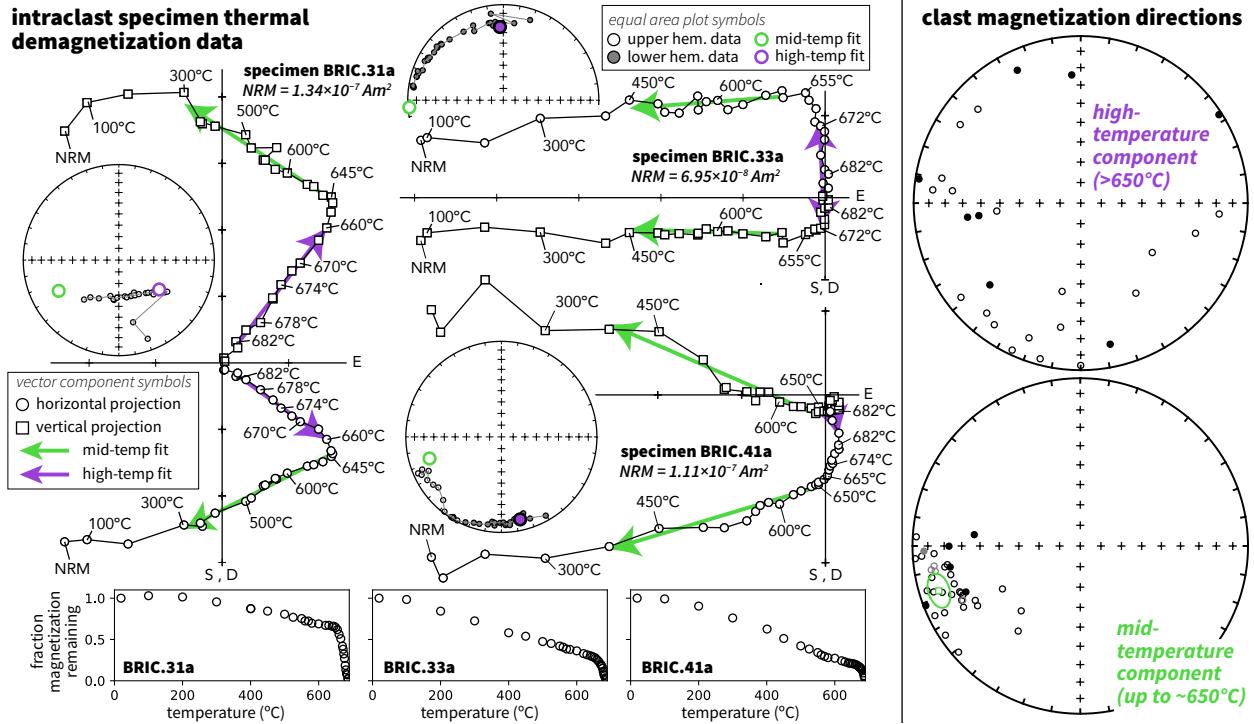
<sup>1</sup>GSA Data Repository item 2018XXX, is available online at [www.geosociety.org/pubs/ft2018.htm](http://www.geosociety.org/pubs/ft2018.htm), or on request from [editing@geosociety.org](mailto:editing@geosociety.org) or Documents Secretary, GSA, P.O. Box 9140, Boulder, CO 80301, USA.

106 are channel deposits and the siltstones are inner-bank or over-bank deposits. The coarse-grained  
107 sandstone contains horizons of tabular cm-scale intraclasts comprised of the dark red siltstone  
108 lithology that is present in underlying beds of intact siltstone (Fig. 1). These tabular clayey-silt  
109 intraclasts were eroded within the depositional environment and redeposited in the sandstone.  
110 Due to migrating channels in fluvial systems, it is expected for a river to erode its own sediments.  
111 The intraclasts would have been held together through cohesion resulting from the clay  
112 component within the sediment. Given that the clasts are large (1 to 7 cm) relative to their host  
113 sediment, and that they would have been fragile at the time of deposition, it is unlikely that they  
114 were transported far.

## 115 METHODS and RESULTS

116 Oriented samples were collected and analyzed from 39 Freda Formation intraclasts. The  
117 dimensions of the sampled clasts range from 2.2 x 1.4 x 0.5 cm to 7.2 x 2.3 x 1.2 cm. Given that  
118 the clasts were typically smaller than the 1-inch-diameter drill cores used for sampling, they were  
119 collected along with their sandstone matrix. These oriented cores were mounted onto quartz glass  
120 discs with Omega CC cement and the matrix material was micro-drilled away. The mounted  
121 clasts underwent stepwise thermal demagnetization in the UC Berkeley Paleomagnetism Lab  
122 using an ASC demagnetizer (residual fields <10 nT) with measurements made on a 2G  
123 DC-SQUID magnetometer. The demagnetization protocol had high resolution (5°C to 2°C to  
124 1°C) approaching the Neél temperature of hematite resulting in 30 total thermal demagnetization  
125 steps (Fig. 2). All paleomagnetic data are available to the measurement level in the MagIC  
126 database (<https://earthref.org/MagIC/doi/>). *So that reviewers have access to the data, they are*  
127 *currently available in CIT lab format and MagIC format here:*  
128 [https://github.com/Swanson-Hysell-Group/2018\\_Red\\_Bed\\_Intraclasts](https://github.com/Swanson-Hysell-Group/2018_Red_Bed_Intraclasts).

