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# International Conference on Rock Magnetism 2017



Fort Hoofddijk laboratory, photo: Ingeborg van Oorschot (http://www.geo.uu.nl/~forth/history/history.htm)

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One hundred geoscientists gathered in Utrecht in July for a week-long, discussion-oriented conference, the latest in the biennial "Santa Fe" series of meetings on rock magnetism and its applications in Earth, environmental and planetary sciences. Ten of the previous meetings in the series have been held in Santa Fe, New Mexico, and this was the third collaboration to bring the meeting to Europe, following the gatherings in Erice, Sicily in 2002 and Cargèse, Corsica in 2008. In addition to the usual purpose of these conferences (namely interactive assessment of the state of the science and directions for future research), the "Santa Utrecht" meeting served as a tribute to Cor Langereis on the occasion of his retirement as head of the Fort Hoofddijk lab, following a sterling career.

#### A tale of two forts: Utrecht and Fort Hoofddijk

The charming city of Utrecht is steeped in a history that extends back to the establishment of a Roman fort in about 50 CE, under the reign of Claudius, on what was then the main branch of the lower Rhine River. This *castellum* was part of the *Limes Germanicus*, the line of defense along the Germanic frontier, which included a number of fortifications on the lower Rhine. The fort

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# Visiting Fellow Reports

Low temperature magnetic hysteresis of 4C monoclinic Pyrrhotite (Fe<sub>7</sub>S<sub>8</sub>). Michael Volk IRM (was LMU Munich) mvolk@umn.edu

Pyrrhotite refers to a group of non-stoichiometric iron sulphides ( $Fe_7S_8$ - $Fe_{11}S_{12}$ ). They are common to the Earth's crust but more importantly are a major magnetic phase in some meteorite classes, especially Martian. Therefore, understanding the remanence properties of pyrrhotite is important.

The pyrrhotite crystal structure is hexagonal of NiAs type and consists of alternating Fe and S layers. Different stacking of iron filled and vacancy layers along the crystallographic c-axis, leads to several superstructures (e.g. 4C, 5C) [Morimoto et al., 1970]. The most important one is the 4C phase (Fe<sub>7</sub>S<sub>8</sub>), where alternating layers of ordered Fe vacancies create a slight distortion of the unit cell. This distortion lowers the crystal's symmetry to monoclinic and creates an imbalance in the magnetic moment of the sublattices. Therefore, the monoclinic (or pseudo-hexagonal) variety is ferrimagnetic while the hexagonal phases are antiferromagnetic.

At ~30 K pyrrhotite undergoes the Besnus transition ( $T_{Bes}$  see IRM Quarterly 21:1 [Rochette et al., 2011]) for review and history). The transition is marked by a demagnetization of the remanent magnetization and drastic changes of the rock-magnetic parameters. The mechanism behind the transition, however, is not well understood. One possible explanation is a crystallographic transition from monoclinic to triclinic as proposed by [Wolfers et al., 2011]. Another possible mechanism is the interaction between different superstructures, thus the transition is extrinsic to the 4C phase [Charilaou et al., 2015; Koulialias et al., 2015].

Inspired by the investigation of the basal plane anisotropy of [Fabian et al., 2011], we used a similar approach to explore the magnetic properties of a single crystal of 4C pyrrhotite from Morro Velho, Brazil. However, as we were interested in the changes across the Besnus transition, the experiments had to be done at low temperatures spanning the transition (30 K).

Thermomagnetic curves (Ludwig Maximilians University, Munich), showed no contamination of either hexagonal pyrrhotite or other magnetic phases. The crystallographic orientation was determined at the characterization facilities of the University of Minnesota with electron backscatter diffraction (EBSD) (JEOL 6500). The c-axis of the crystal is oriented almost normal to the sample surface. We defined the pseudo-hexagonal  $a_1$ -axis at 0° giving  $a_2 = 57\circ$  and  $a_3 = 116\circ$  with respect to  $a_1$ , close to the expected values.

Using the IRMs Princeton Measurements Corporation vibrating sample magnetometer (VSM) with He cryostat we measured hysteresis loops and backfield for every temperature. I was able to measure 22 temperature steps spanning the Besnus transition [Besnus and Meyer, 1964; Rochette et al., 2011]. The sample was rotated from 0-360° with a full hysteresis loop was measured every 5° and backfield curves every 10°. This was repeated in 2 K steps from 50 K to below the Besnus transition at 20 K, totaling 2160 measurements.

The hysteresis loops above the Besnus transition show a strong directional dependence (Fig. 1). This shows a considerable anisotropy within the basal plane, even though the c-axis is the magnetic hard axis. At room temperature, the a-axis is easiest to magnetize, while measuring 30° away is significantly harder. The shape of the hysteresis loops changed considerably at low temperatures (Fig. 1b). At 40 K, hysteresis loops in between a-axis display a second inflection, as described by [Koulialias et al., 2015]. Because we have a series of hysteresis loops at different orientations we can see that the double inflection (DI) only appears at certain crystallographic orientations. Below the Besnus transition,  $B_{(300 \text{ K})} = 1.5 \text{ mT}$  increases to  $B_{(20 \text{ K})} = 57 \text{ mT}$ ,  $B_{rr}(300 \text{ K}) = 2.9 \text{ mT to } B_{rr}(20 \text{ K}) = 47 \text{ mT and } M_{rs}(300 \text{ K})$ K) = 1.2 Am<sup>2</sup>/kg to  $M_{re}(20 \text{ K}) = 15 \text{ Am}^2/\text{kg}$ , while  $M_{e}$ stays approximately constant.

