

#### "BELGIAN ANTARCTIC METEORITES: CURATION AND RESEARCH"

#### **«BELAM»**

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# SCIENCE FOR A SUSTAINABLE DEVELOPMENT (SSD)



#### Data management

#### FINAL REPORT

## BELGIAN ANTARCTIC METEORITES: CURATION AND RESEARCH "BELAM"

#### SD/DA/01A

Promotors

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### ACRONYMS, ABBREVIATIONS AND UNITS

| BELAM     | Belgian Antarctic Meteorites   |  |
|-----------|--|--|
| Belspo    | Belgian Science Policy   |  |
| BRAIN     | Belgian Research Action through Interdisciplinary Networks, a BELSPO       |  |
| programme |  |  |
| Darwin    | Data Research Waterhouse Information Network (RBINS owned system)          |  |
| EDS       | Energy Dispersive Spectroscopy   |  |
| EMAT      | Electron Microscopy for Materials Science, University of Antwerp           |  |
| ESA       | European Space Agency  |  |
| IT        | Information Technology, service of RBINS                                   |  |
| JARE      | Japanese Antarctic Research Expedition (e.g. 51st in the 2009-2010 field   |  |
| season)   |  |  |
| Mars      | Plone <sup>®</sup> Open Source CMS/WCM by the Plone Foundation and friends |  |
| MoU       | Memorandum of Understanding, between NIPR and Belgian partners             |  |
| NIPR      | National Institute of Polar Research, Japan                                |  |
| RBINS     | Royal Belgian Institute of Natural Sciences                                |  |
| SAMBA     | Search for Antarctic Meteorites (BELSPO-supported project of VUB-ULB with  |  |
| NIPR)     |  |  |
| SEM       | Scanning Electron Microscope   |  |
| SLC       | Scientific Loan Committee  |  |
| ULB       | Université Libre de Bruxelles  |  |
| VUB       | Vrije Universiteit Brussel   |  |
|           |  |  |

#### 1. Introduction

Meteorites are the leftover building blocks of the Solar System, providing clues on its origin and evolution. They are classified in groups corresponding to different evolutionary phases of the Solar Nebula. Chondrites originated from the break-up of undifferentiated planetary bodies. Achondrites (iron, stony-iron and stones) derive from more evolved planetary bodies that have undergone differentiation comparable to the formation of the core, mantle and crust on Earth, and well as episode(s) of shock metamorphism during planetary collisions. The value of meteorites to document astronomical, solar system and terrestrial processes does not have to be further demonstrated. They have supplied and continue to provide data on stellar evolution and nucleosynthesis, the chronology of the Solar System, the formation of planets, cosmic rays bombardment, the deep crust of Mars and the Moon, and the different types of asteroids. They are often used to "calibrate" the instruments of the orbiters and landers used in planetary exploration. Moreover, meteorites attract public attention; they are important reference objects in museums and contribute to the promotion of natural sciences.

Meteorites from Antarctica are especially valuable because they are preserved in near pristine state and show no alterations due to temperature changes, contact with soils and chemical interactions. They concentrate in areas with glacier ablation, such as the foothills of the Sør Rondane mountain region, which makes search operations in this area more rewarding.

Joint field seasons in the Sør Rondane region of Antarctica in 2009-2010 (51st Japanese Antarctic Research Expedition; JARE 51), 2010-2011 and 2012-2013 (framed into the BELSPO supported SAMBA project) have yielded 1343 new meteorites, that are shared equally between Belgium and Japan. These field campaigns resulted from a successful collaboration between the National Institute of Polar Research (NIPR, Japan), the VUB and the ULB. The Belgian partners wanted to expand their partnership to the Royal Belgian Institute of Natural Sciences (RBINS, Belgium), where the Belgian share of the meteorites is now deposited for curation purposes.

Though a storage room for meteorites was initially present at the RBINS, there was a need to renovate it and make it suitable and optimal to host the new batch of meteorites coming from Antarctica, notably for temperature and humidity shifts. Similarly, a laboratory dedicated to meteorite processing had to be set up with the material required for the curation tasks. Moreover, rules to organize the curation, conservation and access to these precious and fragile samples had to be establish. Consequently, an important objective of the project was to set up a state-of-the-art curation facility for meteorites at RBINS, using the following methodology:

- ✓ Gather experience from well-established collections, in particular at NIPR, which manages a large collection of Antarctic meteorites, by visiting these facilities, and organising workshops in Belgium to benefit from European institutions with an established meteorite curation system.
- ✓ Set up facilities and curation processes according to international standards. This includes the establishment of a repository, loan guidelines, quality control, and a meteorite lab user manual that will present the different curation directives at RBINS.

✓ Make the collection publicly available through an easily accessible online database, after identification and characterisation of the samples.

A second objective was to develop a classification on meteorites from the collection. As a general rule for classification, the optical identification and classification of the meteorites followed the basic petrography, evaluating the relative amount of different minerals, the presence of chondrules, the amount and the distribution of metal phases, etc. (e.g., Hutchinson, 2004; Weisberg et al., 2006; Devouard and Zanda, 2013). The detailed classification was then performed by analyzing the chemical composition of the individual minerals, typically olivine and puroxene (e.g., Van Schmus and Wood, 1967; Rubin, 1990; Papike, 1998). To develop this meteorite classification in the frame of the BELAM project, the analytical facilities at RBINS, ULB and VUB were preferentially used. The aim was to be able to perform a quick and cheap classification in-house (i.e. using the ESEM-EDS and the Raman spectrometer). Moreover, the expertise of close collaborators of the BELAM project's partners was requested to assist in developing alternative methodologies (i.e. magnetic susceptibility with the CEREGE, Aix-en-Provence, France).

A last objective was to strengthen scientific activities oriented towards meteorites. Considering (i) the high value of the Antarctic meteorites, as stated above, and (ii) the new (and abundant) material collected in the field, the research activities were understandably oriented towards (micro-)meteorites coming from Antarctic and collected by the teams involved in the project. A variety of methods was applied to study these objects. Equipment available at RBINS, VUB and ULB was first used for petro-mineralogical characterization of the meteorites. Then, further in-situ investigations were carried out in laboratories belonging to the international network developed by the BELAM project's partners.

With the BELAM project, the RBINS gathered an impressive collection of Antarctic meteorites, which allows groundbreaking research and gives unique insights into this continent of extremes. This enhances the quality of Belgian research in this field and reinforces the strategic and international position of Belgium regarding Antarctic research.

#### 2. Methodology and results

#### 2.1. *Methodology*

The objectives of this project dedicated to Antarctic meteorites were to establish and organise the meteorite curation facilities at the Royal Belgian Institute of Natural Sciences (RBINS). This involved various steps. The first step was the organisation of the cooperation between partners with different missions and backgrounds. Registration in a national repository involved discussions on ownership and regulation of availability for research. Another aspect of the collaboration concerns the transfer of samples from Japan to Belgium and how to share the samples. Numbers of documents (agreements and MoU between the partners) had to be prepared with that aim.

To ensure the preservation of the samples, a renovation of the storage room was needed, including a close monitoring of the humidity and temperature. Similarly, a laboratory dedicated to meteorite solely had to be set up in order to prevent any kind of contamination during the processing these samples. To ensure best practices in the lab, a user manual was needed (see Annex).

Once the sample arrive at the RBINS, a routine had to be set up to describe the samples and register them in an online database suited for (or developed for) this kind of material.

One of the most critical steps for meteorite characterization is their identification/classification. This task, done in collaboration with NIPR, constitutes a crucial part of the curation work. The detailed description and accurate classification of the new meteorites guarantee the quality of the research performed on these samples.

At the RBINS, the identification of new meteorites was performed using the optical and electron microscope on polished mounts or petrographic (30  $\mu$ m thick) section. The preparation of polished mounts was done in the museum's meteorite lab, whereas the thin sections were delivered from an external laboratory. The optical identification and classification of the meteorites followed the basic petrography, evaluating the relative amount of different minerals, the presence of chondrules, the amount and the distribution of metal phases, etc. (e.g., Hutchinson, 2004; Weisberg et al., 2006; Devouard and Zanda, 2013). The detailed classification was then performed by analyzing the chemical composition of the individual minerals. As a microprobe was not available, we opted for EDS analyses (Quanta 20 ESEM (FEI), with energy-dispersive spectroscopy (Apollo 10 Sillicon Drift EDS detector; EDAX) at the RBINS), calibrated with a self-prepared meteorite reference sample, a polished mount of a highly equilibrated (petrologic type 6) ordinary chondrite belonging to the chemical group H (chemically reduced) and measured several times on a microprobe to become an in-house standard. These EDS analyses were performed under high vacuum, at a voltage of 20.00kV, magnification 3000x, spot size 6 on carbon-coated polished sections. The following Astimex standard were used: plagioclase, Cr-diopside and olivine for Na, Mg, Al, Si, K, Ca, Cr and Fe). This method is based on the experience acquired at the National Institute of Polar Science (Tachikawa, Japan), where hundreds of new meteorites are classified every year, and on the chemical classification of meteorites available in the literature (e.g., Van Schmus and Wood, 1967; Rubin, 1990; Papike, 1998).

In addition, during the project, an alternative classification method for ordinary chondrites, based on Raman spectroscopy (the Raman spectrometer is a Senterra, Olympus BX51, Bruker optics, at the RBINS), has been developed (Pittarello et al., 2015). This classification is based on the correlation between the Raman shift and the Fe content in olivine and pyroxene (Figure 1), in order to develop a quick method of chemical classification for ordinary chondrites. Besides, a classification by magnetic susceptibility has been used with the help of the CEREGE team (Pierre Rochette and Jerome Gattacceca; U. Aix-Marseille) (Debaille et al., 2017). Since the oxidation state and the amount of metallic iron are highly variable in meteorites, studying their magnetic susceptibility can also be helpful to characterize meteorites (e.g., Rochette et al., 2003; Folco et al., 2006). In meteorites, the magnetic susceptibility is proportional to the amount of ferro-magnetic phases, essentially Fe-Ni metal, magnetite and pyrrhotite (Rochette et al., 2012). In ordinary chondrite, the magnetic susceptibility is a direct proxy to the Fe-Ni metal amount because magnetite and pyrrhotite are absent.



Figure 1. Raman spectra and correlations deduced from the peak intensity and chemistry of the mineral phases investigated in meteorites in order to classify them

To make the meteorite collection publicly available for local and international researchers, clear rules for sample loan, destructive analyses, etc. must be established, based on existing rules and experience of RBINS, and in coordination with NIPR. It was important that a website developed and hosted by RBINS displayed this information. The DaRWIN (Data Research Warehouse Information Network) is an in-house solution developed by the Royal Belgian Institute of Natural Sciences (RBINS), as a Natural History collections management system for biological and geological samples in collections. The DaRWIN database currently manages information on more than 600,000 records (about 4 million specimens) housed at the RBINS. DaRWIN is an open source system, consisting of a PostgreSQL database and a customizable web-interface based on the Symfony framework (https://symfony.com). DaRWIN is divided into 2 parts: one public section that gives a "read-only" access to digitised specimens, one section for registered users, with different levels of access rights (user, encoder, conservator and administrator), customizable for each collection and allowing update of specimens and collections, daily management of collections, and the potential for dealing with sensitive information. DaRWIN stores sample data and related information such as place and date of collection, missions and collectors, identifiers, technicians involved, taxonomy, identification information (type, stage, state, etc.), bibliography, related files, storage, etc. Other features that deal with dayto-day curation operations are available: loans, printing of labels for storage, statistics and reporting. DaRWIN features its own JSON (JavaScript Object Notation) webservice for specimens and scientific names and can export data in tab-delimited, Excel, PDF and GeoJSON formats. More recently, a procedure for importing batches of data has been developed, based on tab-delimited files, making integration of data from (old/historical) databases faster and better controlled. Finally, quality control and data cleaning on several tables have been implemented, e.g. mapping of locality names with vocabularies like Geonames, adding ISO 3166 two-letter country codes (https://www.iso.org/iso-3166-country-codes.html), cleaning duplicates from people/institutions and taxonomy catalogues. DaRWIN is accessible online (http://darwin.naturalsciences.be). A Github repository is also available (<u>https://github.com/naturalsciences/natural\_heritage\_darwin; publication http://biblio.naturalsciences.be/library-1/rbins-staff-publications-2019/adam2019a</u>). Access is free and login is easily found on the start-page.

Still to make the Belgian collection of meteorites available for local and international researchers, a Scientific Loan Committee was established to give an expert advice about the loan requests. The documents/procedures (loan rules, lab user guide, etc) and Committee mentioned here above are the basics of meteorite curation facility.

Additional objectives were to set up a research network on meteorites, based on existing VUB-ULB collaboration and reaching out to colleagues in other institutions, and to carry out quality research on the Antarctic meteorites recently collected. These objectives can be achieved, for instance, through (i) networking and meetings in international conferences and (ii) peer to peer collaboration framed by joint research. This will also help gathering international experts participating in the meteorite curation work.

To achieve the BELAM project, the expertise of the partners RBINS-VUB-ULB and the complementarity of their skills appear as a key asset: The RBINS manages scientific collections in most fields of natural sciences and makes this material available to the scientific community. Moreover, this Institute is well equipped regarding mineralogy. The VUB is conducting research in the field of impact craters and their geochemical and isotopic signature, especially platinoid metals and light stable isotopes (e.g., oxygen isotope variability) to trace the origin of meteorite and asteroid parent bodies. This leads to a better understanding of large-scale planetary processes. The ULB has expertise in isotope geochemistry and geochronology, for both radiogenic isotopes and heavy stable isotopes. Privileged research themes include planetary differentiation from meteorite investigation, and the chronology of the early solar system. Representatives from VUB, ULB and RBINS are involved in the Scientific Loan Committee that decides on sample allocation for research and for exhibitions of Antarctic meteorites.

In this network, a post-doc (first Céline Martin and later Lidia Pittarello) was recruited for this project over a period of 4 years and shared her time between RBINS and VUB-ULB, thus constituting a link between the partners. The postdoc researcher participated in the whole chain of Antarctic meteorite handling and study, from registration and identification of all specimens and their website publication – which are conducted at RBINS – to focused and high-level research on selected meteorite types– which is conducted at VUB and ULB.

#### 2.2. Results

#### 2.2.1. Long-term preservation of the Antarctic meteorites at RBINS

<u>Agreements and MoU</u>: The first step of the BELAM project was the organisation of a cooperation between partners with different missions and backgrounds. This resulted in three agreements (a copy of these agreements is provided as Annexes): an Internal Agreement on curation and research of Belgian Antarctic meteorites between VUB, ULB and RBINS, a Deposit agreement on Antarctic Meteorites between VUB, ULB and RBINS, including the functioning of a Scientific Loan Committee, and a MoU between NIPR, VUB, ULB, RBINS concerning scientific cooperation and ways of sharing the Antarctic meteorites. The Scientific Loan Committee is active for allocating samples for either research or exhibitions.

<u>Receiving and handling the samples</u>: The Antarctic meteorites were obtained from the joint Belgian – Japanese search campaigns (2009-2010, 2010-2011 and 2012-2013) to the Balchenfjella and Nansen ice field areas, to the east and south of the Sør Rondane Mountains of East Antarctica. From a practical point of view, all Antarctic meteorites are first shipped and stored in Japan from where the Belgian share must come, after initial description, eventual splitting into equal parts (for most of the meteorite specimens > 50 g) and classification done at NIPR. As a result, meteorites come to Belgium in batches of about 20 to 100 specimens. Altogether 415 specimens have been transferred, while half of the meteorites < 50g are kept in Japan, according to the MoU. One particular specimen is the largest meteorite recovered by the Samba project, representing the 5th largest ever found on the East Antarctic shield, generously donated intact by NIPR (i.e. not shared in half) on the condition that it is used for museum exhibit. A permanent exhibit at the Natural Sciences museum in Brussels is now realised in the hall of 250 years of Natural Sciences.

Preliminary classification is done so that each specimen has a unique code and can be identified based on its external physical properties.

The steps following arrival of the specimens are related to their registration and usage: photographing and weighing the meteorites, completing the Darwin database; making polished thin sections in the dedicated laboratory or other preparations.

The (415) meteorites arrived according to their IG number (= definitive registration in the RBINS collection: IG32283:31; IG32340:24; IG32540:50; IG32628:51; IG32794:96; IG33222:42; IG33409:121) during the years 2012-2017. Definitive registration implies measurement of weight, photographs, individual file with at least provisional classification and introduction in Darwin database.

<u>Repository</u>: The storage room was entirely reorganized (Figure 2). In June 2015, the Antarctic meteorites have been classified by type in the repository (see the types considered in section 2.2.2 of this report). In order to improve the meteorite conservation, silica gel bags were put in the drawers containing the meteorites.

Since the temperature and relative humidity for the meteorites should be ideally between 18 ° C and 22 ° C and below 40 %, respectively, a dehumidification tool has been installed and is keeping the relative humidity to 30 %, while an air conditioning unit keeps the temperature close to the ideal temperature.

Temperature and humidity sensors (Niphargus, developed in-house; Burlet et al., 2015) have been installed to make sure the conservation conditions in the room are optimal. The data loggers are set so that a measurement is taken once an hour. The values recorded by

the data loggers are read, checked and classified at least once per trimester by the members of the WDP-section Geology. Reading the results on the Niphargus tool is performed with the Schrimp program. Results are kept on the collections.naturalsciences.be website.



Figure 2. Photos of (i) the repository at the RBINS, (ii) the samples classified in the drawers, (iii) the silicagel used in order to prevent humidity in the drawers, and (iv) the dehumidifier that has been installed in the room.