129 The clasts typically reveal two distinct magnetization directions. One direction was similar  
130 throughout the intraclasts and was typically removed between 200°C and 650°C (Fig. 2). This



**Figure 2.** Paleomagnetic data from intraclasts reveal a mid-temperature component that typically unblocks prior to 655°C and a high-temperature component that typically unblocks between 655°C and 687°C. These components are present as varying fractions of the overall remanence as seen in the three individual clasts for which data are shown on vector component plots and measurement-level equal area plots in tilt-corrected coordinates (developed using PmagPy; Tauxe et al., 2016). The direction of the mid-temperature component is shown as purple arrows on the vector component plots and purple circles on the equal area plots while the high-temperature component is shown with green symbols. The mid-temperature component has a similar direction throughout the clasts as can be seen on the component directions equal area plots (mean declination: 252.4, inclination: -12.5,  $\alpha_{95}$ : 6.6). In contrast, the high-temperature component directions are dispersed.

131 mid-temperature component is continuously unblocked between these temperatures with no or  
 132 minimal downward inflection at  $\sim 580^\circ\text{C}$  that would indicate remanence associated with magnetite  
 133 (Fig. 2). This component is directionally well-grouped indicating that it was acquired following  
 134 deposition of the clasts (Fig. 2). The other component trends towards the origin and is removed  
 135 by thermal demagnetization steps at the highest levels such that it typically can be fit by a  
 136 least-square line between 665°C and 688°C. The relative magnitude of the components varies  
 137 between intraclasts (Fig. 2). While the high-temperature component can sometimes be fit as a  
 138 line with a lower temperature bound of 660°C (BRIC.31a in Fig. 2), due to overlapping

139 unblocking temperatures between the mid-temperature and high-temperature components the  
140 lower bounds of the high-temperature fits sometimes need to be as high as 680°C (BRIC.41a in  
141 Fig. 2). Note that while the Neél temperature of hematite is sometimes given as 675°C in the  
142 paleomagnetic literature, experimental data often show the Neél temperature to be as high as  
143 690°C (?). There is typically a significant directional change in the specimen magnetization  
144 between the mid-temperature component and the high-temperature component (Fig. 2). As a  
145 result, 29 of the 39 analyzed intraclast specimens could be fit with distinct mid-temperature and  
146 high-temperature least-squares lines. An additional 5 specimens were undergoing directional  
147 change through the highest thermal demagnetization steps indicative of the presence of a distinct  
148 high-temperature component, but this component was not well-expressed enough to be fit. 5 of  
149 the specimens showed no directional change and could be fit with a single mid-high-temperature  
150 component that is grouped with the mid-temperature component. In contrast to the well-grouped  
151 mid-temperature component, the high-temperature component directions are dispersed,  
152 indicating that the component was acquired prior to erosion and redeposition of the clasts. The  
153 high-temperature component directions are more dispersed in declination than inclination leading  
154 to a distribution that is not randomly dispersed on a sphere. Given that the clasts are tabular  
155 and were liberated along their depositional lamination and subsequently landed roughly  
156 bedding-parallel, it is to be expected that the rotations were largely around a vertical axis which  
157 would preferentially change declination.

158 Petrography on the intraclasts reveals two distinct populations of hematite (Fig. 1). One  
159 population is fine-grained pigmentary hematite present dominantly within the clay-sized matrix  
160 and rimming detrital silt-sized grains. The zones of pigmentary hematite within the matrix  
161 remain cloudy to high magnification indicating that the grains are submicron in size. The other  
162 population of hematite has similar sizes and shapes to other detrital silt-sized grains – typically  
163 ranging from 2 to 50  $\mu\text{m}$  in diameter. These hematite grains were identified through reflected  
164 light microscopy with their mineralogy supported by energy-dispersive x-ray spectroscopy (EDS)  
165 and confirmed by electron backscatter diffraction (EBSD).