To visualize the SI phenomenon, we calculated the first derivative of the descending branch of the hysteresis loop, where the SI appears as a secondary peak. At 46 K (Fig. 2a), the SI is only visible in-between the crys-



Figure 1. Hysteresis loops at room temperature, 46 K (above  $T_B$ ) and 20 K (below  $T_B$ ) measured along one of the crystallographic a-axis (blue) and 30° with respect to a axis (green).



Figure 2.dM/dT of the descending branch of the hysteresis loops at 46 K (a) and 20 K (b). Dashed lines show the crystallographic axis, the square marker shows the projection of the c axis on the basal plane. Red patches show second inflection as positive dM/dB. Radial distance is the applied field in T.

tallographic a-axis and completely disappears below the Besnus transition (Fig. 2b).

The study has now been published under [Volk et al., 2016].

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## Recognizing rock magnetic properties of organic-rich shale rocks from Northern Poland.

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#### Introduction

In this project we focused on Silurian organic-rich mudstones, which represent a potential source of unconventional gas (e.g. Grotek, 1999, Karcz et al., 2013). Our aim was to recognize rock magnetic properties of samples from Silurian shale rocks from Northern Poland, which may inform about burial diagenesis and conditions during sedimentation. The results of the measurements so far have given us a general concept of the magnetic mineral assemblage in the shale rocks of the Pelplin Formation and Jantar Member, the sweet spot layer. Therefore, possibility of occurrence of minerals like magnetite, pyrrhotite and maghemite were demonstrated. However, occurrence of pyrrhotite or maghemite was questionable and further investigation was necessary. In order to fully recognize this problem and to determine magnetic nanoparticles, additional rockmagnetic measurements were performed at the Institute for Rock Magnetism.

To better understand the burial diagenesis and conditions of sedimentation we performed detailed measurements in the 300 K - 10 K temperature range. These analyses provided additional information about magnetic nanoparticles, not detectible in previous research to fully characterize the magnetic mineral composition.

#### Geological setting

The analyzed Wenlockian shales belong to Pelplin Shale Formation and Llandoverian Jantar Member. The



Figure 1. Results of MPMS measurements of remanence in low temperature range (10-300K) for selected samples from the Pelplin Formation.

rocks were deposited in the western part of the Baltic Basin (a distal Caledonian foredeep basin, where the active front of the Caledonian collision was the main factor of subsidence and flexural bending), in the siliciclastic zone, which was recognized as the accretionary prism of Caledonides (e.g. Jaworowski, 2002). However, the significant abundance of carbonates may suggest a secondary source of sediment - eastward carbonate platform on the Baltica margin.

#### Samples and methods

Samples come from two lithological facies from two shale gas exploration wells (A, B) located in Northern Poland. Samples JAN1 - 3 represent the most prospective (in context of gas exploration) shale layer, called Jantar Member, where reflectance index values ranging up to 1.42% Ro, while TOC even achieved 5% (Karcz et al., 2013). Samples were collected from depths below 3600 m (drill core A) and below 3900 m (drill core B). A second type of samples (PEL1 - 3) represent typical mudstones from the middle part of the Pelplin formation, which contains smaller amounts of TOC (not excluded 1.5%). The values of reflectance index ranging from approximately 1.20% Ro. The depth of the collected samples reaches ~ 3600 m.

The basic measurements start with applying 2.5 T field to reach SIRM (Saturated Isothermal Remanent Magnetization), the sample is then cooled down sample from 300 K to 10 K in a weak magnetic field (5  $\mu$ T) in 5 K steps. This part of the measurements is called Room Temperature SIRM (RT-SIRM) on cooling. In general, the SIRM (as a measurement of remanence) is

performed in zero field, but according to Aubourg and Pozzi (2010) application of 5  $\mu$ T field allows to intensify the P-behaviour, determined by the occurrence of paramagnetic minerals. This procedure was investigated by C. Aubourg and M. Kars on claystone samples. The next step was to apply a 2.5 T field at 10 K (LT-SIRM). After switching off the magnetic field the sample was warmed up to 300 K in Zero Field (ZFC) and the remanence measured in 10K steps. A more comprehensive measurement sequence comprises the two previous steps, the warming of the RT-SIRM, and a Field Cooling (FC) curve, which is a measurement of the remanent magnetization upon warming of a continuously induced 2.5 T field during cooling.

Hysteresis Loops as a function of temperature were measured to determine changes of mineral composition, which influence the different shapes of the ZFC curves. Two samples JAN1 and JAN3 (sweet spot formation) were comprehensively measured. Hysteresis loops was measured at 10, 20, 50, 90, 150 and 300 K.

#### Results

Samples from the Pelplin Formation (mudstones and calcareous concretions) display well developed Vervey transitions on the RT-SIRM curves (Fig.1b,d), while in both ZFC and FC curves the transition is hardly visible (Fig. 1a,c). The transition occurs at ~110-115 K. For this formation the Morin transition of hematite is well marked and occurs at ~220-240 K on the RT-SIRM curves (Fig.1b). Higher values of ZFC than FC remanence inform about the occurrence of multidomains (MD) grains (Fig.1a), which is confirmed by substantially lower re-

covery of magnetization on RT remanence curve during heating (Fig.1b; Bilardello and Jackson, 2013).