Laboratory: The laboratory for meteorite preparation was set up in a separate room to avoid contamination induced by the processing of other geological samples. A first laboratory was first set up in the building of the Geological Survey of Belgium. About four years ago, the lab was moved to a room (of about 10m<sup>2</sup>) on the 7th floor of the De Vestel building (Figure 3). The main aim of this moving was to decrease the distance from the repository to the lab, and thus limit contamination of the samples during transport. This new room, which was dedicated to X-ray diffraction before, needed some changes, with an in-depth cleaning/painting, new built-in furniture, workplan, electricity and lighting. The meteorite lab is exclusively dedicated to make polished section and chips of meteorites (needed for conservation/classification purposes, but also in case of loan) and general samples from meteorites response to specific requests. Beside basic tools to crush meteorite into smaller

fragments (hammer, chisel, metallic plate...), the lab was equipped with two kind of saws (Figure 4): a Lapidary Trim Saw (Lortone TS10) is used to cut middle-size and big samples. This saw can be used with two kinds of blades: one for stony meteorites and the other for iron meteorites. A small diamond wire saw (Well 5052), including sample holders, has been bought to cut smaller samples. This small saw allows a safe handling of the small meteorites during the sawing and avoids too much loss of material. For both saws, we use a coolant, mainly ethanol for Antarctic meteorites and demineralized water for desert or other meteorites. The type of coolant is chosen according to the type of meteorite but also to the type of analysis to perform. To prepare polished sections, the samples are embedded in resin (Araldite (DBF resin + HY 956 hardener - Escil) or Epoxy (EpoFix kit – Struers)) in an aluminium ring. The sections are polished by hand on different silicon carbide papers (from 80 to 2400 meshs). An ultrasonic bath (Leuchturn) is used after each step of polishing. Finally, a mechanical polisher (DP10 – Struers) with diamond water suspension of 9, 3 and 1  $\mu$ m grains size is used. A binocular allows checking the quality of polishing regularly during the polishing process. The lab has been equipped later on with a carver PROXXON (Micromot system; Figure 4) and a coring system (drilling machine PROXXON) TBH) to collect a part of a meteorite without crushing or sawing it. A magnetic susceptibility meter SM30 from ZH instruments was acquired in order to help for a quick classification of the meteorites (as described in different places of the Methodology and Results sections of the present report).

The manager of the lab (Thierry Leduc, statutory staff of the RBINS) and his assistant for this task (Thomas Goovaerts, geologist with a permanent contract at the RBINS) followed a special training at NIPR in December 2016 to improve the handling and processing of the samples at RBINS. This formation was of a great help for long-term curation of the meteorite collections. The report of this training at the NIPR is provided in Annex. In addition, a meteorite laboratory user manual (provided in Annex) was written and aims at describing how to handle and process properly meteorite samples in a dedicated laboratory.



Figure 3. General view of the meteorite lab at the RBINS

Loans: Sample deposit protocol and transfer agreement was completed in March 2013. A Scientific Loan Committee (SLC) was set up to decide on allocation of samples for research and exhibitions. It is composed of Philippe Claeys (VUB), Vinciane Debaille (ULB) and Marleen De Ceukelaire (RBINS). Its role and working method are described in the deposit agreement. Several meetings of the SLC were organized in the course of the project. From 2012 to 2019, 21 loans for 102 samples were registered.



Figure 4. Detailed photos of the Proxxon drilling device and of the saws. A polished section of a meteorite is also illustrated.

2.2.2. Accessibility on-line (identification, characterisation, classification, databasing)

#### Official identifications

The task of classifying new meteorites ran through the whole project. Most of the Antarctic meteorites brought to Belgium are being classified at NIPR. Classification at the RBINS involved systematic SEM/EDS and optical microscopy work, according to the methodology described before.

Identification/classification using Raman spectroscopy and magnetic susceptibility, which are quick and unexpensive methods (see methodology for more detailed information and references), was applied and led to a successful classification to numerous samples. For instance, visits of Pierre Rochette (June 2016) and Jérôme Gattacecca (July 2017) from the CEREGE (Aix-en-Provence, France) resulted in the classification of ~230 meteorites from the RBINS using this method.

Regarding their classification, meteorites can in general be divided into undifferentiated (chondrites) and differentiated meteorites (achondrites), which are further subdivided into classes, clans and groups (Figure 5).

#### Chondrites:

#### Carbonaceous chondrites:

CM (Mighei-type): These carbonaceous chondrites resemble their type specimen, the Mighei meteorite (the fall occurred in 1889 in the Ukraine). The CM chondrites originate from primitive water-rich asteroids, which formed during the early solar system. Their spectra closely match with near-Earth asteroids Ryugu and Bennu, which are the target of current sample return missions (Hayabusa-2 and OSIRIS-Rex).

CO (Ornans-type): The meteorites of this group are named after their type specimen Ornans, which's fall was observed in 1868 (in France). These carbonaceous chondrites are characterized by refractory inclusions and their relatively small chondrules compared to other chondrites.

CV (Vigarano-type): This group of carbonaceous chondrites is distinguished by large (mmsized) chondrules, many of which are surrounded by igneous rims, large refractory inclusions and abundant matrix (40 vol%); CV chondrites may be divided into oxidized and reduced subgroups.

CK (Karoonda-type). The prototype of these carbonaceous chondrites is the Karoonda meteorite, which fell in 1930 near the South Australian town Kardoonda. It is distinguished

by abundant fine-grained matrix (~75 vol%), a high degree of oxidation and mm-sized chondrules.

Ordinary chondrites:

H (high-iron): H-chondrites are a meteorite group belonging to the ordinary chondrites. They are characterized by a high siderophile element content and relatively small chondrules (0.3 mm).

L (low-iron): These ordinary chondrites exhibit a relatively low siderophile element content and moderate sized chondrules 0.7 mm). As parent body, the asteroids 433 Eros, 8 Flora or the Flora family as a whole were suggested.

LL (low-iron and low-metal): These meteorites contain about 19-22 wt% total iron, and only 1-3 wt% free metal and their chondrules are relatively large 0.9 mm) when compared to the H- and L-chondrites. They represent least common group of ordinary chondrites. Their parent body has not been identified yet, but their spectra match the spectra of S-type asteroids.

#### Achondrites:

Ureilite: Ureilites are a major group of primitive achondrites. They are carbon-bearing ultramafic rocks (mainly composed of olivine and pigeonite), and they contain interstitial carbon as graphite or microdiamonds.

Winonaite (Win): Winonaites are primitive achondrites, and therefore have lost their chondritic texture, but their mineralogy and composition are nearly chondritic. Compared to H chondrites, their mineral assemblage is more reduced.

Stony-iron meteorites:

Mesosiderite (Mes): Mesosiderites belong to the group of stony-iron meteorites, which are roughly composed of equal portions of silicates and FeNi-metal. Most of the mesosiderites are brecciated. Their parent body(ies) remain unknown, but it was suggested that Vesta may be the parent body of mesosiderite silicates.

HED meteorites (Howardites, Eucrites, Diogenites)

Eucrite: Eucrites are the most common of the achondrites and belong to the group of HED (Howardites, Eucrites, Diogenites) meteorites. HED meteorites are thought to have originated from asteroid Vesta, which is the second largest object in the asteroid belt. Eucrites can be subdivided into cumulate and basaltic eucrites: the cumulate eucrites are similar to terrestrial gabbros and are thought to have crystallized in depth, whereas the basaltic eucrites resemble terrestrial basalts and therefore, possibly formed near or on the surface of Vesta.

Diogenite: These meteorites are magmatic rocks and as such, they deliver important information about igneous processes of their parent body. Diogenites are strongly linked with the eucrites and howardites. Thus, they possibly also originate from asteroid Vesta, and it is believed that the diogenites form the lower crust of asteroid Vesta.



Figure 5. Simplified meteorite classification

#### Statistical information on the entire meteorite collection available at RBINS:

Total number of specimens in years 2012-2018: 415 Total weight: 20.278 kg (A12389 of ca. 18 kg in permanent display not included) Ordinary chondrites: 145 (77 H, 54 L, 14 LL) Iron meteorites: 2 Carbonaceous: 7 Others: 7 (2 diogenite, 2 ureilite, 1 mesosiderite, 1 winonaite, 1 angrite)

Unclassified: 254

#### Database

Web access to the collection of Antarctic meteorites is possible in 2 ways.

All Antarctic meteorites have been registered in the Darwin database by postdoc L. Pittarello, on schedule according to the BELAM programme and calendar. http://darwin.naturalsciences.be [/search/view/id/759438 as an example]. Login is required for access to detailed information of the samples. Both samples and conditions for loan can be accessed.

A more rapid online access to the Antarctic meteorite collection is via Mars website, collection management system allowing more straightforward access, as a result of data transfer by M. DeCeukelaire: http://mars.naturalsciences.be/geology/Meteorites/antarctic.

To access select the module geology on the start page, clicking further to meteorites and Antarctica. Via listings, particular meteorites can be selected, via an automated transfer to the Darwin system (example given in Figure 6). They are also registered in the MetSoc database.



Figure 6. The meteorite A09179 as appearing in the Darwin database

#### 2.2.3. Recognition of the curation center and quality control

To establish the RBINS as a curation center for Antarctic meteorites, a first step was set by the recognition of RBINS as a meteorite type specimen repository.

A full chain of curation operations and protocols was then defined to provide guidelines about how to register, store, classify and process meteorites (see sections 2.1 and 2.2 above). This aims at helping for all the curation tasks and at assisting RBINS in the daily management of the collection. In addition, the activities of the SLC and the loan regulations for external researchers (both general RBINS regulations and specific rules applicable to meteorites) ensure both the accessibility to the Belgian collection of meteorites for local and international researchers and the preservation of this precious collection.

An important step of the recognition process was the organization of the national BELAM project symposium on 14.11.2015 under the theme 'From Dinosaurs to Meteorites'. This symposium started from the connection between both themes by the end-Cretaceous Chixculub impact, dwelled on comparable research methodologies and on the importance for planetary sciences and finally brought the attention of the press and the public to BELSPO sponsored Antarctic research and the importance of meteorites for a natural sciences museum. Further, an international workshop "Curation of Antarctic Meteorites: Concluding workshop of the BELAM (Belgian Antarctic Meteorites) project" was organized in October 2016. This workshop tremendously helped to establish the RBINS as a curation center for Antarctic meteorites.

2.2.4. Establishing a research network, formation in the field of planetary sciences and collaborations dedicated to the curation of (Antarctic) meteorites

Partners of the project took part to international conferences (e.g., the Goldschmidt conference, the EGU General Assembly; cf. abstract list). This led to lots of new collaborations and research projects oriented toward Antarctic meteorites (that are kept at the RBINS). Part of the research involves PhD students, ensuring the formation of new researchers in the field of planetary sciences at the ULB-VUB.

In addition, a post-doc was recruited over a period of 4 years and shared her time between RBINS and VUB-ULB, thus constituting a link between the partners. The position was first held by Céline Martin and then by Lidia Pittarello. The postdoc researcher participated in the whole chain of Antarctic meteorite handling and study, from registration and identification of all specimens and their website publication – which were conducted at RBINS – to focused and high-level research on selected meteorite types– which was conducted at VUB and ULB. After their postdoc in Brussels for the BELAM project, both researchers found permanent positions at the American Museum of Natural History (C. Martin) and the Vienna NHM (L. Pittarello).

Finally, attendance to meetings dedicated to meteorites and meteorite curation (e.g. International Symposium on Antarctic Earth Science, NIPR Symposium on Antarctic meteorites, International Meeting of the Meteoritical Society; cf. abstract list) was particularly helpful to establish the RBINS as curation center at an international level and be part of a network of curators. The presence of many international experts at the international workshop "Curation of Antarctic Meteorites: Concluding workshop of the BELAM (Belgian Antarctic Meteorites) project" testifies for the efficiency of this network.

#### 3. Policy support

Considering the missions of BELSPO, which encompass the reinforcement of scientific activities at the Princess Elisabeth station and visibility of this research (BELSPO, 2008), the BELAM project can be considered as bringing long-lasting benefits to BELSPO. First, fruitful missions (with the Princess Elisabeth station as main base) were organized and lead to the collection of hundreds of meteorites that are now part of the Belgian heritage. Then, the ground-breaking research on the collected material enhances the visibility of BELSPO at international scale regarding scientific works in Antarctica. One of the most striking examples is given by a paper published very recently (March 2021) in the journal *Science Advances* (Press release provided in Figure 7). This publication focuses on a large meteoritic event over Antarctica and the key role played by BELSPO (through the BELAM project) in the organization of the mission in Antarctica is clearly mentioned.

In the future, more projects joining high level research in Belgium and missions in Antarctica will help in consolidating the role of BELSPO in Antarctica. This is ensured until at least 2024 thanks to the projects mentioned below (section 3.1).

Besides, the BELAM project mostly aims at a long-term management of a meteorite collection, enhanced access to the collections and efficient curation. This is one of the missions to fulfil by the RBINS under the umbrella of BELSPO, as stated in BELSPO's "Contrat d'administration 2016-2018" (BELSPO, 2015). In the course of the project, the partners of the project did their best to « secure » this curation and favour the access to the collection until 2024 thanks to the ongoing project (see below, section 3.1). This was also the main concern of the follow-up committee. However, efforts should be paid by the authorities to keep it – and even develop it – for the following years.

#### New study discovers ancient meteoritic impact over Antarctica 430,000 years ago

A research team of international space scientists, led by Dr Matthias van Ginneken from the University of Kent's School of Physical Sciences, has found new evidence of a lowaltitude meteoritic touchdown event reaching the Antarctic ice sheet 430,000 years ago.

Extra-terrestrial particles (condensation spherules) were found during the 2017-2018 Belgian Antarctic Meteorites (BELAM) expedition based at the Belgian Princess Elisabeth Antarctic station and funded by the Belgian Science Policy (Belspo). The particles were recovered on the summit of Walnumfjellet (WN) within the Sør Rondane Mountains, Queen Maud Land, East Antarctica, indicate an unusual touchdown event where a jet of melted and vaporised meteoritic material resulting from the atmospheric entry of an asteroid at least 100 m in size reached the surface at high velocity.

This type of explosion caused by a single-asteroid impact is described as intermediate, as it is larger than an airburst, but smaller than an impact cratering event.

The chondritic bulk major, trace element chemistry and high nickel content of the debris demonstrate the extraterrestrial nature of the recovered particles. Their unique oxygen isotopic signatures indicate that their interacted with oxygen derived from the Antarctic ice sheet during their formation in the impact plume.

The findings indicate an impact much more hazardous that the Tunguska and Chelyabinsk events over Russia in 1908 and 2013, respectively.

This research, published by *Science Advances*, guides an important discovery for the geological record where evidence of such events in scarce. This is primarily due to

the difficult in identifying and characterising impact particles.

The study highlights the importance of reassessing the threat of medium-sized asteroids, as it likely that similar touchdown events will produce similar particles. Such an event would be entirely destructive over a large area, corresponding to the area of interaction between the hot jet and the ground.

Dr van Ginneken said: 'To complete Earth's asteroid impact record, we recommend that future studies should focus on the identification of similar events on different targets, such as rocky or shallow oceanic basements, as the Antarctic ice sheet only covers 9% of Earth's land surface. Our research may also prove useful for the identification of these events in deep sea sediment cores and, if plume expansion reaches landmasses, the sedimentary record.

'While touchdown events may not threaten human activity if occurring over Antarctica, if it was to take place above a densely populated area, it would result in millions of casualties and severe damages over distances of up to hundreds of kilometres.'

The research paper 'A large meteoritic event over Antarctica ca. 430 ka ago inferred from chondritic spherules from the Sør Rondane Mountains' (M. van Ginneken - University of Kent; S. Goderis, F. Van Maldeghem, P. Claeys, B. Soens -Vrije Universiteit Brussel; N. Artemieva - Planetary Science Institute and Russian Academy of Sciences; V. Debaille -Université Libre de Bruxelles; S. Decrée - Belgian Geological Survey and Royal Belgian Institute of Natural Sciences; R. P. Harvey, K. Huwig - Case Western Reserve University; L. Hecht - Museum für Naturkunde Berlin and Freie Universit.t Berlin; F. E. D. Kaufmann - Museum für Naturkunde Berlin; S. Yang, M. Humayun - National High Magnetic Field Laboratory and Department of Earth; M. J. Genge - Imperial College London) is published by *Science Advances*.



Figure 7. Press release – paper "A large meteoritic event over Antarctica" Van Ginneken et al. (2021) – *Science Advances.* Pictures credited to Mark Garlick / markgarlick.com (left side) and Scott Peterson / micro-meteorites.com (right side)

#### 3.1. Long-term continuation of the curation: New projects funded

The most direct way for a federal scientific institute to get high-level postdoc worker is through the BRAIN programme (Belgian Research Action through Interdisciplinary Networks). Taking into account the recommendations of the follow-up committee, three BRAIN projects were formulated and approved for funding.

The first project, introduced under Thematic axis 6: Management of collections by RBINS-ULB-VUB, is entitled 'Antarctic meteorites curation, digitalization and conservation' (AMUNDSEN; <u>http://www.belspo.be/belspo/brain-be/themes\_6\_Collect\_en.stm#AMUNDSEN; 15/03/2016-28/02/2022</u>). This project is dedicated to the conservation, classification, valorisation and digitalization of meteorites at the RBINS with the goal to improve the maintenance of this fragile collection, develop best practice meteorite curation protocols, provide the most appropriate sampling procedure and stimulate and facilitate the scientific usage of the collection by the international research community.

Three multidisciplinary approaches are proposed: (1) The first part of this project relates to the most troubling problems of meteorite conservation: their rapid alteration, which even in the case of this freshly collected collection, is already observed within some of the specimens. To better constrain the rate of this weathering process and optimize the conservation conditions, a set of alteration/oxidation experiments are planned, with the aim to propose possible remediation processes (2) We aim to provide on-line broaden access to rare and unique meteorite by digitizing thin sections of the most outstanding samples (achondrites and specific types of ordinary chondrites), providing directly online a navigable images obtained with the optical microscope and coupled to a detailed chemical map of the area at high-resolution, as produced by micro-X-ray Fluorescence. Such digitized thin sections will contribute to the study of RBINS meteorites, avoiding excessive handling (sawing, etc), and will help requesters in their sample selection. (3) As a curation center recognized by the Meteoritical Society, the RBINS is committed to provide the best curation procedures possible. We plan to improve and advance the existing meteorite classification procedure already in use, e.g. by using working with thick sections instead of thin sections, when possible and possibly testing the use of Raman and micro-X-Ray Fluorescence ( $\mu$ -XRF) procedures.

The second project, introduced under Thematic axis 2, is entitled 'Belgian Antarctic Meteorites and Micrometeorites to document solar system formation and evolution' (**BAMM!**; belspo.be/belspo/brain-be/projects/BAMM\_en.pdf; 01/01/2017 – 15/04/2022). This novel BRAINS project, gathering the same partners, builds on and expands the assembled expertise, and centers on a number of highly promising, but previously unexplored research opportunities provided by this valuable set of newly recovered extraterrestrial samples. Antarctic (micro)meteorites constitute an enormous volume of extraterrestrial material that was preserved under excellent conditions thanks to a dry and cold climate. Using the meteorites and micrometeorites recently collected in the Sør Rondane Mountains of Antarctica, the following two complementary approaches further constrain our understanding of the formation and evolution of solar system materials: (1)A detailed study of micrometeorites and their igneous textures to better document their parent body precursors (possibly not sampled by larger meteorites), quantify the continuum between unmelted and fully molten objects, and further constrain the effects of rapid melting, melt extraction and silicate-metal segregation on the petrological, chemical and

isotopic characteristics of the precursor materials. (2) A precise characterization of the isotope anomalies existing in bulk meteorite samples, and their counterparts in the constituent mineralogical phases measured by in situ mass spectrometry to better understand the presence and destruction of nucleosynthetic anomaly carrier phases during nebular and planetary processes.