<sup>166</sup> **DISCUSSION**

<sup>167</sup> Single-domain hematite grains have high coercivities ( $>150$  mT; Özdemir and Dunlop, 2014) and  
<sup>168</sup> high unblocking temperatures. As a result, populations of hematite within rocks are stable on  
<sup>169</sup> long timescales, resistant to overprinting, and therefore attractive for paleomagnetic study. In  
<sup>170</sup> contrast to magnetite, hematite grains retain stable single-domain behavior in crystals  $>1\mu\text{m}$   
<sup>171</sup> with the threshold to multidomain behavior occurring when grain diameters exceed  $\sim 100\mu\text{m}$   
<sup>172</sup> (Kletetschka and Wasilewski, 2002; Özdemir and Dunlop, 2014). Hematite nanoparticles with  
<sup>173</sup> diameters  $<30$  nm have superparamagnetic behavior wherein thermal fluctuation energy  
<sup>174</sup> overwhelms the ability of the grain to retain a stable magnetization at Earth surface temperatures  
<sup>175</sup> (Özdemir and Dunlop, 2014). Hematite grains become progressively less influenced by thermal  
<sup>176</sup> fluctuations as they reach grain sizes of a few hundred nanometers at which point they are stable  
<sup>177</sup> up to temperatures approaching the Néel temperature of  $\sim 685^\circ\text{C}$  (Swanson-Hysell et al., 2011;  
<sup>178</sup> Özdemir and Dunlop, 2014). As a result, there is a strong relationship between grain volume and  
<sup>179</sup> unblocking temperature that can be utilized to estimate grain size. A hematite population that is  
<sup>180</sup> progressively unblocking at thermal demagnetization steps well below the Néel transition  
<sup>181</sup> temperature, such as the mid-temperature component of the intraclasts, is comprised of grains  
<sup>182</sup> within the  $\sim 30$  to  $\sim 400$  nm size range. This fine-grain size is consistent with the pigmentary  
<sup>183</sup> phase within the intraclasts (Fig. 1).

<sup>184</sup> Given the directional consistency of the mid-temperature component among the intraclasts,  
<sup>185</sup> this component must have dominantly formed as a chemical remanent magnetization after the  
<sup>186</sup> intraclasts were redeposited in the channel. Chemical remanent magnetization acquisition by  
<sup>187</sup> pigmentary hematite would have occurred as hematite grains grew to sizes above the  
<sup>188</sup> superparamagnetic to stable single-domain transition resulting in the wide range of unblocking  
<sup>189</sup> temperatures that is observed. In contrast, given its sharp unblocking temperature close to the  
<sup>190</sup> Néel temperature, the high-temperature component is dominantly held by hematite grains that  
<sup>191</sup> are  $>400$  nm such as the silt-sized hematite grains observed petrographically (Fig. 1). The

192 high-temperature remanence component held by these grains was rotated along with the clasts  
193 indicating that it is primary and was acquired prior to the redeposition of the cohesive silt clasts.  
194 That this component is held by larger grains sizes supports it being a detrital remanent  
195 magnetization rather than a chemical remanent magnetization that formed very early prior to  
196 clast erosion.

197 Oxidation of iron in aqueous environments often proceeds through the formation of  
198 fine-grained poorly crystalline ferrihydrite, which transforms to stable crystalline hematite at  
199 neutral pH on geologically short timescales (Cudennec and Lecerf, 2006). The broad unblocking  
200 temperatures we observe for the chemical remanent magnetization in the Freda intraclasts are  
201 similar to those in hematite populations produced through experimental ferrihydrite to hematite  
202 conversion (Jiang et al., 2015). The differential unblocking temperature spectra of the two  
203 components within the Freda intraclasts provides strong support for the argument of Jiang et al.  
204 (2015) that chemical and detrital remanent magnetization can be distinguished; due to detrital  
205 remanence unblocking at the highest temperatures. However, it is also clear from the Freda  
206 intraclast data that while the detrital remanent magnetization can be well-isolated at  
207 temperatures as low as 650°C (specimen BRIC.31a in Fig. 2), the chemical remanent  
208 magnetization thermal unblocking spectra can overlap with that of the detrital remanence and  
209 extend up to temperatures closer to the Néel temperature (specimen BRIC.41a in Fig. 2).  
210 Therefore, to isolate primary remanence in red beds, best practice should be to proceed with very  
211 high resolution thermal demagnetization steps above 600°C, and particularly above 650°C. These  
212 intraclast data reveal that directional change at the highest unblocking temperatures provides an  
213 effective means to discriminate primary and secondary magnetizations within siltstones of the  
214 Freda formation and other red beds. The formation of coarse-grained secondary hematite can  
215 occur, particularly in high permeability lithologies and deeply weathered profiles. However, the  
216 isolation of primary detrital hematite in >1 billion-year-old siltstones lends confidence to  
217 magnetostratigraphic records and paleogeographic interpretations that are based on  
218 interpretations of primary magnetization in ancient red beds.

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