Samples from Jantar Member display very well developed Vervey transitions on RT-SIRM, which are also well visible in both ZFC and FC curves. In all specimens, the transitions occur at ~120 K, suggesting probably pseudo-single domain (PSD) or multidomain (MD) magnetite, due to not fully recovered remanence upon warming of the RT-SIRM (op. cit.). The higher values of ZFC than FC remanence is indicative for multidomains (MD) grains. Around 60% of FC imparted at 10K is lost upon warming to 35 K, may suggest occurrence of very small, probably superparamagnetic particles (e.g. Passier and Dekkers, 2002).

#### Conclusions

Various magnetic mineral assemblage of shale rocks exhibit changing of sedimentation and diagenesis processes. The Jantar Member, deeper and organic-rich layer, show presence of SD or PSD magnetite, which is probably a result of chemical process (illitization of smectite). Absence of pyrrhotite suggest that these rocks were buried to a maximum of 200°C, which corresponds to values of thermal maturity of organic matter (not excluded 1.42% Ro).

The Pelplin Formation, which is shallower and exhibits lower values of TOC, has different magnetic mineral composition, however MD magnetite is also present. Similarly, the occurrence of magnetite is probably the result of illitization. In the Pelplin Formation the appearance of hematite is interesting: we suggest that hematite was transported by bottom currents into oxic conditions at the bottom. This hypothesis is in agreement with precipitation of calcareous concretion in suboxic conditions at the bottom and lower values of organic matter. Moreover, an occurrence of hematite in mudstones and concretions in Peplin Formation determine that during sedimentation of clastic material, compaction and cementation of concretions stable oxic conditions were present at the bottom.

This project adds important information about the magnetic mineral assemblage of the analyzed shale rocks, and allows us to fully understand the changing of sedimentation and burial conditions and also, to add some details for previous investigations. The occurrence of hematite was quite surprising, however it also informs us about the sedimentation conditions and preservation of organic matter. The results obtained from the Visiting Fellowship will be included in an article for publication.

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# Service announcement!

## The IRM will be hosting a new

## **Summer School for Rock Magnetism**

Magnetic geoscience research uses sensitive, nondestructive measurements on natural materials to illuminate geomagnetic field history, tectonic processes and environmental changes. Learn more about the fundamentals and applications at the fourth biennial Summer School in Rock Magnetism (SSRM), which will be held June 4-13th, 2018 at the Institute for Rock Magnetism (IRM) in Minneapolis, MN. The 10-day program is targeted at graduate students and advanced undergraduate students in rock magnetism, paleomagnetism, and associated fields. Students will receive intensive instruction in rock magnetic theory and laboratory techniques. A daily schedule of lectures, hands-on laboratory measurements, and data processing will introduce students to the fundamentals of rock magnetism and paleomagnetism and the practical aspects of collecting and interpreting data responsibly. Instructors for the summer school will be primarily IRM faculty and staff.

Details for application and housing will be posted soon on our website:

# www.irm.umn.edu

# **Current Articles**

A list of current research articles dealing with various topics in the physics and chemistry of magnetism is a regular feature of the IRM Quarterly. Articles published in familiar geology and geophysics journals are included; special emphasis is given to current articles from physics, chemistry, and materials-science journals. Most are taken from ISI Web of Knowledge, after which they are subjected to Procrustean culling for this newsletter. An extensive reference list of articles (primarily about rock magnetism, the physics and chemistry of magnetism, and some paleomagnetism) is continually updated at the IRM. This list, with more than 10,000 references, is available free of charge. Your contributions both to the list and to the Current Articles section of the IRM Quarterly are always welcome.

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#### cont'd. from pg. 1...

# News in Rock and Paleomagnetism!

We congratulate Fabio Florindo of the Istituto Nazionale di Geofisica e Vulcanologia (INGV) in Rome, Italy, for becoming the new president of the EGU

# "Earth Magnetism and Rock Physics" division!

Fabio has recently also become the new editor in chief for Reviews of Geophyscs

https://eos.org/editors-vox/introducing-thenew-editor-in-chief-for-reviews-of-geophysics was destroyed in about 270 CE, and the medieval town of Utrecht was eventually established on the same site; some remnants of the castellum have been found in excavations beneath the present cathedral square. The Roman name Traiectum alludes to a crossing point on the river, and it survives in the second syllable of Utrecht, the first syllable of which comes from the Old Dutch uut (downriver), apparently added to distinguish U-trecht from Maas-tricht, on the river Maas (aka Meuse).

Sixteen centuries later, during the Eighty Years War (1568–1648) for Dutch independence from Spain, another series of fortifications was built in the region, which, in combination with natural bodies of water and intentional flooding of low-lying areas, could provide an effective line of defense. This was the Hollandse Waterlinie or Dutch Waterline. Still later, after the end of the Napoleonic Wars in 1815 and the establishment of the United Kingdom of the Netherlands, the Nieuwe Hollandse Waterlinie was formed, passing just east of Utrecht. Many of the Waterline fortifications are well preserved, including Fort Hoofdijk, built in 1875-1877 and located in what is now the botanical garden of the University of Utrecht. Because of the isolation from modern mechanical disturbances, the magnetically quiet environment, and the iron-free construction of the gunpowder storage room (to reduce the danger of sparks), the Fort was designed (fortuitously) as an ideal home for a paleomagnetic and rock magnetic laboratory. Such a laboratory was indeed established in 1954 in the buildings of the Royal Dutch Meteorological Institute, and moved to the Fort in 1963. In 1963 the Fort was in a



Attendees listen closely during one of the sessions.