In addition, the BAMM! project further expands the Belgian Antarctic meteorite collection and encourages a reliable, long-term protective curation program of Antarctic meteorites at the RBINS, boosting at the same its position as a key Antarctic (micro)meteorite curation center in Europe. The Belgian meteorite classification expertise will be expanded, and implemented not only to Antarctic meteorites, but also to non-Antarctic samples. Last but not least, this project supports the preservation and, through its research output, the valorization of the Belgium museum collections and national heritage.

A third project, 'Tracing differentiation processes through siderophile elements, from meteorites to giant ore deposits' (**DESIRED**; belspo.be/belspo/brain2-be/projects/DESIRED\_E.pdf;15/12/2019 - 15/03/2024) aims at expanding and improving the efficient curation of all Antarctic meteorites at the RBINS. Thanks to the two meteorite recovery missions planned within the framework of this project (2021-2022 and 2022-2023), the size of the Antarctic RBINS meteorite collection (>1300 specimens to date) is expected to increase substantially, ensuring sufficiently large meteorite masses needed to apply high-precision isotopic methods, as planned in the scientific tasks of the project.

#### 3.2. Long-term continuation of the curation: Nomination of a curator

Vinciane Debaille was appointed on 15.10.2014 by RBINS as scientific collaborator – scientific curator to represent RBINS at international meetings of curators.

#### 4. Dissemination and valorization

Besides the major progress in terms of curation and conservation that are extensively detailed in the "2.2 Results" section of this report, it is worth mentioning that a lot of work was done regarding the diffusion and the valorization of the results. Apart from the scientific outcome, which can be evaluated thanks to the peer-reviewed papers and conference abstracts listed in the "5. Publication" section of this report, one can mention (i) the exhibit of an outstanding meteorite found in Antarctica coupled to a national meeting in 2014, and (ii) the organization of an international workshop at the RBINS in 2016.

#### 4.1. National meeting and exhibit 2014

The national BELAM project meeting on 14.11.2014 consisted of a symposium and inauguration of a meteorite display (Figure 8). The symposium on Antarctic meteorites and their curation in Belgium "From dinosaurs to meteorites" attracted 54 registered participants from most universities and the federal research institutes from the poles nature and space for 9 presentations including a keynote lecture by D. Herwartz (Univ. Köln). The inauguration of a permanent Antarctic meteorite display attracted the press. This 18 kg meteorite is a proof of the successful collaboration between Japanese and Belgian researchers. It was found by the team of 5 Belgian scientists from the VUB and ULB, and 3 Japanese researchers from the NIPR during the 2012-2013 expedition to the Nansen Ice Field in Antarctica. This field mission financed by BELSPO and NIPR was a real success. The team discovered 425 meteorites that are currently being inventoried. The meteorite that attracted most attention was the one of 18 kilograms. It concerns the largest specimen discovered in 25 years in East Antarctica. It is also the fifth heaviest ever discovered in this part of Antarctica of the 16,000 specimens alreadv found. [http://we.vub.ac.be/dntk/nl/node/220]

This specimen is therefore without doubt exceptional because of its size. The meteorite belongs to the "chondrite" type that is most common on Earth. Therefore, this exceptional specimen was not cut and reserved for exhibits. Thanks to the benevolent cooperation of NIPR this specimen was shipped to Belgium and is allowed to stay here on condition that it is exhibited. A temporary exhibit was made upon arrival, and the specimen has since then integrated the permanent exhibit of the RBINS.



Figure 8. Pictures of the national BELAM project meeting on 14.11.2014

#### 4.2. International workshop

On October 3-4 2016 the international workshop "Curation of Antarctic Meteorites: Concluding workshop of the BELAM (Belgian Antarctic Meteorites) project" was organized at the Royal Belgian Institute of Natural Sciences (Figure 9). The rationale of the workshop was the following: "In the frame of the BELAM project, funded by the Belgian Science Policy (Belspo), a new curation facility dedicated to Antarctic meteorite was installed at the Royal Belgian Institute of Natural Sciences in Brussels. As the project is now finishing, we would like to present those facilities to the scientific community, as well as the scientific results obtained so far on the Belgian Antarctic collection. In addition, we would like to take the opportunity of this meeting for gathering worldwide experts in curation, in order to share experience and best practices." This workshop consisted of a symposium with 10 talks, a visit of the meteorite collection repository and meteorite laboratory and an afternoon of roundtable discussions. Eight international experts were invited: Cari Corrigan (USA-Smithsonian Institute); Luigi Folco (Italy-University of Pisa); Jérome Gattacceca (France-CEREGE); Christian Koeberl (Austria-Natural History Museum Wien); Kevin Righter (USA-NASA-JSC); Caroline Smith (UK-Natural History Museum of London); Akira Yamaguchi (Japan-National Institute of Polar Research); Brigitte Zanda (France-Musée d'Histoires Naturelles de Paris). These experts gave presentations about conservation/curation in their research institutes. They also provided advice during roundtable discussions. A summary of the BELAM project was given through three introductive talks: Conservation and curation at the RBINS (Sophie Decrée, Marleen Deceukelaire, Vinciane Debaille); Report of the Belgian Antarctic missions (Steven Goderis); Belgian scientific research dedicated to Antarctic meteorites (Vinciane Debaille). Thirty-seven registered participants attended this workshop.



### Curation of Antarctic Meteorite Workshop



AMGC is co-organising an international workshop of the curation of Antarctic meteorites at the Royal Institute of Natural Sciences in Brussels (RBINS). This is an opportunity to present the new ~ 1300 meteorites recently collected by joint Belgian and Japanese expeditions (2009 - 2013) in the surroundings of Antarctic Station Princess Elisabeth, in the framework of the SAMBA and BELAM projects financed by BELSPO (more info); for SAMBA project report, <u>click here</u>.

> Analytical.

Environmental and

> Chemistry Department

Geo- Chemistry

> Vakgroep Chemie

With support of the VUB and ULB, these meteorites are curated at the RBINS, which now possesses one of the

largest Antarctic meteorite collection in Europe. The goal of this workshop is to promote the study of these unique samples by the international community.

More info on the workshop: <u>http://gtime.ulb.ac.be/BELAM.html</u>

Figure 9. Announcement of the international workshop "Curation of Antarctic Meteorites: Concluding workshop of the BELAM (Belgian Antarctic Meteorites) project" organized on October 3-4 2016, as appearing on the website of the VUB

#### 4.3. Research activities and scientific outcome

Within the frame of the BELAM project, several research axes have been investigated, in order to valorize the Antarctic Belgian collection and the meteorite collection at the RBINS in general. Notably, we have investigated the atmospheric entry of meteorites by

the formation of fusion crust, the impact process by shock features and cratering process on Earth. We have also investigated the large, peculiar Mont Dieu meteorite that was acquired by the RBINS and is now in the permanent exhibit. During the BELAM Antarctic campaign, we also took the chance to investigate the relationship between the number of meteorites and the ice flux, and several micrometeorites were collected, leading to several publications using both micrometeorites and meteorites. Several publications related to the BELAM project are still in preparation, with the support of the following projects such as the AMUNDSEN project.

#### 5. Publication

# **5.1.** Peer review (all publications are available online and provided as annexes) 2013

• Martin, C., Debaille, V., Lanari, P., Goderis, S., Vandendael, I., Vanhaecke, F., Vidal, O., and Claeys, P., 2013. REE and Hf distribution among mineral phases in the CV-CK clan: A way to explain present-day Hf isotopic variations in chondrites. Geochimica et Cosmochimica Acta 120 (2013) 496-513.

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# Annex 1. Publications (only the first page is presented, the papers are provided as separate files)



Geochimica et Cosmochimica Acta

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# High spatial resolution geochemistry and textural characteristics of 'microtektite' glass spherules in proximal Cretaceous–Paleogene sections: Insights into glass alteration patterns and precursor melt lithologies

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### Abstract

Using laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS), we have conducted spatially resolved trace element analysis on fresh, unaltered microtektite glasses linked to the Cretaceous-Paleogene (K-Pg) boundary Chicxulub crater and on their surrounding alteration phases. This unique approach offers the opportunity to study in situ and at high spatial resolution both the mixing of different target lithologies and the variation of the major and trace element budget during the alteration process. In addition, two-dimensional element distribution maps reveal important geochemical information beyond the capabilities of single spot laser drilling. Glasses from two localities in opposite quadrants from the source crater were studied. At the Beloc locality (Haiti), the glass population is dominated by the presence of yellow high-Ca glass and black andesitic glass formed by admixture of carbonate/dolomite/anhydrite platform lithologies with crystalline basement. These glasses alter according to the well-established hydration-palagonitization model postulated for mafic volcanic glasses. REEs become progressively leached from the glass to below the detection limit for the applied spot size, while immobile Zr, Hf, Nb, and Ta passively accumulate in the process exhibiting both inter-element ratios and absolute concentrations similar to those for the original glass. In contrast, The Arroyo El Mimbral locality (NE Mexico) is characterized by abundant green glass fragments high in Si, Al and alkalis, and low in Mg, Ca, Fe. Low Si black glass is less abundant though similar in composition to the black glass variety at Beloc. The alteration pattern of high-Si, Al green glass at the Mimbral locality is more complex, including numerous competing reaction processes (ion-exchange, hydration, dissolution, and secondary mineral precipitation) generally controlled by the pH and composition of the surrounding fluid. All green, high-Si, Al glasses are hydrated and variably enriched in Sr, Ba, and Cs, indicating preferred adsorption from seawater during hydration. Despite the onset of ion-exchange reactions, which only seem to have affected the alkalis, the trace element composition of the green high-Si, Al glass is still largely representative of the original melt composition. Refining the geochemical signature of (altered) melt lithologies may advance our current understanding of glass stability in the natural environment and provide insight into the origin and emplacement of ejecta material during crater formation. © 2014 Elsevier Ltd. All rights reserved.

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# JAAS





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# Development of an isolation procedure and MC-ICP-MS measurement protocol for the study of stable isotope ratio variations of nickel<sup>†</sup>

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Variations in the isotopic composition of Ni resulting from natural mass-dependent processes in terrestrial or extraterrestrial conditions, inhomogeneous distribution of nucleosynthetic components and/or ingrowth from radioactive parent nuclides, help us to further understand the early formation history of Solar System materials and the nature of the processes these materials subsequently experienced. In studies of Ni isotope systematics, mass-dependent variations in the isotopic composition of Ni are often bypassed because of the challenges associated with the sample preparation. At the level of natural variation studied, Ni isotope ratio measurements are extremely sensitive to spectral interference, artificial on-column isotope fractionation and possibly even to the mass bias correction model applied. To adequately address these complications, an isolation procedure and measurement protocol relying on multi-collector ICP-mass spectrometry (MC-ICP-MS) have been designed and validated in this work. The overall reproducibility obtained based on repeated measurement of a Sigma-Aldrich high-purity Ni standard is 0.036‰, 0.049% 0.078\% and 0.53% for  $\delta^{60/58}$ Ni,  $\delta^{61/58}$ Ni,  $\delta^{62/58}$ Ni and  $\delta^{64/58}$ Ni, respectively (n = 14; 2 SD). Nickel isotope ratio variations have been studied in a set of iron meteorites and geological reference materials, and the results obtained, except for those suffering from an elevated <sup>64</sup>Zn background, show good agreement with the available literature data. By using the flexible generalized power law with a variable discrimination exponent and the three-isotope method, the processes underlying natural mass fractionation of Ni for terrestrial reference materials were found to have a mixed equilibrium/kinetic nature. Mass-dependent Ni fractionation was observed between sample fractions of the Canyon Diablo iron meteorite, and the extracted fractionation factor  $\beta$  corresponds to isotope partitioning following the power law.

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# Introduction

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In the process of the formation and evolution of meteorite parent bodies, the isotopic composition of various transition metals (*e.g.*, Fe, Cr, Ni, Cu, Zn) has been established to result from multiple superimposed processes, including nucleosynthesis in different stellar environments, (incomplete) mixing of the presolar carriers of these nucleosynthetic components in the crescent solar nebula, isotope fractionation during differentiation processes, ingrowth through radiogenic decay, and perturbation of the isotopic composition due to spallation by cosmic rays.<sup>1,2</sup>

In this context, isotopic analysis of Ni shows great potential for geo- and planetary applications. As an abundant element in

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the Solar System, Ni is present in sufficiently high concentrations in most meteorites. Ni, mostly partitioned into the metalsilicate-troilite system, experiences mass-dependent fractionation of its isotopes during evaporation/condensation, core formation, and magmatic processes. Other transition metals that have recently been studied in this context include Fe,3 Cr,4 Mo,<sup>5</sup> Zn<sup>6</sup> and W.<sup>7</sup> In contrast to other elements, Ni only occurs in a single stable oxidation state and surrounding coordination only, apart from its neutral metal state. Hence, redox-controlled mass-dependent fractionation processes do not affect the isotopic composition of Ni, restricting the mechanism underlying mass-dependent fractionation to isotopic exchange between phases.8-11 A comparative study of the effect of massdependent isotope fractionation observed for Ni in genetically related meteorite fractions could not only show a correlation with the concentration of particular major elements (e.g., P, S), but also with the cooling rate of the meteorite and/or the isotopic composition of other fractionated elements (e.g., O, Cr).

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Geochimica et Cosmochimica Acta

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# Effect of parent body evolution on equilibrium and kinetic isotope fractionation: a combined Ni and Fe isotope study of iron and stony-iron meteorites

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#### Abstract

Various iron and stony-iron meteorites have been characterized for their Ni and Fe isotopic compositions using multicollector inductively coupled plasma-mass spectrometry (MC-ICP-MS) after sample digestion and chromatographic separation of the target elements in an attempt to further constrain the planetary differentiation processes that shifted these isotope ratios and to shed light on the formational history and evolution of selected achondrite parent body asteroids. Emphasis was placed on spatially resolved isotopic analysis of iron meteorites, known to be inhomogeneous at the µm to mm scale, and on the isotopic characterization of adjacent metal and silicate phases in main group pallasites (PMG), mesosiderites, and the IIE and IAB complex silicate-bearing iron meteorites. In a 3-isotope plot of <sup>60/58</sup>Ni versus <sup>62/58</sup>Ni, the slope of the best-fitting straight line through the laterally resolved Ni isotope ratio data for iron meteorites reveals kinetically controlled isotope fractionation ( $\beta_{exper} = 1.981 \pm 0.039, 1$  SD), predominantly resulting from sub-solidus diffusion (with the fractionation exponent  $\beta$  connecting the isotope fractionation factors, as  $\alpha_{62/58} = \alpha_{60/58}^{\beta}$ ). The observed relation between  $\delta^{56/54}$ Fe and Ir concentration in the metal fractions of PMGs and in IIIAB iron meteorites indicates a dependence of the bulk Fe isotopic composition on the fractional crystallization of an asteroidal metal core. No such fractional crystallization trends were found for the corresponding Ni isotope ratios or for other iron meteorite groups, such as the IIABs. In the case of the IIE and IAB silicatebearing iron meteorites, the Fe and Ni isotopic signatures potentially reflect the influence of impact processes, as the degree of diffusion-controlled Ni isotope fractionation is closer to that of Fe compared to what is observed for magmatic iron meteorite types. Between the metal and olivine counterparts of pallasites, the Fe and Ni isotopic compositions show clearly resolvable differences, similar in magnitude but opposite in sign ( $\Delta^{56/54}$ Fe<sub>met-oliv</sub> of +0.178 ± 0.092% and  $\Delta^{60/58}$ Ni<sub>met-oliv</sub> of -0.212 ± 0.082‰, 2SD). As such, the heavier Fe isotope ratios for the metal ( $\delta^{56/54}$ Fe = +0.023‰ to +0.247‰) and lighter values for the corresponding olivines ( $\delta^{56/54}$ Fe = -0.155‰ to -0.075‰) are interpreted to reflect later-stage Fe isotopic re-equilibration between these phases, rather than a pristine record of mantle-core differentiation. In the case of mesosiderites, the similarly lighter Ni and Fe isotopic signatures found for the silicate phase (-0.149% to +0.023% for  $\delta^{60/58}$ Ni, -0.214% to -0.149%for  $\delta^{56/54}$ Fe) compared to the metal phase (+0.168% to +0.191% for  $\delta^{60/58}$ Ni, +0.018% to +0.120% for  $\delta^{56/54}$ Fe) likely result from Fe and Ni diffusion. Overall, the Fe and Ni isotopic compositions of iron-rich meteorites reflect multiple, often super-

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# Thermal equilibration of iron meteorite and pallasite parent bodies recorded at the mineral scale by Fe and Ni isotope systematics

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#### Abstract

In this work, a femtosecond laser ablation (LA) system coupled to a multi-collector inductively coupled plasma-mass spectrometer (fs-LA-MC-ICP-MS) was used to obtain laterally resolved (30-80 µm), high-precision combined Ni and Fe stable isotope ratio data for a variety of mineral phases (olivine, kamacite, taenite, schreibersite and troilite) composing main group pallasites (PMG) and iron meteorites. The stable isotopic signatures of Fe and Ni at the mineral scale, in combination with the factors governing the kinetic or equilibrium isotope fractionation processes, are used to interpret the thermal histories of small differentiated asteroidal bodies. As Fe isotopic zoning is only barely resolvable within the internal precision level of the isotope ratio measurements within a single olivine in Esquel PMG, the isotopically lighter olivine core relative to the rim  $(\Delta^{56/54} Fe_{rim-core} = 0.059\%)$  suggests that the olivines were largely thermally equilibrated. The observed hint of an isotopic and concentration gradient for Fe of crudely similar width is interpreted here to reflect Fe loss from olivine in the process of partial reduction of the olivine rim. The ranges of the determined Fe and Ni isotopic signatures of troilite ( $\delta^{56/54}$ Fe of -0.66 to -0.09%) and schreibersite ( $\delta^{56/54}$ Fe of -0.48 to -0.09%, and  $\delta^{62/60}$ Ni of -0.64 to +0.29%) may result from thermal equilibration. Schreibersite and troilite likely remained in equilibrium with their enclosing metal to temperatures significantly below their point of crystallization. The Ni isotopic signatures of bulk metal and schreibersite correlate negatively, with isotopically lighter Ni in the metal of PMGs and isotopically heavier Ni in the metal of the iron meteorites analyzed. As such, the light Ni isotopic signatures previously observed in PMG metal relative to chondrites may not result from heterogeneity in the Solar Nebula, but rather reflect fractionation in the metal-schreibersite system. Comparison between the isotope ratio profiles of Fe and Ni determined across kamacite-taenite interfaces ( $\Delta^{56/54}$ Fe<sub>kam-tae</sub> = -0.51 to -0.69% and  $\Delta^{62/60}$ Ni<sub>kam-tae</sub> = +1.59 to +2.50%) and theoretical taenite sub-solidus diffusive isotopic zoning broadly constrain the cooling rates of Esquel, CMS 04071 PMGs and Udei Station IAB to between ~25 and 500 °C/Myr.