Conference selfie, photo by Priyeshu Srivastava.

remote location in the polders East of Utrecht, but since then the campus of Utrecht University has developed around it. The paleomagnetic laboratory Fort Hoofddijk has been in continuous operation in the same location longer perhaps than any other paleomagnetic lab in the world.

As and Zijderveld [1958] describe a then-recently-built astatic magnetometer capable of measuring moments down to 10<sup>-7</sup> emu (10<sup>-10</sup> Am<sup>2</sup>), comparable to the sensitivity of many RF-SQUID magnetometers, as well as a routine to demagnetize paleomagnetic samples using alternating fields. It was housed in a seismographic vault (at the Royal Dutch Meteorological Institute), with constant temperature and relatively high humidity to minimize problems due to static electrical charges. Prior to that, students did qualitative paleomagnetic polarity work on lavas using in-situ field measurements with a magnetic compass [Rutten and Veldkamp, 1958]. The Fort is now a state-of-the-art laboratory for modern paleomagnetic and rock-magnetic research.

#### **ICRM 2017**

The Santa Fe Conference series was designed to complement large society meetings such as AGU, by providing the opportunity for group discussions that are in-depth, interactive, informal and more sustained than is generally possible in large-scale meetings. One of the special features of the Santa Fe conferences is what Subir Banerjee has always enjoyed calling the "cultural enrichment" sessions, in which invited keynote speakers from fields outside of rock- and paleomagnetism give their perspectives on problems of mutual interest, with the aim of catalyzing new interdisciplinary approaches and collaborations. This year we were fortunate to have two gifted scientists and communicators in this role: **Oliver Plümper** (University of Utrecht) and **Benjamin Gilbert** (Lawrence Berkeley National Laboratory).

The oceanic lithosphere plays a significant role in the global water cycle. Large volumes of water are incorporated by serpentinization (hydration of olivine to produce serpentine, magnetite, brucite, hydrogen and heat, as well as substantial volume increases) near spreading ridges, and then liberated at subduction zones, where increased pressure and temperature drive the reaction the other way, liberating large amounts of water. The involvement of magnetite, of course, attracts our interest. Oliver Plümper works broadly in mineralogy, fluid-rock interaction and rock deformation on various spatial scales, using electron microscopy and other methods, and he opened the meeting by presenting interesting research on serpentinization and related structural phenomena, including the development of high-diffusivity fluid pathways by crystal-plastic deformation in olivine [Plümper et al., 2012a] and by reaction-induced fracturing [Plümper et al., 2012b], which allow water to get in and react during serpentinization, as well as fluid channelization in dehydrating serpentinites, allowing water to escape back to the hydrosphere [Plümper et al., 2017a].

Ben Gilbert is co-founder (with Jill Banfield and Glen Waychunas (keynote speaker at the Santa Fe V conference in 2000)) of the Berkeley Nanogeoscience program, using advanced analytical techniques such as synchrotron spectromicroscopy to study phenomena including crystal growth and nanophase structure. A



Cor fest 1: Cor Langereis wearing red in the center, facing the camera.

very interesting aspect of his work is the observation of iron redox dynamics at the nanosecond scale, using time-resolved x-ray spectroscopy. Ben discussed a field study of clay weathering and its effects on water quality in a Colorado watershed. A series of mineralwater reactions, initiated by pyrite oxidation, release metal contaminants and nanoscale iron (oxyhydr)oxides into ground- and river water. Contaminant mobility is closely linked to the biogeochemical reactions that cycle iron between redox states and between soluble species and nanoscale mineral precipitates. He also discussed the structure of ferrihydrite, a hydrated nanophase ferric oxyhydroxide that accounts for much of the bioavailable and geochemically reactive iron in aerobic sediments, but whose detailed architecture has remained elusive. A novel structural analysis of total X-ray scattering data acquired from 6-line ferrihydrite, using whole-nanoparticle models and a reverse Monte Carlo (RMC) approach, showed that  $\sim 20\%$  of the iron is in tetrahedrally coordinated sites, and that long-range vacancy disorder is essential for optimum fits to the scattering data.

These keynote lectures and ensuing discussions were complemented by six topical sessions, which we briefly summarize below, in which invited speakers gave their views on the current state of research and future trends in six specific areas: 1) Fundamental rock magnetism; 2) Sources and significance of crustal magnetic anomalies; 3) Environmental magnetism; 4) Determining geomagnetic field behavior from terrestrial and extraterrestrial materials; 5) Sediment magnetization - acquisition and preservation; and 6) Advanced approaches to rock magnetic characterization and data analysis. Two poster sessions included additional interactive presentations in these six areas. The entire program and abstract volume can be downloaded from the conference website (https://icrm2017.sites.uu.nl/home/)

1) Fundamental rock magnetism. The bulk magnetic behavior of rock samples is ultimately governed by the nanoscale and microscale structures and processes that occur within individual magnetic mineral particles, as well as the magnetic interactions among particles. In numerical models of the nonuniform magnetization states within individual particles, the physics require a spatial resolution of just a few nanometers (comparable to the exchange length). In combination with limitations on computer processing capability, this has restricted the models to particle sizes in the fine PSD range, but improvements in hardware and software continue to expand this. At the same time, our ability to make direct observations of micromagnetic structures at submicron resolution has continued to improve, through techniques including magnetic force microscopy, offaxis electron holography, Lorentz microscopy, focusedion-beam nanotomography and x-ray photo-emission electron microscopy. This convergence of modeling and observation capabilities is now revolutionizing our understanding of the major carriers of remanence in most natural materials.