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Keywords: Pallasites; Iron meteorites; Fe and Ni stable isotope ratios; Core formation; Schreibersite

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# Evidence for a chondritic impactor, evaporation-condensation effects and melting of the Precambrian basement beneath the 'target' Deccan basalts at Lonar crater, India

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#### Abstract

The  $\sim$ 1.88 km diameter Lonar impact crater formed  $\sim$ 570 ka ago and is an almost circular depression hosted entirely in the Poladpur suite of the ~65 Ma old basalts of the Deccan Traps. To understand the effects of impact cratering on basaltic targets, commonly found on the surfaces of inner Solar System planetary bodies, major and trace element concentrations as well as Nd and Sr isotopic compositions were determined on a suite of selected samples composed of: basalts, a red bole sample, which is a product of basalt alteration, impact breccia, and impact glasses, either in the form of spherules (<1 mm in diameter) or non-spherical impact glasses (>1 mm and <1 cm). These data include the first highly siderophile element (HSE) concentrations for the Lonar spherules. The chemical index of alteration (CIA) values for the basalts and impact breccia (36.4–42.7) are low while the red bole sample shows a high CIA value (55.6 in the acid-leached sample), consistent with its origin by aqueous alteration of the basalts. The Lonar spherules are classified into two main groups based on their CIA values. Most spherules show low CIA values (Group 1: 34.7-40.5) overlapping with the basalts and impact breccia, while seven spherules show significantly higher CIA values (Group 2: >43.0). The Group 1 spherules are further subdivided into Groups la and lb, with Group la spherules showing higher Ni and mostly higher Cr compared to the Group lb spherules. Iridium and Cr concentrations of the spherules are consistent with the admixture of 1-8 wt% of a chondritic impactor to the basaltic target rocks. The impactor contribution is most prominent in the Group 1a and Group 2 spherules, which show higher Ni/Co, Ni/Cr and Cr/Co ratios compared to the target basalts. In contrast, the Group 1b spherules show major and trace element compositions that overlap with those of the impact breccia and are characterized by high EF<sub>Th</sub> (Enrichment Factor for Th defined as the Nb-normalized concentration of Th relative to that of the average basalt) as well as fractionated La/Sm(N), and higher large ion lithophile element (LILE) concentrations compared to the basalts. The relatively more radiogenic Sr and less radiogenic Nd isotopic composition of the impact breccia and non-spherical impact glasses compared to the target basalts are consistent with melting and mixing of the Precambrian basement beneath the Deccan basalt with up to 15 wt% contribution of the basement to these samples. Variations in the moderately siderophile element (MSE) concentration ratios of the impact breccia as well as all the spherules are best explained by contributions from three components - a chondritic impactor, the basaltic target rocks at Lonar and the basement underlying the Deccan basalts. The large variations in concentrations of

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# The role of phosphates for the Lu-Hf chronology of meteorites

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## A R T I C L E I N F O

# ABSTRACT

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Keywords: chondrites phosphates Lu–Hf chronology diffusion The <sup>176</sup>Lu-<sup>176</sup>Hf isotopic system is widely used for dating and tracing cosmochemical and geological processes, but still suffers from two uncertainties. First, Lu-Hf isochrons for some early Solar System materials have excess slope of unknown origin that should not be expected for meteorites with ages precisely determined with other isotopic chronometers. This observation translates to an apparent Lu decay constant higher than the one calculated by comparing ages obtained with various dating methods on terrestrial samples. Second, unlike the well constrained Sm/Nd value (to within 2%) for the chondritic uniform reservoir (CHUR), the Lu/Hf ratios in chondrites vary up to 18% when considering all chondrites, adding uncertainty to the Lu/Hf CHUR value. In order to better understand the Lu-Hf systematics of chondrites, we analyzed mineral fractions from the Richardton H5 chondrite to construct an internal Lu-Hf isochron, and set up a numerical model to investigate the effect of preferential diffusion of Lu compared to Hf from phosphate, the phase with the highest Lu-Hf ratio in chondrites, to other minerals. Compared to in non-phosphate, the phase with the highest Lu–Hr fatto in Condities, to other minerals. The isochron yields an age of 4647 ±210 million years (Myr) using the accepted  $^{176}$ Lu decay constant of 1.867 ±0.008 × 10<sup>-11</sup> yr<sup>-1</sup>. Combining this study with the phosphate fractions measured in a previous study yields a slope of 0.08855 ±0.00072, translating to a  $^{176}$ Lu decay constant of 1.862 ±0.016 × 10<sup>-11</sup> yr<sup>-1</sup> using the Pb–Pb age previously obtained, in agreement with the accepted value. The large yr<sup>-1</sup> using the Pb-Pb age previously obtained, in agreement with the accepted value. The large variation of the Lu/Hf phosphates combined with observations in the present study identify phosphates as the key in perturbing Lu-Hf dating and generating the isochron slope discrepancy. This is critical as apatite has substantially higher diffusion rates of rare earth elements than most silicate minerals that comprise stony meteorites. Results of numerical modeling depending of temperature peak, size of the grains and duration of the metamorphic event, show that diffusion processes in phosphate can produce an apparently older Lu-Hf isochron, while this effect will remain negligible in perturbing the Sm-Nd chronology. Our results suggest that only type 3 chondrites with the lowest metamorphic grade and large minerals with minimal diffusive effects are suitable for determination of the Lu-Hf CHUR values and the Lu decay constant respectively.

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### 1. Introduction

The <sup>176</sup>Lu–<sup>176</sup>Hf isotopic system is extensively used for dating cosmochemical and geological processes, and for studying planetary evolution. However, two uncertainties in the Lu–Hf systematic still need to be explained. First, Lu–Hf isochrons for many meteorites have steeper slopes than can be expected using the well-established decay rate of <sup>176</sup>Lu in terrestrial rocks. Isochron comparisons performed on terrestrial and extraterrestrial geological objects give similar values for  $\lambda^{176}$ Lu ("terrestrial" average of ~1.864–1.867 × 10<sup>-11</sup> yr<sup>-1</sup>) (Amelin, 2005; Blichert-Toft and Albarède, 1997; Grinyer et al., 2003; Nir-El and Haquin, 2003; Nir-El and Lavi, 1998; Patchett et al., 2004; Scherer et al., 2001), whereas the isochrons obtained exclusively on some chondrites and achondrites indicate a less straightforward message. Some studies have proposed a  $\lambda^{176}$ Lu slightly higher than observed on Earth ("meteoritic" average of ~1.95 × 10<sup>-11</sup> yr<sup>-1</sup>) (Bizzarro et al., 2003, 2012; Blichert-Toft et al., 2002; Patchett and Tatsumoto, 1980; Thrane et al., 2010) while other studies have found  $\lambda^{176}$ Lu similar to the "terrestrial value" either on mineral separates (Bast et al., 2017; Sanborn et al., 2015) or bulk rock isochrons (Bouvier et al., 2015). Direct counting experiments have, so far, not been particularly useful for understanding the apparent decay constant

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ASTROBIOLOGY



# PLANET TOPERS: Planets, Tracing the Transfer, Origin, Preservation, and Evolution of their ReservoirS

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**Abstract** The Interuniversity Attraction Pole (IAP) 'PLANET TOPERS' (Planets: Tracing the Transfer, Origin, Preservation, and Evolution of their Reservoirs) addresses the fundamental understanding of the thermal and compositional evolution of the different reservoirs of planetary bodies (core, mantle, crust, atmosphere, hydrosphere, cryosphere, and space)

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# s-Process Os isotope enrichment in ureilites by planetary processing

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## A R T I C L E I N F O

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### ABSTRACT

Ubiquitous nucleosynthetic isotope anomalies relative to the terrestrial isotopic composition in Mo, Ru, and other elements are known from both bulk chondrites and differentiated meteorites, but Os isotope ratios reported from such meteorites have been found to be indistinguishable from the terrestrial value. The carriers of s- and r-process Os must thus have been homogeneously distributed in the solar nebula. As large Os isotope anomalies are known from acid leachates and residues of primitive chondrites, the constant relative proportions of presolar s- and r-process carriers in such chondrites must have been maintained during nebular processes. It has long been assumed that partial melting of primitive chondrites would homogenize the isotopic heterogeneity carried by presolar grains. Here, ureilites, carbon-rich ultramafic achondrites dominantly composed of olivine and low-Ca pyroxene, are shown to be the first differentiated bulk Solar System materials for which nucleosynthetic Os isotope anomalies have been identified. These anomalies consist of enrichment in s-process Os heterogeneously distributed in different ureilites. Given the observed homogeneity of Os isotopes in all types of primitive chondrites, this Os isotope variability among ureilites must have been caused by selective removal of s-process-poor Os host phases, probably metal, during rapid localized melting on the ureilite parent body. While Mo and Ru isotope anomalies for all meteorites measured so far exhibit s-process deficits relative to the Earth, the opposite holds for the Os isotope anomalies in ureilites reported here. This might indicate that the Earth preferentially accreted olivine-rich restites and inherited a s-process excess relative to smaller meteorite bodies, consistent with Earth's high Mg/Si ratio and enrichment of s-process nuclides in Mo, Ru, and Nd isotopes. Our new Os isotope results imply that caution must be used when applying nucleosynthetic isotope anomalies as provenance indicators between different classes of meteorite

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#### 1. Introduction

Isotopic anomalies are defined as deviations from terrestrial or bulk solar isotopic abundances that have resulted from processes other than radioactive decay or mass-dependent fractionation. Presolar grains, widespread in unequilibrated chondrites, are enriched in material derived from distinct nucleosynthetic environments (Zinner, 1998). Small but resolvable isotopic anomalies in bulk meteorites, often most prominent in primitive carbonaceous chondrites, are present for various elements including Ti, Cr, Ni, Zr, Mo, Ru, Ba, Nd, and Sm (e.g., Akram et al., 2015; Burkhardt et al., 2011, 2012; Carlson et al., 2007; Chen et al., 2010;

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http://dx.doi.org/10.1016/j.epsl.2015.09.021 0012-821X/© 2015 Elsevier B.V. All rights reserved. Dauphas et al., 2002; Qin et al., 2010; Regelous et al., 2008; Trinquier et al., 2009; Yin et al., 2002). Uniform isotopic compositions in bulk chondrites are reported for other elements, most prominently Os and Hf (Brandon et al., 2005; Sprung et al., 2010; van Acken et al., 2011; Walker, 2012; Wittig et al., 2013; Yokoyama et al., 2007). The presence of isotope anomalies in bulk planetary materials, such as chondrites and achondrites, is explained by a variety of mechanisms that resulted in isotope heterogeneity at the planetesimal scale. These include, for example, inheritance from a heterogeneous molecular cloud core, formation of localized heterogeneities due to differential physical sorting of dust grains (e.g., metals, silicates and sulfides), late supernova injection, or selective destruction of thermally labile presolar carrier phases in the nebula (e.g., Andreasen and Sharma, 2007; Burkhardt et al., 2011; Carlson et al., 2007; Chen et al., 2010; Dauphas et al., 2002; Qin et al., 2008; Trinquier et al., 2009). Alternatively, secondary processes, such as aqueous alteration have been suggested to selec-

# JAAS

# **TUTORIAL REVIEW**



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# Isotopes in cosmochemistry: recipe for a Solar System

Steven Goderis,\*<sup>ab</sup> Ramananda Chakrabarti,<sup>c</sup> Vinciane Debaille<sup>d</sup> and János Kodolányi<sup>e</sup>

Extreme isotopic variations among extraterrestrial materials provide great insights into the origin and evolution of the Solar System. In this tutorial review, we summarize how the measurement of isotope ratios can expand our knowledge of the processes that took place before and during the formation of our Solar System and its subsequent early evolution. The continuous improvement of mass spectrometers with high precision and increased spatial resolution, including secondary ion mass spectrometry (SIMS), thermal ionization mass spectrometry (TIMS) and multi collector-inductively coupled plasma-mass spectrometry (MC-ICP-MS), along with the ever growing amounts of available extraterrestrial samples have significantly increased the temporal and spatial constraints on the sequence of events that took place since and before the formation of the first Solar System condensates (i.e., Ca-Al-rich inclusions). Grains sampling distinct stellar environments with a wide range of isotopic compositions were admixed to, but possibly not fully homogenized in, the Sun's parent molecular cloud or the nascent Solar System. Before, during and after accretion of the nebula, as well as the formation and subsequent evolution of planetesimals and planets, chemical and physical fractionation processes irrevocably changed the chemical and isotopic compositions of all Solar System bodies. Since the formation of the first Solar System minerals and rocks 4.568 Gyr ago, short- and long-lived radioactive decay and cosmic ray interaction also contributed to the modification of the isotopic framework of the Solar System, and permit to trace the formation and evolution of directly accessible and inferred planetary and stellar isotopic reservoirs.

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# Introduction to the Solar System and Galactic environment

As for any good recipe, the right ingredients and perfect timing are crucial for the preparation of a Solar System capable of sustaining life. At a distance of  $8.33 \pm 0.35$  kiloparsecs from the Galactic Center,<sup>1</sup> currently at ~25 parsecs from the central plane of the Milky Way,<sup>2</sup> our Sun and associated planetary bodies formed ~4.567 Gyr<sup>3</sup> to ~4.568 Gyr<sup>4</sup> ago within the Milky Way's Galactic habitable zone. As a younger generation star in a universe of  $13.7 \pm 0.2$  Gyr,<sup>5</sup> the Solar System's chemical and isotopic composition is largely the result of nucleosynthesis in multiple generations of Milky Way stars, which enriched the interstellar medium (ISM) with their products of nucleosynthesis, as well as mixing and sampling of Galactic matter, and thus represents several billion years of Galactic chemical evolution.

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Most of the isotopes making up the baryonic matter in our Universe were produced by complex nuclear processes (nucleosynthesis) in specific astrophysical settings, such as the Big Bang and stars (including supernovae). Big bang (primordial) nucleosynthesis, taking place 10 s to 20 min after the Big Bang, produced most of the universe's <sup>1</sup>H, stable helium (in the form of <sup>4</sup>He) along with small amounts of deuterium (<sup>2</sup>H or D), and <sup>3</sup>He and stable lithium-7 (primordially produced radioactive <sup>3</sup>H and <sup>7</sup>Be decayed to <sup>3</sup>He and <sup>7</sup>Li). Since the late 50s, stellar nucleosynthesis in evolving and exploding stars has been known to be the source of all the elements heavier than Li and Be, and it is considered the main "kitchen stove" preparing the chemical evolution of the Milky Way since its formation.<sup>6</sup> Most stars (and their planetary disks), including our Sun, form in groups or clusters of stars in giant molecular clouds, the densest parts of the ISM (e.g., ref. 7). When gravitational contraction of a localized, dense region of a large interstellar molecular cloud leads to the accretion of a central star, the chemical and isotopic compositions of the surrounding rotating disk of gas and fine dust grains will have been influenced by irradiation and influx of matter from older stars.8 For

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# Differentiation and magmatic activity in Vesta evidenced by <sup>26</sup>Al-<sup>26</sup>Mg dating in eucrites and diogenites

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## Abstract

The <sup>26</sup>Al-<sup>26</sup>Mg short-lived chronometer has been widely used for dating ancient objects in studying the early Solar System. Here, we use this chronometer to investigate and refine the geological history of the asteroid 4-Vesta. Ten meteorites widely believed to come from Vesta (4 basaltic eucrites, 3 cumulate eucrites and 3 diogenites) and the unique achondrite Asuka 881394 were selected for this study. All samples were analyzed for their  $\delta^{26}Mg^*$  and  ${}^{27}Al/{}^{24}Mg$  ratios, in order to construct both whole rock and model whole rock isochrons. Mineral separation was performed on 8 of the HED's in order to obtain internal isochrons. While whole rock Al-Mg analyses of HED's plot on a regression that could be interpreted as a vestan planetary isochron, internal mineral isochrons indicate a more complex history. Crystallization ages obtained from internal <sup>26</sup>Al-<sup>26</sup>Mg systematic in basaltic eucrites show that Vesta's upper crust was formed during a short period of magmatic activity at 2.66<sup>+1.39</sup><sub>-0.58</sub> million years (Ma) after Calcium-Aluminum inclusions (after CAI). We also suggest that impact metamorphism and subsequent age resetting could have taken place at the surface of Vesta while  $^{26}$ Al was still extant. Cumulate eucrites crystallized progressively from  $5.48^{+1.56}_{-0.60}$  to >7.25 Ma after CAI. Model ages obtained for both basaltic and cumulate eucrites are similar and suggest that the timing of differentiation of a common eucrite source from a chondritic body can be modeled at  $2.88^{+0.14}_{-0.12}$  Ma after CAI, i.e. contemporaneously from the onset of the basaltic eucritic crust. Based on their cumulate texture, we suggest cumulate eucrites were likely formed deeper in the crust of Vesta. Diogenites have a more complicated history and their  ${}^{26}Al - {}^{26}Mg$  systematics show that they likely formed after the complete decay of  ${}^{26}Al$  and thus are younger than eucrites. This refined chronology for eucrites and diogenites is consistent with a short magma ocean stage on 4-Vesta from which the basaltic eucrites rapidly crystallized. In order to explain the younger age and the complex history of diogenites, we postulate that a second episode of magmatism was possibly triggered by a mantle overturn. We bring a refined chronology of the geological history of Vesta that shows that the asteroid has known a more-complex differentiation than previously thought. © 2017 The Authors. Published by Elsevier Ltd. This is an open access article under the CC BY license (http://creativecommons.org/ licenses/by/4.0/).