**Karl Fabian** appraised the state of the art in micromagnetic modeling of larger PSD and MD particles, where many possible local-energy-minimum remanent states exist, separated by energy barriers which can be overcome by thermal energy to drive transitions between states. Computation of all the states and barriers in an ensemble of particles will ultimately provide the long-sought basis for modeling the statistical evolution of thermoviscous remanence, and greatly strengthen the quantitative link between remanence and paleofields. From the observation side, **Trevor Almeida** reviewed his work on electron-holographic images of fine PSD (~200 nm) magnetite particles at elevated temperature (up to 580°C). These commonly show vortex structures, which in finer particles reorganize at a blocking

temperature well below the Curie temperature  $T_c$ , while in larger particles remaining stable to just below  $T_c$ , directly demonstrating the ability of such structures to hold high-stability paleofield records. In his assessment of the PSD state, **Andrew Roberts** provocatively suggested that the term PSD should be eliminated from future rock-magnetic publications, replaced by the term vortex, which more explicitly describes the micromagnetic configurations that now seem to be almost universal in those particles. He also argued that the Day plot of hysteresis ratios has outlived its usefulness, which prompted lively discussion for the rest of the conference.

Multi-particle micromagnetic structures of varying complexity are present in intracellular bacterial magnetite in intact chains, double chains and collapsed chains, for which **Ramon Egli** ran forward models of FORC behavior that required several years of processing on a number of desktop computers. Moment reversal in isolated SD particles, by coherent rotation, contrasts with reversal in magnetosome chains by a mechanism including fanning. The differences were reflected in distinctive FORC signatures, thus providing a potential means of distinguishing between biogenic and abiogenic origins for SD magnetites in natural sediments.

2) Sources and significance of crustal magnetic anomalies. The heterogeneous distribution of magnetic materials in the crusts of the Earth and other planets results in magnetic field anomalies, which are mapped at different elevations and spatial scales by satellites, aircraft or ships. Inversely, these field anomalies provide information about crustal structure, depth to the Curie isotherm, etc., albeit with some degree of nonuniqueness.

Although these anomalies are commonly due primarily to magnetizations induced by the planetary core field, in soft crustal carriers like magnetite, remanencedominated anomalies move to the forefront in extinctdynamo planets like Mars, and in certain terranes containing very stable magnetic phases with high remanent/ induced magnetization ratios (Q). Suzanne McEnroe has researched remanent-based anomalies extensively, mostly associated with rocks containing rhombohedral oxides of the hematite-ilmenite series containing exsolution structures down to nanometric scales, leading to the remarkable discovery of interfacial magnetic ordering or "lamellar magnetism". She discussed the canonical case study of the Modum area in south Norway, as well as the Peculiar Knob ore deposit in Australia, and Laurie Brown described the magnetization history of similar rocks of the Tellnes ore body, an ilmenite-rich norite that intrudes anorthosite in the Rogaland Igneous Province in southernmost Norway.

The terrestrial marine magnetic anomalies document the formation of oceanic lithosphere and evolution of plate geometries and relative and absolute motions. **Carmen Gaina** described dramatic changes in the MMA configuration in the Eocene crust of the North Atlantic, that appear to reflect a significant decoupling between lithosphere and mantle at that time. Evidence



Cor fest 2.



Cor fest 3.

for this is also found in the Indian Ocean and the Pacific.

The relationship between magnetic anomalies and large impact basins on Mars has led to the idea of shock demagnetization related to high-pressure magnetic transitions [e.g., Rochette et al., 2003]. **Pierre Rochette** discussed new experimental magnetic measurements under variable pressure and temperature on hematite in a "Russian alloy" cell with a 2G magnetometer. The results show that the temperature of the Morin transition increases linearly with pressure, from 250K at zero P to 300K at 2 GPa, and thus impacts can cause demagnetization of hematite.

The problem of nonuniqueness in inverting magnetic field data was insightfully reviewed by **Dave Clark** who outlined a number of approaches for optimizing the amount of information recoverable from remanent and total magnetic anomalies [Clark, 2012, 2014]. One example is Helbig analysis, in which the source is assumed to be finite and isolated, which enables determination of the total magnetic moment of the source, independent of the non-unique geometric distribution, and also the location of the source center. Another example is remote in-situ determination of the NRM and induced magnetization vectors and Koenigsberger ratio, by deploying dual vector magnetic variations, operating in base station mode within a magnetic anomaly of interest.

Magnetic anomaly mapping and source modeling extend down to microscopic scales in scanning SQUID magnetometry. **Hirokuni Oda** described some successful applications in paleomagnetism and rock magnetism, including sub-millimeter scale magnetostratigraphy of a ferromanganese crust; magnetic mapping of fault gouge samples; calculation of NRM dipole moment vectors of single mineral grains; and even the possibility of dating single zircon crystals by matching their zoned magnetic polarities to the global reversal time scale. In general, inverse modeling is more applicable to environmental magnetism, where the approximation of unidirectional magnetization is appropriate for ARMs and IRMs, than to paleomagnetism, where the source distribution varies in both direction and intensity.