Keywords: Al-Mg dating; Vesta; Eucrites; Diogenites

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## 1. INTRODUCTION

Eucrites and diogenites are igneous rocks belonging to the howardite-eucrite-diogenite meteorite (HED) series. The HED's are widely believed to originate from early magmatic activity on the same parent body, one of the three largest asteroids of the asteroid belt, 4-Vesta (Vesta hereafter) (Lugmair and Shukolyukov, 1998; Drake, 2001; Mittlefehldt, 2015). The common origin of these three types —報告— Report

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# Report of the JARE-54 and BELARE 2012–2013 joint expedition to collect meteorites on the Nansen Ice Field, Antarctica

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第54次日本南極地域観測隊と2012-2013 年ベルギー南極観測隊との 合同によるナンセン氷原における隕石探査報告

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要旨:第54次日本南極地域観測隊員4名とベルギー南極観測隊員6名の合計 10名から構成される隕石探査隊は、2012年12月から2013年2月まで、セール・ ロンダーネ山地南部に広がるナンセン氷原(南緯72°30′-73°、東経23°-25°、標高 約2900-3000m)において隕石探査を実施した。ナンセン氷原には2012年12月 26日から2013年2月2日まで39日間滞在した。今回の探査域は第29次日本南 極地域観測隊以降探査が行われていない、探査の結果、採集した隕石の総数は 424個、合計重量は約70kgであった。隕石発見地点は携帯GPSに記録されたので、 探査域における隕石の分布が明確になった。これは隕石集積機構解明のための基 礎データだけでなく、今後の探査計画に活用できる。本稿は主に日本隊による準 備期間を含む実施報告書である。

*Abstract*: This paper reports on a joint expedition (JARE-54 and BELARE 2012–2013) that conducted a search for meteorites on the Nansen Ice Field, Antarctica, in an area south of the Sør Rondane Mountains  $(72^{\circ}30'-73^{\circ}S, 23^{\circ}-25^{\circ}E)$ ; elevation 2900–3000 m). The expedition took place over a period of 39 days during the austral summer, between 26

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# Chicxulub and the Exploration of Large Peak-Ring Impact Craters through Scientific Drilling

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## ABSTRACT

The Chicxulub crater is the only wellpreserved peak-ring crater on Earth and linked, famously, to the K-T or K-Pg mass extinction event. For the first time, geologists have drilled into the peak ring of that crater in the International Ocean Discovery Program and International Continental Scientific Drilling Program (IODP-ICDP) Expedition 364. The Chicxulub impact event, the environmental calamity it produced, and the paleobiological consequences are among the most captivating topics being discussed in the geologic community. Here we focus attention on the geological processes that shaped the ~200-km-wide impact crater responsible for that discussion and the expedition's first year results.

### INTRODUCTION

The Chicxulub crater (Hildebrand et al., 1991) on the Yucatán Peninsula of Mexico was produced by a terminal Cretaceous impact that has been linked to regional and global K-T or K-Pg boundary deposits (see reviews by Smit, 1999; Kring, 2000, 2007; Schulte et al., 2010). The subsurface structure was initially detected with geophysical techniques (Cornejo Toledo and Hernandez Osuna, 1950). While exploring the source of those anomalies, Petróleos Mexicanos (PEMEX) drilled three exploration wells (all dry) into the structure. Petrologic analyses of polymict breccias and melt rock in recovered core samples revealed shock-metamorphic and shockmelted features diagnostic of impact cratering (Kring et al., 1991; Kring and Boynton, 1992; Swisher et al., 1992, Sharpton et al., 1992; Claeys et al., 2003),

proving the structure had an impact origin. The buried structure was confirmed by seismic surveys conducted in 1996 and 2005 to be a large ~180–200-km–diameter impact crater with an intact peak ring (Morgan et al., 1997; Gulick et al., 2008).

The discovery of the Chicxulub impact structure initially prompted two scientific drilling campaigns. In the mid-1990s, a series of shallow onshore wells up to 700 m deep were drilled by the Universidad Nacional Autónoma de México (UNAM; Urrutia-Fucugauchi et al., 1996) to sample near-surface impact breccias in the ejecta blanket surrounding the crater. In 2002, the International Continental Scientific Drilling Program (ICDP) also sponsored a deep drilling project, producing a 1511 m borehole between the peak ring and the crater rim. Continuous core beneath 404 m included Tertiary marine sediments, polymict impact breccias, an impact melt unit, and one or more blocks of Cretaceous sedimentary target rocks. We refer readers to two special issues of Meteoritics & Planetary Science (Jull, 2004a, 2004b) for the major results of that ICDP project, but note that the project left unresolved, among other things, the geologic processes that produced the peak-ring morphology of the crater.

The Chicxulub crater is the best-preserved peak-ring impact basin on Earth, so it is an essential target for additional study. The only other known similarly sized surviving impact structures, Sudbury and Vredefort, are tectonically deformed and eroded. Recently, the International Ocean Discovery Program (IODP) and ICDP drilled an offshore borehole into the crater (Fig. 1), recovering core from a depth of 505.7–1334.7 m below the sea floor (mbsf), to assess the depth of origin of the peakring rock types and determine how they were deformed during the crater-forming event. That information is needed to effectively test how peak-ring craters form on planetary bodies.

The expedition was also designed to measure any hydrothermal alteration in the peak ring and physical properties of the rocks, such as porosity and permeability, to calibrate geophysical data, test models of impact-generated hydrothermal systems, evaluate the habitability of the peak ring, and investigate the recovery of life in a sterilized portion of Earth's surface. The recovered rocks also make it possible to evaluate shock deformation of Earth's crust, including the vaporization of rocks that may have contributed to climate-altering effects of the impact. A large number of geological, environmental, and biological results will emerge from the expedition. Here, we focus on the planetary geoscience findings: how the peak-ring crater formed and what peak-ring and multi-ring craters can reveal about deep planetary crusts. As the borehole pierced only a single location within the crater, we begin by looking at a fully exposed peak-ring crater on the Moon, which provides a picture of a similar structure to that targeted by Expedition 364.

### EXPOSED PEAK-RING CRATERS

The Schrödinger basin near the south pole on the lunar far side is the youngest and best preserved peak-ring crater on the Moon (Fig. 2A). The ~320-km-diameter crater contains an ~150-km-diameter peak ring that rises up to 2.5 km above the crater floor (Shoemaker et al., 1994). The peak ring is topographically complex, with

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# Mn–Cr dating of Fe- and Ca-rich olivine from 'quenched' and 'plutonic' angrite meteorites using Secondary Ion Mass Spectrometry

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### Abstract

Angrite meteorites are suitable for Mn-Cr relative dating (<sup>53</sup>Mn decays to <sup>53</sup>Cr with a half life of 3.7 Myr) using Secondary Ion Mass Spectrometry (SIMS) because they contain olivine and kirschsteinite with very high <sup>55</sup>Mn/<sup>52</sup>Cr ratios arising from very low Cr concentrations. Discrepant Mn-Cr and U-Pb time intervals between the extrusive or 'quenched' angrite D'Orbigny and some slowly cooled or 'plutonic' angrites suggests that some have been affected by secondary disturbances, but this seems to have occurred in quenched rather than in slow-cooled plutonic angrites, where such disturbance or delay of isotopic closure might be expected. Using SIMS, we investigate the Mn-Cr systematics of quenched angrites to higher precision than previously achieved by this method and extend our investigation to non-quenched (plutonic or sub-volcanic) angrites. High values of  $3.54 (\pm 0.18) \times 10^{-6}$  and  $3.40 (\pm 0.19) \times 10^{-6}$  (2-sigma) are found for the initial <sup>53</sup>Mn/<sup>55</sup>Mn of the initial <sup>53</sup>Mn of the initial <sup>53</sup>Mn of the initial <sup>53</sup>Mn of the initial <sup>53</sup>Mn quenched angrites D'Orbigny and Sahara 99555, which are preserved by Cr-poor olivine and kirschsteinite. The previously reported initial <sup>53</sup>Mn/<sup>55</sup>Mn value of D'Orbigny obtained from bulk-rock and mineral separates is slightly lower and was probably controlled by Cr-rich olivine. Results can be interpreted in terms of the diffusivity of Cr in this mineral. Very low Cr concentrations in Ca-rich olivine and kirschsteinite are probably charge balanced by Al; this substitutes for Si and likely diffuses at a very slow rate because Si is the slowest-diffusing cation in olivine. Diffusion in Cr-rich Mg-Fe olivine is probably controlled by cation vacancies because of deficiency in charge-balancing Al and is therefore more prone to disturbance. The higher initial <sup>53</sup>Mn/<sup>55</sup>Mn found by SIMS for extrusive angrites is more likely to reflect closure of Cr in kirschsteinite at the time of crystallisation, simultaneous with closure of U-Pb and Hf-W isotope systematics for these meteorites obtained from pyroxenes. For the younger angrites Northwest Africa (NWA) 4590 and 4801 we have found initial <sup>53</sup>Mn/<sup>55</sup>Mn values which are consistent with more precise work, at 0.90 ( $\pm 0.4$ )  $\times 10^{-6}$  and 0.13 ( $\pm 1.1$ )  $\times 10^{-6}$  respectively. Our work shows that SIMS can usefully constrain and distinguish the ages of angrites of different petrologic groups. In reviewing the petrology of angrites, we suggest that NWA 2999, 4590, and 4801 underwent a secondary partial melting and Cr (+/-Pb) disturbance event that the sub-volcanic Lewis Cliff 86010, and perhaps the plutonic Angra dos Reis, did not. With our higher initial <sup>53</sup>Mn/<sup>55</sup>Mn for D'Orbigny and Sahara 99555 as well as previous data, a combined quenched angrite initial <sup>53</sup>Mn/<sup>55</sup>Mn of  $3.47 (\pm 0.12) \times 10^{-6}$  (2-sigma, MSWD 1.00) yields consistent Mn–Cr and U–Pb intervals between these angrites and Lewis Cliff 86010. Discrepant Mn-Cr timescales for other plutonic and sub-volcanic angrites represents resetting during the

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# REE and Hf distribution among mineral phases in the CV–CK clan: A way to explain present-day Hf isotopic variations in chondrites

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### Abstract

Chondrites are among the most primitive objects in the Solar System and constitute the main building blocks of telluric planets. Among the radiochronometers currently used for dating geological events, Sm-Nd and Lu-Hf are both composed of refractory, lithophile element. They are thought to behave similarly as the parent elements (Sm and Lu) are generally less incompatible than the daughter elements (Nd and Hf) during geological processes. As such, their respective average isotopic compositions for the solar system should be well defined by the average of chondrites, called Chondritic Uniform Reservoir (CHUR). However, while the Sm-Nd isotopic system shows an actual spread of less than 4% in the average chondritic record, the Lu-Hf system shows a larger variation range of 28% [Bouvier A., Vervoort J. D. and Patchett P. J. (2008) The Lu-Hf and Sm-Nd isotopic composition of CHUR: Constraints from unequilibrated chondrites and implications for the bulk composition of terrestrial planets. Earth Planet. Sci. Lett. 273, 48-57]. To better understand the contrast between Sm-Nd and Lu-Hf systems, the REE and Hf distribution among mineral phases during metamorphism of Karoonda (CK) and Vigarano-type (CV) carbonaceous chondrites has been examined. Mineral modes were determined from elemental mapping on a set of five CK chondrites (from types 3-6) and one CV3 chondrite. Trace-element patterns are obtained for the first time in all the chondrite-forming minerals of a given class (CK chondrites) as well as one CV3 sample. This study reveals that REE are distributed among both phosphates and silicates. Only 30-50% of Sm and Nd are stored in phosphates (at least in chondrites types 3-5); as such, they are not mobilized during early stages of metamorphism. The remaining fraction of Sm and Nd is distributed among the same mineral phases; these elements are therefore not decoupled during metamorphism. Of the whole-rock total of Lu, the fraction held in phosphate decreases significantly as the degree of metamorphism increases (30% for types 3 and 4, less than 5% in type 6). In contrast to Lu, Hf is mainly hosted by silicates with little contribution from phosphates throughout the CK metamorphic sequence. A significant part of Sm and Nd are stored in phosphates in types 3-5, and these elements behave similarly during CK chondrite metamorphism. That explains the robustness of the Sm/Nd ratios in chondrites through metamorphism, and the slight discrepancies observed in the present-day isotopic Nd values in chondrites. On the contrary, Lu and Hf are borne by several different minerals and consequently they are redistributed during

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# Rapid cooling of planetesimal core-mantle reaction zones from Mn-Cr isotopes in pallasites

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# Abstract



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Pallasite meteorites, which consist of olivine-metal mixtures and accessory phosphates crystallised from silico-phosphate melts, are thought to represent core-mantle reaction zones of early differentiating planetesimals. Pallasite meteorites can be linked to five distinct planetesimals, indicating that they are default products of differentiation. However, their formation modes (deep, shallow, and impact environments) and age are still elusive. We have investigated the trace element and Mn-Cr isotopic signatures of Main-Group pallasite olivine, finding enhanced Mn, P and <sup>53</sup>Cr/<sup>52</sup>Cr near crystal rims which indicates early ingrowth of radiogenic <sup>53</sup>Cr\* in silico-phosphate melts. Mn-Cr isotopic data corroborate previous Hf-W isotopic data, indicating an early metal-silicate separation event but additionally that rapid cooling generated silico-phosphate eutectic melts with high Mn/Cr within ~2.5 to 4 Myr of Solar System formation. These melts formed before most known samples of planetesimal crusts (eucrite and angrite meteorites) and are among the earliest evolved planetary silicates. Additionally, Mn-rich phosphates in other, non-Main-Group pallasite meteorites suggest that core-mantle reaction zones are generic, datable features of differentiation.

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# Introduction

The accumulated oxygen isotopic evidence for five distinct parent bodies of olivine and metal-rich pallasite meteorites (summarised in Boesenberg *et al.*, 2012) suggests that pallasitic material is the default end-product of planetesimal differentiation. Pallasites may represent samples of quiescent core-mantle boundaries (Boesenberg *et al.*, 2012), or violently formed mixtures of core and mantle materials (Yang *et al.*, 2010; Tarduno *et al.*, 2012) and are often discussed

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Stratigraphic record of the asteroidal Veritas breakup in the Tortonian Monte dei Corvi section (Ancona, Italy)

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### ABSTRACT

The discovery of elevated concentrations of the cosmogenic radionuclide <sup>3</sup>He in deepsea sediments from Ocean Drilling Program (ODP) Site 926 (Atlantic Ocean) and ODP Site 757 (Indian Ocean) points toward accretion of extraterrestrial matter, probably as a result of the catastrophic disruption of a large asteroid that produced the Veritas familv of asteroids at ca.  $8.3 \pm 0.5$  Ma. and which may have had important effects on the global climatic and ecologic systems. Here, we investigated the signatures possibly related to the Veritas event by performing a highresolution multiproxy stratigraphic analysis through the late Tortonian-early Messinian Monte dei Corvi section near Ancona, Italy. Closely spaced bulk-rock samples through a 36-m-thick section, approximately spanning from ca. 9.9 Ma to ca. 6.4 Ma, show an ~5fold <sup>3</sup>He anomaly starting at ca. 8.5 Ma and returning to background values at ca. 6.9 Ma, confirming the global nature of the event. We then analyzed, at 5 cm intervals, bulk-rock samples for sedimentary and environmental proxies such as magnetic susceptibility, calcium carbonate content, total organic carbon, and bulk carbonate  $\delta^{18}$ O and  $\delta^{13}$ C, through a 21-m-thick section encompassing the <sup>3</sup>He anomaly. Available high-resolution sea-surface temperature data (via alkenone analyses) for this site show a temperature decrease starting exactly at the inception of the <sup>3</sup>He anomaly. Cyclostratigraphic fast-Fourier-transform spectral analyses of the proxies indicate an age of  $8.47 \pm 0.05$  Ma for the inception of the <sup>3</sup>He anomaly. A search for impact ejecta (analogous to what is present in the late Eocene, where both a <sup>3</sup>He anomaly and large-scale impact events are recorded) was not successful. Detailed cyclostratigraphic analyses of our data suggest that the changes in the stable isotope series and environmental proxy series through this late Tortonian time interval had a common forcing agent, and that perturbations of orbitally forced climate cycles are present exactly through the interval with the enhanced influx of extraterrestrial <sup>3</sup>He. Thus, the chemostratigraphic evidence for a collisional event that created the Veritas family of asteroids, coinciding with climate perturbations on Earth, suggests vet another form of interaction between Earth and the solar system.

#### INTRODUCTION

#### Geologic Setting

As a consequence of the Cretaceous-to-present tectono-sedimentary evolution of the Northern Apennines accretionary wedge, as synthetically illustrated in Figure 1A (e.g., D'Argenio, 1970; Alvarez et al., 1974; Channell et al., 1979; Castellarin et al., 1982; Treves, 1984; Marroni et al., 2001; Cornamusini et al., 2002; Argnani et al., 2006, and references therein), the complete and continuous Jurassic to early Miocene pelagic succession of the Umbria-Marche Basin, for example, in the type locality of Gubbio, is interrupted in the mid-Miocene by the arrival of the siliciclastic turbidites of the Marnoso-Arenacea Flysch. The much-celebrated stratigraphic completeness of the classic Gubbio pelagic succession has, in the past half a century or so, contributed considerably to the ever-improving resolution of event and integrated stratigraphy of Earth history (e.g., Menichetti et al., 2016, and references therein). However, with the arrival of the flysch, detailed integrated stratigraphic study cannot be extended into the Miocene. This interruption did not occur in the easternmost part of the Umbria-Marche Basin, along the Cònero Riviera, which extends along the anticlinal Adriatic promontory between the city of Ancona and Monte Cònero (for location, see Fig. 1A). Here, turbidite-free, pelagic and hemipelagic carbonates covering the entire Miocene Epoch all the way up to the early Pliocene are continuously exposed on sea cliffs. In particular, the monoclinal sections covering the Langhian to early Pliocene portion of this pelagic succession on the eastern cliffs of Monte dei Corvi (for location, see Fig. 1B) have, in recent years, promoted high-resolution integrated stratigraphic studies of mid- and late Miocene events, including biostratigraphic, chemostratigraphic, magnetostratigraphic events and their radioisotopic and astrochronologic calibrations (Montanari et al., 1997; Cleaveland et al., 2002; Hilgen et al., 2003; Mader et al., 2004; Hüsing et al., 2010; Wotzlaw et al., 2014; Tzanova et al., 2015). The global stratotype section and point (GSSP) for the base of the Tortonian Stage was established in the Monte dei Corvi section by Hilgen et al. (2005). Recently, the stratigraphic stretch comprising the Upper Tortonian to the basal Messinian exposed in a new Monte dei Corvi Beach section, i.e., the stratigraphical equivalent of the La Sardella section of Montanari et al. (1997) exposed up high on the Monte dei Corvi cliff (Fig. 1B), was proposed by Hüsing et al. (2007)

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#### GEOLOGY

# The formation of peak rings in large impact craters

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Large impacts provide a mechanism for resurfacing planets through mixing near-surface rocks with deeper material. Central peaks are formed from the dynamic uplift of rocks during crater formation. As crater size increases, central peaks transition to peak rings. Without samples, debate surrounds the mechanics of peak-ring formation and their depth of origin. Chicxulub is the only known impact structure on Earth with an unequivocal peak ring, but it is buried and only accessible through drilling. Expedition 364 sampled the Chicxulub peak ring, which we found was formed from uplifted, fractured, shocked, felsic basement rocks. The peak-ring rocks are cross-cut by dikes and shear zones and have an unusually low density and seismic velocity. Large impacts therefore generate vertical fluxes and increase porosity in planetary crust.

mpacts of asteroids and comets play a major role in planetary evolution by fracturing upper-crustal lithologies, excavating and ejecting material from the impact site, producing melt pools, and uplifting and exposing subsurface rocks. The uplift of material during impact cratering rejuvenates planetary surfaces with deeper material. Complex impact craters on rocky planetary bodies possess a central peak or a ring of peaks internal to the crater rim, and the craters with these features are termed central-peak and peak-ring craters, respectively (1). Most known peak-ring craters occur on planetary bodies other than Earth, prohibiting assessment of their physical state and depth of origin. Here, we address the question of how peak rings are formed, using geophysical data, numerical simulations, and samples of the Chicxulub peak ring obtained in a joint drilling expedition by the International Ocean Discovery Program (IODP) and International Continental Scientific Drilling Program (ICDP).

Upon impact, a transient cavity is initially formed, which then collapses to produce a final crater that is both shallower and wider than the transient cavity (*I*). Dynamic uplift of rocks during the collapse of the transient cavity in the early stages of crater formation (Fig. 1, B and C) likely forms central peaks (*2*). The dynamic collapse model of peak-ring formation attributes the origin of peak rings to the collapse of overheightened central peaks (*3*). The observational evidence for this model is most obvious on Venus, where central peaks gradually evolve into peak rings with increasing crater size (4). The peak-ring-diameter-to-crater-rim-diameter ratio increases with crater size on Venus but does not get much larger than ~0.5. The lack of any further increase in this ratio led to the suggestion that in larger craters, the outward collapse of peakring material is halted when it meets the collapsing transient cavity rim (4).

A different concept for peak-ring formationthe nested melt-cavity hypothesis-evolved from observations of peak-ring craters on the Moon and Mercury (5-7). This alternative hypothesis envisions that the uppermost central uplift is melted during impact, and an attenuated central uplift remains below the impact melt sheet and does not overshoot the crater floor during the modification stage. Hence, in contrast to the dynamic collapse model (Fig. 1), this nested meltcavity hypothesis would not predict outward thrusting of uplifted rocks above the collapsed transient cavity rim material. The origin and shock state of rocks that form a peak ring are less clear in the nested melt-cavity hypothesis because they have not been evaluated with numerical simulations. Head, however, postulated that material in the outer margin of the melt cavity forms the peak ring and therefore should be close to melting (6). This requires shock pressures of just below 60 GPa. In contrast, Baker et al. propose that peak rings are formed from inwardly slumped rotated blocks of transient cavity rim material originating at shallow depths and thus should have experienced lower average shock pressures than simulated in the dynamic collapse model (7).

The transition from central-peak to peak-ring craters with increasing crater size scales inversely with gravity (*I*), suggesting that the same transition diameter of ~30 km found on Venus (*4*) should also hold for Earth, and that craters >30 km in diameter should possess a peak ring. Craters on Earth often display internal ring-like structures, but complications and uncertainties owing to target heterogeneity, erosion, and sedimentation make it difficult to distinguish peak rings that are genetically linked to their extraterrestrial counterparts (*8*, *9*). Seismic reflection data across the ~200-km-diameter Chicxulub multi-ring impact structure revealed it to be the Downloaded from http://science.sciencemag.org/ on December 1, 2016

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# Cathodoluminescence as a tool to discriminate impact melt, shocked and unshocked volcanics: A case study of samples from the El'gygytgyn impact structure

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Abstract-El'gygytgyn (Chukotka, Arctic Russia) is a well-preserved impact structure, mostly excavated in siliceous volcanic rocks. For this reason, the El'gygytgyn structure has been investigated in recent years and drilled in 2009 in the framework of an ICDP (International Continental Scientific Drilling Program) project. The target rocks mostly consist of rhyodacitic ignimbrites and tuffs, which make it difficult to distinguish impact melt clasts from fragments of unshocked target rock within the impact breccia. Several chemical and petrologic attempts, other than dating individual clasts, have been considered to distinguish impact melt from unshocked volcanic rock of the targets, but none has proven reliable. Here, we propose to use cathodoluminescence (imaging and spectrometry), whose intensity is inversely correlated with the degree of shock metamorphism experienced by the investigated lithology, to aid in such a distinction. Specifically, impact melt rocks display low cathodoluminescence intensity, whereas unshocked volcanic rocks from the area typically show high luminescence. This high luminescence decreases with the degree of shock experienced by the individual clasts in the impact breccia, down to almost undetectable when the groundmass is completely molten. This might apply only to El'gygytgyn, because the luminescence in volcanic rocks might be due to devitrification and recrystallization processes of the relatively old (Cretaceous) target rock with respect to the young impactites (3.58 Ma). The alteration that affects most samples from the drill core does not have a significant effect on the cathodoluminescence response. In conclusion, cathodoluminescence imaging and spectra, supported by Raman spectroscopy, potentially provide a useful tool for in situ characterization of siliceous impactites formed in volcanic target.

## **INTRODUCTION**

Impact craters on Earth can be considered analogs of those on the Moon, Mars, and other planetary bodies with solid surfaces, except for the general target composition. Craters excavated in volcanic rocks are rare on Earth: Lonar in India (Kieffer et al. 1976), Logancha in Russia (Masaitis 1999), as well as Vista Alegre and Vargeão in Brazil (Crósta et al. 2010, 2012) were all formed in basaltic target rocks, and the El'gygytgyn impact structure in Russia (Dietz and McHone 1976) that was formed in siliceous volcanic rocks. The Monturaqui impact crater was formed in target rocks that include some ignimbrites, but the impact event mostly affected the underlying granites (e.g., Ugalde et al. 2007). Impact craters excavated in volcanic rocks provide information on shock metamorphism in volcanic terrains, and, for instance, those formed in basalts are comparable to the craters on the crust of the Moon. In particular, El'gygytgyn represents the only known impact structure excavated in a Cretaceous volcanic suite of mostly rhyodacitic

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# Melting and cataclastic features in shatter cones in basalt from the Vista Alegre impact structure, Brazil

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Abstract-Shatter cones are one of the most widely recognized pieces of evidence for meteorite impact events on Earth, but the process responsible for their formation is still debated. Evidence of melting on shatter cone surfaces has been rarely reported in the literature from terrestrial impact craters but has been recently observed in impact experiments. Although several models for shatter cones formation have been proposed, so far, no one can explain all the observed features. Shatter cones' from the Vista Alegre impact structure, Brazil, formed in fine-grained basalt of the Jurassic-Cretaceous Serra Geral Formation (Paraná large igneous province). A continuous quenched melt film, consisting of a crystalline phase, mica, and amorphous material, decorates the striated surface. Ultracataclasites, containing subrounded pyroxene clasts in an ultrafine-grained matrix, occur subparallel to the striated surface. Several techniques were applied to characterize the crystalline phase in the melt, including Raman spectroscopy and transmission electron microscopy. Results are not consistent with any known mineral, but they do suggest a possible rare or new type of clinopyroxene. This peculiar evidence of melting and cataclasis in relation with shatter cone surfaces is interpreted as the result of tensile fracturing at the tip of a fast propagating shock-induced rupture, which led to the formation of shatter cones at the tail of the shock front, likely during the early stage of the impact events.

## **INTRODUCTION**

Shatter cones consist of multiple sets of penetrative striated conical fractures that preferentially (but not exclusively) form in fine-grained rocks as result of a meteorite impact event, according to the definition by Dietz (1947, 1960). They can occur individually or in clusters of hierarchically related, roughly conical structures. As similar features cannot be produced by any other terrestrial geological process, shatter cones are considered to be the only macroscopic evidence of an impact event (e.g., French and Koeberl [2010] and references therein). Nevertheless, the process that generates these features is still debated. Shatter cones are generally considered to have formed under low shock pressure (e.g., experiments by Schneider and Wagner 1976), but cases of high shock pressure (up to ca. 25 GPa) have been reported, as suggested by the occurrence of planar deformation features (PDF) in quartz grains across the shatter cone (Hargraves and White 1996; Ferriere and Osinski 2010; Ferriere et al. 2010; Vasconcelos et al. 2013). Hypotheses on shatter cone formation include the following.

1. Scattering of shock waves due to the presence of heterogeneities in the target rock, proposed by Baratoux and Melosh (2003). This numerical model

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## From olivine to ringwoodite: a TEM study of a complex process

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Abstract-The study of shock metamorphism of olivine might help to constrain impact events in the history of meteorites. Although shock features in olivine are well known, so far, there are processes that are not yet completely understood. In shock veins, olivine clasts with a complex structure, with a ringwoodite rim and a dense network of lamellae of unidentified nature in the core, have been reported in the literature. A highly shocked (S5-6), L6 meteorite, Asuka 09584, which was recently collected in Antarctica by a Belgian-Japanese joint expedition, contains this type of shocked olivine clasts and has been, therefore, selected for detailed investigations of these features by transmission electron microscopy (TEM). Petrographic, geochemical, and crystallographic studies showed that the rim of these shocked clasts consists of an aggregate of nanocrystals of ringwoodite, with lower Mg/Fe ratio than the unshocked olivine. The clast's core consists of an aggregate of iso-oriented grains of olivine and wadsleyite, with higher Mg/Fe ratio than the unshocked olivine. This aggregate is crosscut by veinlets of nanocrystals of olivine, with extremely low Mg/Fe ratio. The formation of the ringwoodite rim is likely due to solid-state, diffusion-controlled, transformation from olivine under high-temperature conditions. The aggregate of iso-oriented olivine and wadsleyite crystals is interpreted to have formed also by a solid-state process, likely by coherent intracrystalline nucleation. Following the compression, shock release is believed to have caused opening of cracks and fractures in olivine and formation of olivine melt, which has lately crystallized under postshock equilibrium pressure conditions as olivine.

## **INTRODUCTION**

The evaluation of the shock-metamorphic stage of ordinary chondrites is part of the standard meteorite classification (Stöffler et al. 1991). In particular, the shock metamorphism of olivine, a common phase in meteorites and with a simpler structure than pyroxene, has been extensively studied. Typical shock features of olivine include, with increasing shock pressure, (i) planar fractures (PF); (ii) planar deformation features (PDF); (iii) transition to high-pressure polymorphs, such as wadsleyite and ringwoodite; and (iv) melting (see Madon and Poirier 1983; for pioneering transmission electron microscopy [TEM] studies and Stöffler et al. [1991] and references therein for peak pressure correlation). The shock-metamorphic effects in olivine provide a shock-barometer that reaches higher shock peak pressure than the one based on quartz for terrestrial rocks (as silica is completely molten above 50 GPa, whereas olivine becomes glass only at ~75 GPa; Stöffler and Langenhorst 1994; Langenhorst 2002). High-pressure polymorphs of olivine and pyroxene are commonly observed in shock veins crosscutting ordinary chondrites (e.g., Stöffler and Langenhorst 1994).

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## Screening and classification of ordinary chondrites by Raman spectroscopy

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Abstract-Classification of ordinary chondrite meteorites generally implies (1) determining the chemical group by the composition in endmembers of olivine and pyroxene, and (2) identifying the petrologic group by microstructural features. The composition of olivine and pyroxene is commonly obtained by microprobe analyses or oil immersion of mineral separates. We propose Raman spectroscopy as an alternative technique to determine the endmember content of olivine and pyroxene in ordinary chondrites, by using the link between the wavelength shift of selected characteristic peaks in the spectra of olivine and pyroxene and the Mg/Fe ratio in these phases. The existing correlation curve has been recalculated from the Raman spectrum of reference minerals of known composition and further refined for the range of chondritic compositions. Although the technique is not as accurate as the microprobe for determining the composition of olivine and pyroxene, for most of the samples the chemical group can be easily determined by Raman spectroscopy. Blind tests with ordinary chondrites of different provenance, weathering, and shock stages have confirmed the potential of the method. Therefore, we suggest that a preliminary screening and the classification of most of the equilibrated ordinary chondrites can be carried out using an optical microscope equipped with a Raman spectrometer.

### INTRODUCTION

Ordinary chondrites are the most common meteorites in the world (Weisberg et al. 2003). Since 2009, joint Belgian–Japanese expeditions in Antarctica have brought back hundreds of new meteorites. New meteorites must be classified for the submission and the approval of their name, as well as for meteorites collected in Antarctica by other missions (e.g., ANSMET) and for those found in the Sahara area (e.g., Idomar et al. 2014). Ordinary chondrites dominate in the historical collections in museums (e.g., Krot et al. 2005) and among the newly collected meteorites. Classification of ordinary chondrites is time consuming and requires qualified scientists. The classification of the Belgian–Japanese ordinary chondrites is done at the National Institute of Polar Research (NIPR) through

two analytical steps (1) evaluation of the Fa and Fs content in olivine and in low-Ca pyroxene, respectively, for determining the chemical group by electron microscopy and (2) identification of the petrologic group (from 3 to 6, depending on the metamorphic grade), weathering, and possibly shock stage by optical microscopy (e.g., Weisberg et al. [2003] and references therein). The Fe range for chemical group is generally expressed as endmember content. For olivine, the endmembers are fayalite (Fa), with formula Fe<sub>2</sub>SiO<sub>4</sub>, and forsterite (Fo), with mineral formula Mg<sub>2</sub>SiO<sub>4</sub>. For low-Ca pyroxene, the endmembers are enstatite (En), with mineral formula Mg2Si2O6, and ferrosilite (Fs), with mineral formula Fe<sub>2</sub>Si<sub>2</sub>O<sub>6</sub>. The chemical groups in ordinary chondrites are: H (Fa<sub>16-20</sub> and Fs<sub>14-18</sub>), L (Fa<sub>22-26</sub> and Fs<sub>19-22</sub>), and LL (Fa<sub>27-32</sub> and Fs<sub>22-26</sub>), according to the definition in Van Schmus and Wood

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# Forum Comment

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## Mercury anomaly, Deccan volcanism, and the end-Cretaceous mass extinction

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In their study of Hg abundances across the Cretaceous-Paleogene (KPg) boundary, Font et al. (2016) discuss links between Hg abundances, Deccan volcanism, and the end-Cretaceous mass extinction, and conclude that a significant Hg concentration increase just below the KPg boundary at Bidart (France), which they consider to be a contributor to the biological crisis, should be ascribed to the Deccan volcanism outflow Phase-2.

However, an earlier interpretation (Lowrie et al., 1990) suggested that the Hg anomaly could have been enriched/scavenged by anoxia on the seafloor and subsequently penetrated into uppermost 10–50 cm of the Maastrichtian. Mercury is, therefore, post-depositional and cannot be directly related to either the Deccan traps eruptions or the Chicxulub impact event. We weigh the two interpretations in a few points here, and conclude that the interpretation relating the Hg supply to the Deccan trap Phase-2 eruptions is unlikely.

(1) In the Bidart section, the zone of the Hg anomaly extends to 50-75 cm below the KPg boundary. The discolored top of the Cretaceous is 50-60 cm thick. Bidart is part of the Basque basin, where at least six extended sections across the KPg boundary are very similar (Mount and Ward, 1986; Ward et al., 1991; Batenburg et al., 2011) and bed-for-bed comparable to one another. Batenburg et al. (2011) clearly showed that the very distinct marl-limestone bedding visible in all the sections is orbitally (Milankovitch-cycle) controlled, and that the layers can be correlated to the precession cycle, lasting on average 20.5 k.y. Three of these cycles are indeed depicted in Font et al.'s figure 2. The thickness of each precessional cycle just below the KPg boundary is ~120 cm, so the weathering zone and the almost coincident Hg anomaly below the KPg boundary represent less than half a cycle, and consequently represent  ${<}10$ k.y. in duration. Phase 2 of the Deccan traps begins somewhere in the top of MagnetoChron 30N. The Chron 29–30 boundary is consistently 18  $\pm$  1 precession cycles (Herbert, 1999; Batenburg et al. 2011; Westerhold et al. 2012) below the KPg boundary, thus roughly 370 k.y. Therefore, the Deccan traps Phase-2 should begin >370 k.y., which corresponds to ~20 m below the KPg boundary in the Bidart section. As a consequence, the Hg concentrations, if derived from the Deccan traps directly, should have been detected throughout the whole 20 m below the KPg boundary, and not be restricted to the top 50-60 cm.

(2) The Chicxulub impact event has led to mass mortality, and therefore to massive anoxia on the deep seafloor, where normally oxidative circumstances reign. The high Corg content, the widespread presence of pyrite and of C40:2 ketones (Yamamoto et al., 1996) indicate that the KPg boundary clay layer directly above the few-millimeter-thick Chicxulub ejecta was deposited under dysoxic conditions. Font et al. (2016) cite Grasby et al. (2013), who states "Dissolved Hg has strong affinity for organic matter (OM) in marine and freshwater environments" and is expected to be enriched/scavenged during that phase. Adding to the Hg enrichment in the anoxic boundary clay, a possible extraterrestrial source of Hg at the KPg boundary has been suggested by Meier et al. (2015). The anoxia easily penetrates, possibly aided by anaerobic

microbial processes, several decimeters into the seafloor, in this case top Cretaceous, leading to dissolution-reprecipitation and enrichment of several elements, among which are U, Zn, S, As, Fe, and Mn. Mercury is presumably also scavenged by these post-depositional processes and reprecipitated below the sediment-water interface. A similar discoloration zone occurs also in the famous Gubbio section (e.g., Montanari, 1991). There, a white layer extends 30-50 cm below the Ir-bearing clay. The presumed zone of decreased magnetic susceptibility, earlier hypothesized by Font et al. (2016) as caused by the Deccan trap eruptions, (see also Font et al., 2011) coincides with the white reduced layer. Lowrie et al.(1990) concluded that the anoxia transforms the ferric Fe to ferrous Fe, explaining the decreased Fe content and susceptibility in that interval. The leached/discolored low-susceptibility intervals in Gubbio and Bidart are comparable in thickness (~50 cm), while their sedimentation rates differ by a factor of five. If the low-susceptibility, bleached zones and Hg anomaly were directly caused by external supply-hypothesized by Font et al. (2016) as derived from the Deccan traps-then the stratigraphic interval representing the duration of external supply should differ in thickness by a factor of five in both sections. This is clearly not the case.