3) Environmental magnetism. The complicated interactions among climate, geology, biology, and chemistry affect the movement of Fe as it cycles through the environment, and strongly influence the formation and alteration of iron mineral assemblages. Marine and continental deposits record the processes and conditions affecting sediment as it travels from sources to sinks, exerting strong controls on the mineral compositions, abundances and particle size distributions of iron minerals. Magnetic characterization methods are sensitive indicators of these physical and chemical characteristics of iron minerals in natural and anthropogenic samples, allowing connections to be made between mineral magnetic properties and past environmental conditions and processes.

Ken Kodama showcased the power of cyclostratigraphic magnetic studies, focusing primarily on the Shuram Neoproterozoic C isotope excursion, which may correspond to oxygenation of the world's oceans. The magnetostratigraphies in sections in Death Valley and the Flinders Range indicate that the excursion was globally synchronous. Rock-magnetic cyclostratigraphies for these and a third section in and South China exhibit Milankovitch periodicities, and the phase of the precession cycle appears to shift annually, suggesting the astonishing possibility of resolving seasonality in the Precambrian sedimentary record.

FORC analysis is evolving rapidly, with continual innovations in data acquisition, processing and interpretation (some of which were also discussed in session 6 as described below). Andrew Roberts presented a set of new FORC-like plots developed by Xiang Zhao, which use non-uniform field spacing to increase measurement efficiency dramatically, and which incorporate some additional measurements to isolate the "transientfree" part of hysteresis behavior (cf. transient hysteresis [Fabian, 2003; Yu and Tauxe, 2005]). Subtracting the transient-free FORC from the full FORC yields the transient FORC, related to domains and nucleation/denucleation of vortex structures. Further lively discussion continued on the values of the Day plot and of the term "PSD".

In a talk entitled "Magnetite is everywhere", Barbara Maher considered the occurrence and properties of magnetite in two distinct environments: loess/ paleosol couplets and the human brain. The evolution of magnetic mineralogy during pedogenesis has been the subject of debate, with one school favoring initial formation of magnetite and subsequent oxidation to maghemite, and another favoring initial formation of "hydromaghemite" and subsequent alteration to hematite. This matters because the mechanism affects the paleoenvironmental interpretation. High-resolution TEM lattice fringe fingerprinting identifies magnetite as the dominant soil-formed ferrite. New work shows that magnetite in the brain is not primarily of biogenic in-situ origin as previously suggested; particle shapes and compositions indicate dominantly exogenic origins (particles with d<200  $\mu$ m can enter the brain through the olfactory bulb).

Three short talks closed the session. Eric Font; considered the role of Deccan volcanism in the terminal Cretaceous extinction. Magnetostratigraphy and radioisotopes place the trap ages within 250-500 kyr of the K-Pg boundary. Akaganeite, a Cl-bearing oxyhydroxide that forms in acidic and hyper-chlorinated environments is present in several boundary sections, and may be linked to the Deccan volcanism. Joy Murazko described the application of FORC PCA unmixing to Iberian margin sediments, identifying three end members (magnetotactic bacteria, fine low-coercivity SP-SSD aggregates, and detrital PSD magnetite) whose stratigraphic variability paints a picture of environmental changes. Tilo von Dobeneck discussed an interesting case of cyclic magnetite dissolution in the NW Pacific. Relative paleointensity dating and the MB boundary show strong magnetite depletion during glacial intervals, controlled by deep water circulation and related to massive glacial C trapping.

4) Determining paleomagnetic field behavior from terrestrial and extraterrestrial materials. Documenting and understanding the behavior of the Earth's magnetic field, and those of other planets and planetesimals, are among the largest research efforts of our scientific community. Field variations through time tell us about convection in the liquid iron core; heat flux across the core-mantle boundary; crystallization of the solid inner core; and other phenomena of the Earth's deep interior. Paleofield intensity is a particular challenge, because of the strong dependence of NRM intensity on the properties of the recording medium. Ancient paleofield records are another distinct challenge, due to degradation of the original signal by numerous mechanisms.

Some remarkable findings have been achieved through the nanopaleomagnetic techniques employed by the Cambridge group, a case study of which was offered by Claire Nichols, involving pallasite meteorite samples. These contain olivine crystals surrounded by a matrix of metallic Fe-Ni alloy unmixed into the Widmanstätten pattern with intergrowths of taenite and kamacite lamellae, between which are "cloudy zones" of tetrataenite islands in an iron-rich matrix, formed by spinodal decomposition. The tetrataenite islands, of the order of 100 nm in diameter, are excellent paleomagnetic recorders, and whereas their remanence cannot be isolated by conventional magnetometry, it can be imaged using X-ray Photoemission Electron Microscopy (X-PEEM) and spatially resolved XMCD. Different samples have different island sizes, reflecting different cooling rates at different depths in the parent body, and therefore different times of remanence acquisition. The sample collection thus represents time sampling of a single parent body, allowing reconstruction of the life cycle of the parent-body dynamo, and providing constraints on the age of core solidification.

A well-behaved (geocentric axial dipole) paleofield allows the study of lithospheric plate behavior, the bread and butter of classical paleomagnetism. Dennis Kent discussed a rapid polar shift near the Jurassic-Cretaceous boundary, recently discovered in a composite APW path constructed for all Earth's major cratons in a common reference frame. A "monster shift" from 160-145 Ma could be due to TPW, resulting from a major mass redistribution, possibly related to slab breakoff and subduction reversal along the western margin of the Americas. This corresponds in time to the development of Ghawar, the world's largest oil field, where Jurassic-Cretaceous boundary source and cap rocks were respectively deposited in equatorial and desert latitudes during rapid plate motion. Andy Biggin commented that there is a large change in reversal frequency at same time as this monster shift, perhaps due to changes in the coremantle boundary thermal configuration.