Therefore, we conclude that the hypothesis of Deccan trap-derived Hg supply should be rejected in favor of the earlier hypothesized post-depositional geochemical leaching and reduction processes.

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## First finding of impact melt in the IIE Netschaëvo meteorite

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Abstract–About half of the IIE nonmagmatic iron meteorites contain silicate inclusions with a primitive to differentiated nature. The presence of preserved chondrules has been reported for two IIE meteorites so far, Netschaëvo and Mont Dieu, which represent the most primitive silicate material within this group. In this study, silicate inclusions from two samples of Netschaëvo were examined. Both silicate inclusions are characterized by a porphyritic texture dominated by clusters of coarse-grained olivine and pyroxene, set in a fine-grained groundmass that consists of new crystals of olivine and a glassy appearing matrix. This texture does not correspond to the description of the previously examined pieces of Netschaëvo, which consist of primitive chondrule-bearing angular clasts. Detailed petrographic observations and geochemical analyses suggest that the investigated samples of Netschaëvo consist of quenched impact melt. This implies that Netschaëvo is a breccia containing metamorphosed and impact-melt rock (IMR) clasts and that collisions played a major role in the formation of the IIE group.

### **INTRODUCTION**

The formation of the majority of iron meteorites can be explained by fractional crystallization of slowly cooling metallic liquids (Scott 1972; Choi et al. 1995), indicating that they originated from the core of differentiated bodies. These meteorites belong to the magmatic or fractionally crystallized groups (IC, IIAB, IIC, IID, IIIAB, IIIE, IIIF, IVA, and IVB). However, the formation of several other iron meteorites, frequently containing silicate inclusions, cannot be explained by fractional crystallization alone (Goldstein et al. 2009). These nonmagmatic or silicate-bearing iron meteorites consist of the IAB complex and IIE.

Among the nonmagmatic iron meteorites, the metal phase of the IIE iron meteorites shows characteristics that distinguish them from the other silicate-bearing groups: (1) a much smaller Ni range (7.2–9.5 wt% for IIE versus 6–60 wt% for the IAB complex), (2) higher As/Ni and Au/Ni ratios, (3) the absence of carbon, and (4) minor amounts of FeS (Wasson and Wang 1986; Choi et al. 1995). About half of the IIE irons contain

silicate inclusions that present a wide variety of characteristics, ranging from large chondritic clasts to smaller molten feldspar-rich globules (Ruzicka 2014). Mittlefehldt et al. (1998) classified silicate-bearing IIE on the basis of the features in the inclusions, from the most primitive (1) to the most differentiated material (5). The first subgroup contains silicate inclusions with preserved chondritic texture and mineralogy. Such angular chondritic clasts that contain preserved chondrules were first found in the IIE iron meteorite Netschaëvo and the meteorite was, thus, chosen to represent subgroup (1) (Olsen and Jarosewich 1971). Until recently this meteorite was also the only member of subgroup (1). Van Roosbroek et al. (2015) proposed that Mont Dieu, a IIE iron containing primitive chondrule-bearing cm-sized silicate inclusions, might also be a member of subgroup (1). The other silicatebearing IIE meteorites are classified in: subgroup (2), e.g., Techado, with silicate inclusions with chondritic bulk composition but without chondritic textures (Casanova et al. 1995); subgroup (3), e.g., Watson, with a chondritic inclusion that has lost its metal and sulfide

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# The formation of IIE iron meteorites investigated by the chondrule-bearing Mont Dieu meteorite

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Abstract–A 435 kg piece of the Mont Dieu iron meteorite (MD) contains cm-sized silicate inclusions. Based on the concentration of Ni, Ga, Ge, and Ir ( $8.59 \pm 0.32 \text{ wt\%}$ , 25.4  $\pm$  0.9 ppm, 61  $\pm$  2 ppm, 7.1  $\pm$  0.4 ppm, respectively) in the metal host, this piece can be classified as a IIE nonmagmatic iron. The silicate inclusions possess a chondritic mineralogy and relict chondrules occur throughout the inclusions. Major element analysis, oxygen isotopic analysis ( $\Delta^{17}O = 0.71 \pm 0.02\%$ ), and mean Fa and Fs molar contents (Fa<sub>15.7  $\pm$  0.4 and Fs<sub>14.4  $\pm$  0.5) indicate that MD originated as an H chondrite. Because of strong similarities with Netschaëvo IIE, MD can be classified in the most primitive subgroup of the IIE sequence. <sup>40</sup>Ar/<sup>39</sup>Ar ages of 4536  $\pm$  59 Ma and 4494  $\pm$  95 Ma obtained on pyroxene and plagioclase inclusions show that MD belongs to the old (~4.5 Ga) group of IIE iron meteorites and that it has not been perturbed by any subsequent heating event following its formation. The primitive character of MD sheds light on the nature of its formation process, its thermal history, and the evolution of its parent body.</sub></sub>

## **INTRODUCTION**

Iron meteorites can be divided into magmatic (IC, IIAB, IIC, IID, IIIAB, IIIE, IIIF, IVA, and IVB) and nonmagmatic groups (IAB, IIICD, and IIE) based on chemical trends (Wasson and Wang 1986). The magmatic irons rarely contain silicate inclusions and their behavior on the siderophile elements versus Ni or Au plots is consistent with fractional crystallization of slowly cooling metallic liquids (Scott 1972; Choi et al. 1995). The nonmagmatic iron meteorites (NMIM) contain silicate inclusions of various nature and show trends on these elemental plots that cannot be explained

by fractional crystallization alone (Scott 1972; Choi et al. 1995; Goldstein et al. 2009).

Within the nonmagmatic groups, a distinction is made between IAB–IIICD and IIE iron meteorites. IAB and IIICD irons are considered as an IAB ironmeteorite complex, consisting of a main group, five subgroups, and several grouplets (Wasson and Kallemeyn 2002). The IIE group can be distinguished from IAB and IIICD by the following characteristics (1) a much smaller Ni range (7.2–9.5 wt% for IIE versus 6– 60 wt% for IAB-IIICD), (2) higher slopes on As-Ni and Au-Ni diagrams, (3) the absence of carbides, and (4) smaller fractions of FeS (Wasson and Wang 1986; Choi

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# Immiscible silicate liquids and phosphoran olivine in Netschaëvo IIE silicate: Analogue for planetesimal core-mantle boundaries

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### Abstract

We have investigated a piece of the Netschaëvo IIE iron meteorite containing a silicate inclusion by means of electron microprobe analysis (EMPA) and transmission electron microscopy (TEM). Netschaëvo contains chondrule-bearing clasts and impact melt rock clasts were also recently found. The examined inclusion belongs to the latter and is characterized by a porphyritic texture dominated by clusters of coarse-grained olivine and pyroxene, set in a fine-grained groundmass that consists of new crystals of olivine and a hyaline matrix. This matrix material has a quasi-basaltic composition in the inner part of the inclusion, whereas the edge of the inclusion has a lower SiO<sub>2</sub> concentration and is enriched in MgO, P<sub>2</sub>O<sub>5</sub>, CaO, and FeO. Close to the metal host, the inclusion also contains euhedral Mg-chromite crystals and small ( $\leq 2 \mu m$ ), Si-rich globules. A TEM foil was cut from this glassy, silico-phosphate material. It shows that the material consists of elongated olivine crystallites containing up to  $14 \text{ wt}\% P_2O_5$ , amorphous material, and interstitial Cl-apatite crystals. The Si-rich silicate glass globules show a second population of Ferich silicate glass droplets, indicating they formed by silicate liquid immiscibility. Together with the presence of phosphoran olivine and quenched Cl-apatite, these textures suggest rapid cooling and quenching as a consequence of an impact event. Moreover, the enrichment of phosphorus in the silicate inclusion close to the metal host (phosphoran olivine and Cl-apatite) indicates that phosphorus re-partitioned from the metal into the silicate phase upon cooling. This probably also took place in pallasite meteorites that contain late-crystallizing phases rich in phosphorus. Accordingly, our findings suggest that oxidation of phosphorus might be a general process in core-mantle environments, bearing on our understanding of planetesimal evolution. Thus, the Netschaëvo sample serves as a natural planetesimal core-mantle boundary experiment and based on our temperature estimates, the following sequence of events takes place: (i) precipitation of olivine ( $1400-1360 \circ C$ ), (ii) re-partitioning of phosphorus from the metal into the silicate phase, and (iii) formation of immiscible melts (1230-1115 °C). © 2016 Elsevier Ltd. All rights reserved.

Keywords: Iron meteorites; Impact melting; Netschaëvo; Silicate liquid immiscibility; Core-mantle boundary

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# Unravelling the high-altitude Nansen blue ice field meteorite trap (East Antarctica) and implications for regional palaeo-conditions

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### Abstract

Antarctic blue ice zones, the most productive locations for meteorite recovery on Earth, contain old ice that is easily accessible and available in large quantities. However, the mechanisms behind these meteorite traps remain a topic of ongoing debate. Here, we propose an interdisciplinary approach to improve our understanding of a meteorite trap in Dronning Maud Land (East Antarctica) on the Nansen blue ice field meteorite trap (2600–3100 m above sea level), where more than half of the Asuka meteorites have been collected. Based on 185 surface blue ice samples, one of the largest observed spatial patterns in oxygen isotopic variation to date is found. Relying on meteorites for which the terrestrial ages are determined using <sup>14</sup>C and <sup>36</sup>Cl, this surface ice is interpreted to date from the Last Interglacial up to the present-day. By combining state-of-the-art satellite derived surface velocities, surface mass balance modelling and ice flow modelling, we estimate that about 75–85% of the meteorites found on the ice field were supplied by ice flow after entering the ice sheet in an accumulation area of a few hundred square kilometres located south (upstream) of the ice field. Less than 0.4 new meteorites per year are supplied to the ice field through ice flow, suggesting that the hundreds of meteorites found 25 years after the first visit to this ice field mostly represent metorites that were previously not found, rather than newly supplied meteorites. By combining these findings, the infall rate of meteorites form space is estimated, which is in line with values from the literature, but situated at the higher end of the range. A comparison of the oxygen isotopic variation of the surface blue ice to that of the European Project for Ice Coring

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# Cosmic spherules from Widerøefjellet, Sør Rondane Mountains (East Antarctica)

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## Abstract

A newly discovered sedimentary accumulation of micrometeorites in the Sør Rondane Mountains of East Antarctica, close to the Widerøefjellet summit at ~2750 m above sea level, is characterized in this work. The focus here lies on 2099 melted cosmic spherules larger than 200  $\mu$ m, extracted from 3.2 kg of sampled sediment. Although the Widerøefjellet deposit shares similarities to the micrometeorite traps encountered in the Transantarctic Mountains, both subtle and more distinct differences in the physicochemical properties of the retrieved extraterrestrial particles and sedimentary host deposits are discernable (e.g., types of bedrock, degree of wind exposure, abundance of metal-rich particles). Unlike the Frontier Mountain and Miller Butte sedimentary traps, the size fraction below 240  $\mu$ m indicates some degree of sorting at Widerøefjellet, potentially through the redistribution by wind, preferential alteration of smaller particles, or processing biases. However, the cosmic spherules larger than 300  $\mu$ m appear largely unbiased following their size distribution, frequency by textural type, and bulk chemical compositions. Based on the available bedrock exposure ages for the Sør Rondane Mountains, extraterrestrial dust is estimated to have accumulated over a time span of ~1–3 Ma at Widerøefjellet. Consequently, the Widerøefjellet collection reflects a substantial reservoir to sample the micrometeorite influx over this time interval. Petrographic observations and 3D microscopic CT imaging are combined with chemical and triple-oxygen isotopic analyses of silicate-rich cosmic spherules larger than 325  $\mu$ m. The major element composition of 49 cosmic spherules confirms their principally chondritic parentage. For 18 glassy, 15 barred olivine, and 11 cryptocrystalline cosmic spherules, trace element concentrations are also reported on.

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# Evidence for the presence of chondrule- and CAI-derived material in an isotopically anomalous Antarctic micrometeorite

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Abstract-We report the discovery of a unique, refractory phase-bearing micrometeorite (WF1202A-001) from the Sør Rondane Mountains, East Antarctica. A silicate-rich cosmic spherule (~400 µm) displays a microporphyritic texture containing Ca-Al-rich inclusion (CAI)-derived material (~5-10 area%), including high-Mg forsterite (Fo<sub>98-99</sub>) and enstatite (En<sub>98-99</sub>, Wo<sub>0-1</sub>). The micrometeorite also hosts a spherical inclusion (~209 µm), reminiscent of chondrules, displaying a barred olivine texture. Oxygen isotopic compositions of the micrometeorite groundmass ( $\delta^{17}O = -3.46_{00}^{\circ}, \delta^{18}O = 10.43_{00}^{\circ}, \Delta^{17}O = -1.96_{00}^{\circ}$ ) are consistent with a carbonaceous chondrite precursor body. Yet, a relict forsterite grain is characterized by  $\delta^{17}O = -45.8_{00}^{\circ}, \delta^{18}O = -43.7_{00}^{\circ}, \Delta^{17}O = -23.1_{00}^{\circ}$ , compatible with CAIs. In contrast, a relict low-Ca pyroxene grain ( $\delta^{17}O = -4.96_{00}^{\circ}, \delta^{18}O = -4.32_{00}^{\circ}, \Delta^{17}O = -2.71_{00}^{\circ}$ ) presumably represents a first-generation silicate grain that accreted <sup>18</sup>O-rich gas or dust in a transient melting scenario. The spherical inclusion displays anomalous oxygen isotope ratios ( $\delta^{17}O$  =  $-0.98\%_{00}$ ,  $\delta^{18}O = -2.16\%_{00}$ ,  $\Delta^{17}O = 0.15\%_{00}$ , comparable to anhydrous interplanetary dust particles (IDPs) and fragments from Comet 81P/Wild2. Based on its major element geochemistry, the chondrule size, and oxygen isotope systematics, micrometeorite WF1202A-001 likely sampled a carbonaceous chondrite parent body similar to, but distinct from CM, CO, or CV chondrites. This observation may suggest that some carbonaceous chondrite bodies can be linked to comets. The reconstructed atmospheric entry parameters of micrometeorite WF1202A-001 suggest that the precursor particle originated from a lowinclination, low-eccentricity source region, most likely either the main belt asteroids or Jupiter family comets (JFCs).

## INTRODUCTION

Chondrules are mm-sized, ferromagnesian objects formed by repeated flash-melting events in the solar nebula (Gooding et al. 1980; Wasson 1993; Hewins 1996; Rubin 2000a). As such, they represent a valuable archive recording the pre-accretionary history of the solar nebula. Various models have previously been proposed to explain chondrule formation, including gamma-ray bursts (McBreen and Hanlon 1999); nebular shock waves (Ciesla and Hood 2002; Desch and Connolly 2002); planetesimal collisions and impacts (Sanders and Scott 2012; Johnson et al. 2015); or, more recently, radiative heating from molten planetesimals or

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### **Research** Paper

# Australasian microtektites across the Antarctic continent: Evidence from the Sør Rondane Mountain range (East Antarctica)



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## ABSTRACT

The ~790 ka Australasian (micro)tektite strewn field is one of the most recent and best-known examples of impact ejecta emplacement as the result of a large-scale cratering event across a considerable part of Earth's surface (>10% in area). The Australasian strewn field is characterized by a tri-lobe pattern consisting of a large central distribution lobe, and two smaller side lobes extending to the west and east. Here, we report on the discovery of microtektite-like particles in sedimentary traps, containing abundant micrometeorite material, in the Sør Rondane Mountain (SRM) range of East Antarctica. The thirty-three glassy particles display a characteristic pale yellow color and are predominantly spherical in shape, except for a single dumbbell-shaped particle. The vitreous spherules range in size from 220 to 570 μm, with an average diameter of ~370 μm. This compares relatively well with the size distribution (75-778 µm) of Australasian microtektites previously recovered from the Transantarctic Mountains (TAM) and located ca. 2500–3000 km from the SRM. In addition, the chemical composition of the SRM particles exhibits limited variation and is nearly identical to the 'normal-type' (i.e., <6% MgO) TAM microtektites. The Sr and Nd isotope systematics for a single batch of SRM particles (n = 26) strongly support their affiliation with TAM microtektites and the Australasian tektite strewn field in general. Furthermore, Sr isotope ratios and Nd model ages suggest that the target material of the SRM particles was composed of a plagioclase- or carbonate-rich lithology derived from a Paleo- or Mesoproterozoic crustal unit. The affiliation to the Australasian strewn field requires long-range transportation, with estimated great circle distances of ca. 11,600 km from the hypothetical source crater, provided transportation occurred along the central distribution lobe. This is in agreement with the observations made for the Australasian microtektites recovered from Victoria Land (ca. 11,000 km) and Larkman Nunatak (ca. 12,000 km), which, on average, decrease in size and alkali concentrations (e.g., Na and K) as their distance from the source crater increases. The values for the SRM particles are intermediate to those of the Victoria Land and Larkman Nunatak microtektites for both parameters, thus supporting this observation. We therefore interpret the SRM particles as 'normal-type' Australasian microtektites, which significantly extend the central distribution lobe of the Australasian strewn field westward. Australasian microtektite distribution thus occurred on a continent-wide scale across Antarctica and allows for the identification of new, potential recovery sites on the Antarctic continent as well as the southeastern part of the Indian Ocean. Similar to volcanic ash layers, the ~790 ka distal Australasian impact ejecta are thus a record of an instantaneous event that can be used for time-stratigraphic correlation across Antarctica.

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#### 1. Introduction

Tektites (and their submillimeter analogues microtektites) are natural, SiO<sub>2</sub>-rich glasses that form during oblique hypervelocity impact of

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asteroidal or cometary bodies with the Earth's surface (e.g., Koeberl, 1994; Artemieva et al., 2002). They are distributed over large geographical areas (>100-1000 km) commonly referred to as 'strewn fields', which have previously been linked to large-scale impact cratering events in North America, Ivory Coast and Central Europe (Glass and Simonson, 2013).