The quest for detailed and reliable paleofield records continues to lead to the study of novel materials that may contain such records. **Josh Feinberg** showed the great potential of speleothems, and **Ron Shaar** reviewed that of archeological slag materials for the preservation and retrieval of high-resolution, high-fidelity records of short-term geomagnetic behavior such as directional "jerks" and intensity spikes. Speleothems acquire remanence by a matrix-crystallization mechanism that locks in the detrital magnetic grains in dripwater, with a lock-in timescale of less than one year and no inclination error. Extremely high dating accuracy is possible with 230Th methods, and annual-scale resolution is made possible by the presence of couplets seen by microscopy. A detailed record of the Laschamp excursion has been obtained in this way [Lascu et al., 2016]. Challenges for broader study of speleothems include their low magnetization intensities and the 600 ka limit of 230Th dating. Similarly, archeomagnetic studies of pottery, slag, ovens, and burnt structures has provided a detailed reconstruction of field behavior in the Levant over the last few millennia, including the discovery of the remarkable Iron Age geomagnetic intensity spikes, with local doublings of field strength on decadal timescales [e.g., Shaar et al., 2017]. Nevertheless, a number of sources of error remain, and careful methodological work is still critical. Ron discussed factors including experimental protocols, anisotropy corrections, coolingrate corrections, reliability criteria and software automation.

5) Sediment magnetization - acquisition and preservation. The quasi-continuous nature of deposition makes sediment magnetizations an essential target for studies of geomagnetic field variations. However, the process through which sediments acquire a depositional remanent magnetization (DRM) is complex and still incompletely understood. Further, DRMs are modified by subsequent bioturbation, diagenetic effects, burial/compaction, dewatering or other mechanisms which give rise to a post-depositional remanent magnetization (pDRM). As a result, there are still important limitations in our ability to disentangle paleomagnetic and environmental signals.

Ramon Egli began the session with an enlightening review of DRM models, which are of two types. Néel-type equilibration models, with the field exerting a weak bias on a decaying randomization process, have the problem of predicting full alignment. Attention has thus focused on aggregation models as an alternative, with flocs of magnetic and nonmagnetic particles resulting in decreased alignment efficiency. Ramon showed by numerical simulations that such models predict relatively strong anisotropy due to two factors: translationrotation coupling of flocs produces strong inclination shallowing; and inclusion of velocity gradients results in a DRM whose orientation and intensity depend on the angle between field and flow. The modeling results imply that syn-depositional DRM always provides biased and environmentally-modulated records of the Earth magnetic field. He concluded that reliable sedimentary paleomagnetic records depend on gradual replacement of DRM by pDRM in the uppermost few cm of sediment.

**Dave Heslop** illustrated that simple model considerations can lead to strong conclusions about the orientation distributions of particles and their remanent vectors in sediments. A summary of sediment magnetic data shows that the ratio NRM/IRM typically ranges from ~10<sup>-3</sup> to ~10<sup>-2</sup> depending on field strength. Consider easy axes and particle moments in the NRM state to be oriented around the field direction according the Fisher distribution ( $0 \le \theta \le \pi$ ), with maximum probability density for  $\theta = 0$ . The net contribution of each to the NRM vector sum is proportional to  $\cos(\theta)$ . In the IRM

state all particle moments are oriented within 90° of the field direction, and the contribution of each to the IRM vector sum is proportional to  $|\cos(\theta)|$ . From this one can conclude that a typical DRM/IRM ratio corresponds to a precision parameter  $\kappa \approx 10^{-2}$ , i.e., the particle moments in the NRM state are almost randomly oriented. If a sediment contains different magnetic populations, e.g. detrital and biogenic, with different alignment efficiencies, the populations will give different relative paleointensities in the same sediment (different NRM/IRM for different coercivity windows).

The lock-in depth of sedimentary remanence remains an unresolved question. The flocculation DRM model predicts zero lock-in depth; biogenic remanence may be acquired at some depth by mobile bacteria; and pDRM mechanisms allow significant lock-in depths. Joe Stoner revisited the question of lock-in depth in deep-sea sediments, through a detailed comparison of Holocene aged ocean sediment core data with ultrahigh resolution (~200 cm/kyr) records collected from the Greenland and Iceland margins and lakes. Tuning the deep sea sediment PSV records to the UHR PSV composite indicates lock-in times of hundreds or thousands of years (lock-in depths 10-30 cm). Significant features of the deep-sea records could be reproduced through convolution modeling using three parameters: the thickness of the mixing zone, the depth of full lock-in, and the shape of the lockin curve (% locked vs depth). Toshi Yamazaki linked the lock-in depth of biogeochemical remanent magnetization carried by magnetofossils to bacterial strains and environmental conditions. Magnetotactic bacteria that produce equant octahedral magnetosomes may be aerotolerant, with largest populations near the sedimentwater interface where nutrient concentrations are high, resulting in near zero lock-in depth. In contrast, MTB that make bullet-shaped magnetosomes prefer chemical conditions near the oxic-anoxic transition zone and may result in remanence acquisition at depth.

Complexities due to changing chemical environments and sediment alteration were highlighted by **Janna Just** in a paleo- and rock-magnetic study of a sediment core from Lake Ohrid, the oldest existing lake in Europe; the record extends back to 1.2 Ma. Rock-magnetic and FTIR data indicate the occurrence of siderite and Fe-sulfide minerals only in glacial sediments, associated with high Fe-concentrations, with a dominance of iron-sulfides prior to and siderite after ~320 ka, likely reflecting a change in pore/lake water chemistry. Polarity transitions are clear when they are located within interglacial sediments, i.e., the Brunhes/Matuyama boundary and the base of the Jaramillo. In contrast the top of the Jaramillo is uncertain, and possibly duplicated by a later growth of greigite below the sediment surface.

Short talks by **Stuart Gilder** and **Rob Van der Voo** closed the session. Stu proposed that ARM anisotropy may be useful for characterizing sedimentary paleointensity, and presented the results of deposition experiments in a range of fields. The increasing alignment efficiency in higher fields was reflected in changes in the AARM tensors; in sufficiently strong fields ARMmax was parallel to DRM. Rob discussed the relationship between sediment remagnetization and the illitization of smectite, which is enhanced by deformation, resulting in ubiquitous syn-folding magnetizations. New techniques allow radiometric dating of illite and thus determination of the ages of folding and of late remanence acquisition.

6) Advanced approaches to rock magnetic characterization and data analysis. Recent advances in data processing and analytical techniques in rock and mineral magnetism have been driven in large part by the continuing importance of the general problem of "unmixing," i.e., estimating the contributions of individual geological inputs, or of individual minerals and grainsize fractions, to data measured on bulk geomaterials, which are inevitably mixtures. Selective measurement techniques allow some degree of direct component isolation, and mathematical unmixing methods help to sort out the contributions of individual components of natural mixtures.

**David Heslop** kicked off the session with a thoughtful overview of the unmixing problem and various mathematical and philosophical approaches involved. He advocated a "blind" unmixing approach in which the endmember components are not predefined but are instead determined from the variances and covariances of measured properties for a sample set. Such endmembers need not correspond to discrete particle sizes of particular mineral compositions; rather they can be thought of as natural system inputs that contain more basic ingredients in fixed proportions. He illustrated the concept by analogy with coffee samples containing variable proportions of cream and sugar; these are the natural endmember components, rather than each molecular species that may be isolated from the brew.

New developments in FORC analysis and related approaches continue to interest our community. A nonlinear method of Preisach analysis presented by Nathan Church holds great promise for characterizing stable remanence carriers in the presence of a large unstable MD background. It greatly improves selective sensitivity compared to FORC analysis by making measurements in zero field, thereby suppressing the unwanted MD signal. And nonlinear field spacing allows much more efficient data collection by sampling densely only where magnetizations change rapidly. Ioan Lascu outlined a principal-components approach to unmixing FORCs, by which individual-specimen FORC distributions can be represented as linear combinations of those of a specified set of endmembers. He also presented an elegant study in which the FORC signatures of vortex (de)nucleation and core reversal were determined by micromagnetic modeling using MERRILL, for a single particle isolated by FIB nanotomography.

Magnetic anisotropy is a relatively rapid, sensitive, quantitative and volume-integrated method of characterizing petrofabric, and various particular anisotropic magnetic properties selectively reveal different aspects of the bulk fabric. **Ann Hirt** provided a comprehensive overview of the physical basis of different anisotropic properties and materials, and evaluated current progress and outstanding problems in the application of magnetic anisotropy methods to petrofabric analysis, including the universal problem of unmixing. Focus areas identified for continuing/future work include: obtaining more data on the intrinsic AMS of para- and diamagnetic minerals; analog experimental studies with controlled materials and processes (e.g., sandbox deformation, 3-d printing of ferromagnetic particles with specified shapes, orientations and spacings); and more comparison of measured and modeled fabrics.

The final talk of this session brought us back full circle to the ideas of session 1, as Richard Harrison presented "a (domain) state-of-the-union address" on the nanopaleomagnetism project that he has led for the past 5 years. This remarkable work involves a combination of methods to obtain a complete characterization of the magnetic particles in a microscale sample volume: FIB nanotomography (serial slicing and imaging) allows detailed 3-D reconstruction of the geometry, orientation and location of each particle; this information provides the basis for detailed micromagnetic modeling (using MERRILL) of the spin architecture of each of the particles; and various techniques of electron microscopy/ holography enable direct imaging of those microstructures. Modeling the magnetic behavior of these particle ensembles as functions of field, temperature and time is the key link between the nanoscale and bulk sample behavior, and FORCulator simulations provide insight into many of the recognizable features in FORC diagrams. The long-awaited convergence of magnetic imaging resolution and modeling resolution/scale is indeed upon us.

#### Salute to Cor

To be both highly esteemed and genuinely well-liked by one's peers requires a special combination of attributes and Cor Langereis possesses these in great measure. The official program of the ICRM was finalized by a short tribute to Cor's career. Dennis Kent, Lisa Tauxe, and Rob Van der Voo were kind enough to share with us some of their memories regarding Cor and his work. They addressed Cor's three 'hobbies': magnetostratigraphy, geomagnetism, and tectonics; and Wout Krijgsman elaborated on what Cor has meant for the paleomagnetic lab in Utrecht - his efforts for (and also achievements with!) the Fort are hard to overstate. All of us who work in these fields, and especially those who have had the opportunity to work with Cor directly, have benefitted significantly from his insights.

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Utrecht Cathedral, photo by Betsy Leach.

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