The Australasian strewn field is among the largest (>10% of the Earth's surface) and most recent (788.1  $\pm$  3.0 ka; Schwarz et al., 2016;

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# Annex 2. Internal Agreement on curation and research of Belgian Antarctic meteorites between VUB, ULB and RBINS

# BELAM : Curation and research of Belgian Antarctic meteorites

# Internal Agreement between VUB, ULB and RBINS

This internal agreement regulates the responsibilities of the partners and the supervision of the work for the different tasks within the work packages of the BELAM project. Reference is made to the technical specifications, annex 1 of the BELAM project, and to the up-to-date man-month scheme of the different tasks over the duration of the project (see attachment). In addition, all parties have concluded that a working stay of 6 to 8 weeks by the postdoc researcher at the National Institute of Polar Research in Japan, as early as possible, is necessary for the quality of the BELAM project.

It is understood that the postdoc researcher will be the person who executes all the tasks of the project except where explicitly stated otherwise.

<u>Work package 1: meteorite curation at RBINS</u>. In general, the RBINS is the supervisor of the work in this work package. The postdoc researcher will be based at RBINS in the first part of the project, about 17.5 months from the start of the contract of the new postdoc researcher (this is until September 2014). During this period, the researcher can spend some 20% of the time at ULB / VUB in connexion with his/her research (see the attached scheme of the man-month repartition over the duration of the project).

<u>WP 1.1. Task 1. Sample deposit protocol</u>. This is replaced by the "**Deposit agreement on Antarctic Meteorites between VUB, ULB and RBINS, including the functioning of a Scientific Loan Committee**" to be signed in parallel to the present agreement. This task does not involve the postdoc researcher.

WP 1.1. Task 2. Dedicated repositories, with different steps.

- Step 1, <u>organise the storage room</u>. This was realised before the end of 2012, under the supervision of RBINS, and with collaboration of the postdoc researcher.
- Step 2, <u>receiving and handling the samples</u>. The postdoc researcher will perform this task under the supervision of the curator of the collections of the RBINS. A distinction has to be made between samples that have already been classified at NIPR (steps 2 and 3 apply), and samples that still need classifying (for these the additional step 4 is to be performed).
- Step 3, <u>photographing and weighing</u>, and providing a short <u>description</u> (or checking the description provided by NIPR). It is done by the postdoc researcher under the supervision of RBINS. So far no samples have been handled in this way.
- Step 4, <u>making preparations</u> (simple polished blocks in Al-rings). This work is done by the postdoc researcher under supervision of RBINS, in the meteorite laboratory. In some cases polished thin sections (PTS) may be required: this is done in the laboratory of the ULB, under supervision of the ULB. Alternatively, it is commissioned abroad.
- Step 5, <u>making subsamples available for research</u>. This can be in response to an external request, or at the initiative of VUB / ULB. The decision to allow this step is taken by the <u>Scientific Loan Committee</u> (SLC). The subsamples are prepared by the postdoc researcher at the meteorite lab of RBINS, under the supervision of RBINS. This step includes the dispatching of the subsample and the follow-up on the loan and the resulting scientific

output. In the man-month scheme, this task is left in yellow colour during 2015-2016, even if no figure 0.5 has been filled in, meaning that it will be executed as needed.

WP 1.2. Task 1. <u>Identifications</u>. The classification of unknown meteorites is executed by the postdoc researcher under the supervision of RBINS in the case of ordinary chondrites (this is about 80% of the meteorites), and with joint supervision by ULB and VUB in the case of rare meteorite types. The SEM/EDS facilities of RBINS, VUB and ULB can all be used as a function of availability, and with adequate planning. During a working stay of 6 to 8 weeks at the NIPR laboratory in 2013, the researcher will get familiar with the curation and classification procedures of NIPR, thus improving the RBINS procedures.

WP 1.2. Task 2. <u>Database</u>. The Darwin database of the RBINS will be used by the postdoc researcher under supervision of the curator of RBINS. Managing the overall Darwin database remains the responsibility of RBINS. The diffusion of the Antarctic meteorite database will be coordinated with NIPR. The loans interface for the public (webpage) will be hosted at RBINS but the content will be jointly managed by the curation team (Scientific Loan Committee and postdoc researcher).

WP 1.3. Task 1. <u>Handbook of quality control of curation</u>. This internal document of RBINS, meant for successors of the postdoc researcher, will be completed after some experience has been gathered in each of the other tasks of WP1, and after the working stay at NIPR. The final edition will be compatible with the NIPR methodology, and supervised at the RBINS. The role of the SLC (scientific loan committee) will be described in detail. Only part of the handbook will be public, e.g. the rules for loans.

WP 1.3. Task 2. <u>Establishing the RBINS as a curation center for Antarctic meteorites</u>. An international workshop will be organised in the second half of 2014 by the postdoc researcher at the RBINS, under the supervision of RBINS, but in collaboration with VUB and ULB.

<u>Work package 2. High-quality research in Belgium on Antarctic meteorites</u>. The ULB and/or VUB will be the supervisors of this work. The nature of the research shall be adapted to the research profile of the postdoc researcher. The postdoc researcher will be based either at ULB or VUB in function of the practical tasks to be performed. He/she will come regularly to the RBINS to continue meteorite identifications, databasing and its follow-up, and to use the meteorite lab for sample preparations.

# General statement

During the first period until 30 September 2014, when based at the RBINS, the postdoc researcher will report his/her activities and absences to the project coordinator at RBINS, even if formal leave of absence will have to be asked at the VUB. An electronic agenda for internal use is available at the Geological Survey (RBINS) for this purpose. During the second period starting 1 October 2014, when based at VUB or ULB, he/she will report activities and absences to either VUB or ULB.

Using e-mail, all partners of the project (RBINS, VUB, ULB) will be informed by the researcher of his/her whereabouts. This also applies to research activities carried out at ULB or VUB.

# Annex 3. Deposit agreement on Antarctic Meteorites between VUB, ULB and RBINS, including the functioning of a Scientific Loan Committee

# Deposit agreement on Antarctic Meteorites between VUB, ULB and RBINS, including the functioning of a Scientific Loan Committee

Recognising the signature on 3 November 2009 of an agreement between Japan and Belgium for the common curation and research on Antarctic meteorites collected in the framework of project JARE-51 (2009-210), whereby these meteorites would be shared between NIPR (National Institute of Polar Research, Japan) and VUB on the principle of equal basis for research,

Given the minutes of the meeting of 3 February 2011, concerning the research program BELISA-SAMBA supervised by Belspo, and which provide broad outlines of an agreement between RBINS on the one hand and VUB/ULB on the other hand,

Recognising the need for a Scientific Loan Committee to decide on the allocation of Antarctic meteorite samples for research, in consultation with NIPR,

Recognising the start of the BELAM project on 1 March 2012 supervised by Belspo, and the Belsposupported installation of a storage facility and meteorite laboratory at RBINS during 2012, allowing the Antarctic meteorites to be stored at adequate stable conditions,

Recognising the successful meteorite prospection campaigns carried out in Antarctica by joint Japanese-Belgian teams in 2009-2010, 2010-2011 and 2012-2013, and the plans for further prospection in the years ahead,

Given the will of RBINS to develop mineralogical capacities, in synergy with the universities, suitable for externally-funded meteorite research,

Given the new MoU between NIPR-AMRC, RBINS, ULB, VUB, for further collaboration after JARE51, which is presently being negotiated.

Given the preparatory meeting of 4 February 2013 where a consensus was reached on the general principles of the present deposit agreement, and further specifications on 1 March 2013,

The Vrije Universiteit Brussel (VUB), the Université Libre de Bruxelles (ULB) and the Royal Belgian Institute of Natural Sciences (RBINS) agree as follows:

Article 1. The Belgian share of the Antarctic meteorites collected jointly with NIPR become Belgian state patrimony as soon as they reach the Belgian territory and are deposited at RBINS. RBINS takes care of their curation.

Article 2. VUB/ULB supervise the repatriation of the Belgian share of the Antarctic meteorites from Japan to Belgium. RBINS supervises shipping of the individual batches of meteorites from NIPR to RBINS and manages the associated shipping list, which should include the name, provisional or not, the weight, classification if applicable, and all other useful information concerning each meteorite. VUB/ULB and RBINS inform each other of every new step in this transfer of samples.

Article 3. All requests for research and for exhibitions shall be addressed to the curator of the geological collections of RBINS, and will be subjected to the internal rules of the RBINS collections, completed with NIPR guidelines for sample allocation adapted to RBINS.

Article 4. A Scientific Loan Committee (SLC) is set up to decide about the validity of the requests and the allocation of samples for research and exhibitions. This SLC is composed of three members, or their representatives : Philippe Claeys representing VUB, Vinciane Debaille representing ULB, and Marleen De Ceukelaire, curator of the geological collections of RBINS. As a rule, decisions are taken by majority, but consensus is encouraged. The SLC can conduct most of its business on a case-by-case basis by e-mail, but it can convene whenever one of its members wishes to do so. The SLC will have a formal meeting at least once a year. A two-way communication is established with NIPR in Tokyo, to inform each other of all requests that have been received concerning the joint Japanese-Belgian meteorites, so as to avoid double allocation. The specific modalities of this communication with NIPR will be worked out by the SLC, together with the curator-in-charge of NIPR. Detailed guidelines for the functioning of the SLC will be described in the "handbook of quality control of curation" to be produced by the BELAM project.

Article 5. VUB and ULB enjoy a priority of research on these Antarctic meteorites until 31 December 2017. VUB/ULB can also obtain a long-term loan of certain Antarctic meteorites for didactical purposes, to be decided by the SLC.

Article 6. For the duration of the BELAM project, the postdoc researcher hired by this project will share his/her time between research and curation. In the field of curation, he/she will prepare the samples for expedition or for research inside Belgium. He/she will receive all necessary instructions by the SLC and may be invited to the SLC meetings for practical reasons without decision right. He/she will be contribute to the Antarctic meteorite database and its diffusion through the Internet, and will collect all scientific output on the Antarctic meteorites, under the supervision of the curator of RBINS. He/she will also be in charge of training permanent collaborators of RBINS (scientists or technicians), as well as some staff and students of VUB/ULB , in the specific know-how of handling Antarctic meteorites and using the instruments in the meteorite lab.

Article 7. Duration. The SLC is established for a period of five years, until 31 December 2017. After that date, the composition of the SLC will be adapted in order to become a national expertise-based committee, and will invite other members, involved in Belgian meteorite research, for their particular expertise.

Signed in Brussels, on March 2013

For VUB

For ULB

For **RBINS** 

Ph. Claeys

V. Debaille

C. Pisani, general director

# Annex 4. MoU between NIPR, VUB, ULB, RBINS concerning scientific cooperation and ways of sharing the Antarctic meteorites



The National Institute of Polar Research, Research Organization of Information and Systems (NIPR), Japan, the Vrije Universiteit Brussel (VUB), Belgium, and the Université Libre de Bruxelles (ULB), Belgium, collaborate on meteorite research under the umbrella of the Statement signed in Tokyo in 2005 between the Japanese Ministry of Education, Culture, Sports, Science and Technology and the Belgian Ministry of Economy, Energy, Foreign Trade and Science Policy. In 2009, NIPR and VUB signed an agreement regarding the sharing of the meteorite collected by Japanese and Belgian scientists in Antarctica during the 51st Japanese Antarctic Research Expedition (JARE).

Recognizing the successful collaboration in collecting meteorites in Antarctica during the past years, NIPR, VUB and ULB want to expand their collaboration in the coming years, and to include the Royal Belgian Institute of Natural Sciences (RBINS), where the Belgian share of the meteorites will be deposited for curation purposes.

## Article 2

## Items of cooperation

1. The meteorites collected through the Antarctic campaigns in the Sør Rondane Mountains in the JARE-51 mission and subsequent joint missions, will be shared evenly between the two countries. This sharing will be accomplished by cutting in half the larger samples (> 50g). The smaller samples (<50g) will not be cut to avoid wasting precious small samples. They will be shared evenly between Japan and Belgium based on either total weight or total number. Priority access for research to the samples curated by the other partner is guaranteed. Specific sharing agreements will be tailored according to the circumstances, for the largest samples (> 4kg) because of their importance in terms of outreach and exhibits. Cutting them should be avoided, except for classification purposes.

2. All meteorites collected during the joint Antarctic campaigns – JARE-51 and subsequent ones – will first be sent to NIPR. Initial processing (defreezing, weighing, measuring, photography, provisional naming, short macroscopic description) of each meteorite will be carried out at Antarctic Meteorite Research Center (AMRC), NIPR in Tokyo. As this task is completed, AMRC-NIPR will make all possible efforts to cut the larger meteorite samples (>50g) and send the Belgian share to RBINS in batches of 30-50 meteorite samples within a reasonable time. If possible this timing should not exceed 15 months after the closing of each campaigns or for the 2009-2010 and 2010-2011 campaigns, the signature of the present MOU.

3. AMRC-NIPR and the RBINS agree on sharing the task of classification, following the existing AMRC-NIPR procedure, sample preparation (thin or polished sections) and analyses (ex. EPMA). Within the frame of the Belgian Antarctic Meteorites (BELAM) project, classification expertise will be

transmitted to the RBINS. Meteorite classification carried out under the full responsibility of RBINS will first be sent by RBINS to AMRC-NIPR for assessment, before being transmitted to the Meteoritical Society Nomenclature Committee for official approval. The ULB and VUB will assist in the classification process of out-of-the-ordinary identifications.

4. AMRC-NIPR and the RBINS agree on sharing the task of curation. AMRC-NIPR and the established Belgian curation system at the RBINS will harmonize their distribution of meteorite samples to avoid redundancy and duplication of requests. The existing regulations of NIPR will stand as a model for finalizing RBINS meteorite-allocation regulations. The Belgian Parties have set up a Scientific Loan Committee (SLC), which includes VUB, ULB and RBINS representatives. The SLC will handle the loan requests in coordination with the Committee at AMRC-NIPR. Before any decision, AMRC-NIPR and the SLC will systematically inform each other of all loan requests and will regularly keep each other informed of the loan follow-up.

5. Efforts will be made to establish strong research collaboration between NIPR and ULB-VUB scientists through the exchange of students and researchers. The Parties will mutually open their research facilities to their students and researchers. The Japanese and Belgian teams will discuss their research plans to avoid duplication and excessive use of sample material.

The meteorites will be referred to in publications and exhibitions as "collected during joint Japanese-Belgian meteorite expedition".

## Article 3

## Validity of this MoU

This MoU shall enter into force upon signature by all Parties and is valid for a period of five years, which will be renewed by tacit agreement for periods of five years. Any Party can decide to end the MoU at any given time, after notifying the partners. In that case, the remaining Parties shall negotiate the conditions for a new MoU, if they wish to do so. The discontinuation of the MoU by one of the partners does not revoke its right and duties for the campaigns already carried out; in particular in such case all efforts should be made by the withdrawing partner in order not to hamper the tasks of the remaining partners. Any amendments affecting the MoU shall be clarified among the Parties.

Kazuyuki Shiraishi, Director-general National Institute of Polar Research Tokyo, Japan Philippe Claeys Professor Vrije Universiteit Brussel Brussels, Belgium Vinciane Debaille FNRS Researcher Université Libre de Bruxelles Brussels, Belgium Camille Pisani Director-general Royal Belgian Institute of Natural Sciences Brussels, Belgium

Date:

Date:

Date:

Date: 2/2

# Annex 5. Meteorite laboratory user manual

# METEORITE LABORATORY USER MANUAL



**RBINS 2013**
This book has been compiled by <u>Celine Martin</u>, Curator of the Antarctic Meteorites at the RBINS until December 2012, <u>Ivan Petrov</u>, PhD Student at the ULB, <u>Claudio Ventura Bordenca</u>, PhD student at the VUB, <u>Walter De Vos</u>, PI of the Belam project until September 2013, <u>Pittarello Lidia</u>, Curator of the Antarctic Meteorites at the RBINS from April 2013, and <u>Thierry Leduc</u>, lab technician. The book has been also checked by <u>Sophie Decrée</u>, the scientific responsible of the meteorite lab, whom any use of the lab has to be reported to. **Remember also to fill the record book in the lab at any visit with the required information**.

**Disclaimer.** This manual describes how polished sections of meteorite samples are prepared at the Meteorite Laboratory of the Royal Belgian Institute of Natural Science (RBINS) of Brussels at the time of its drafting. Therefore, this manual might subject to updates and changes and might not apply in future with the improvements of the meteorite lab.

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# 1. Cutting with Lapidary Trim Saw

(Manual with instructions and parts list available, EN)

This saw is used for samples that do not fit in the Diamond Wire saw and for meteorites with a an amount of metals larger than ordinary chondrite. Iron and stony-iron meteorites are cut with a special CBN blade, which requires an apposite coolant. Follow the instructions on the coolant bottle for dilution.

#### 1.1 Blade installation

- Remove the cover unscrewing the 4 screws at the corners and the mud flap (Fig. 1).
- Slip the blade into the saw slot at an angle and slide it onto the shaft (Fig. 2).
- Slide the second flange next to the blade (Fig. 3). Tighten the arbor nut securely using a monkey wrench (Fig. 4). Do not over-tighten as this can warp the blade.









#### 1.2 Operation

- Fill up the coolant tank with methanol (for precious meteorites), demineralized water (for non-Antarctic meteorites), or a mixture of the specific coolant for the CBN blade and demineralized water if the CBN blade is used, until the coolant covers the blade approximately 1 cm (Fig. 5). If methanol is used, using a small container that fits under the saw can reduce the amount of necessary liquid.
- Place the cover of the Trim Table Assembly (Fig. 6). Lower the splash shield over the blade and fix it with bolts using a screwdriver (Fig. 7).
- Cut slowly using a straightforward motion. Do not force stone into blade; do not apply side pressure on stone (Fig. 8). If for a polished section, cut a slice of max 4 mm of thickness and that fits in the Al-ring used as support.
- Rinse immediately the stone piece with methanol (for Antarctic meteorites) or demineralized water followed by acetone (for desert meteorites).
- After having cut the sample, stop the blade, and remove the residual stone pieces and liquid from the Trim Table Assembly.









#### 1.3 Cleaning

• Remove both cover and blade, and wash them with tap water. Leave them drying.

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• Place the complete Trim Saw on the sink (Fig. 9). Undo the drain plug to evacuate the coolant (Fig. 10). If methanol was used, save the remaining liquid after use: pass it through a coffee filter and collect in a respective bottle for recycling. Rinse inside the Tank Assembly with demineralized water and mop it up with paper.





# 2. Cutting with the Diamond Wire Saw

(Manual with instructions and parts list available, FR & EN)

Normally the wire is already installed. If not, please read first sections 2.2 and 2.3 and then proceed with the 2.1.

#### 2.1 Operation

- Assemble the two casings over the rotating wheels (Fig. 11) and place the rectangular tray beneath them (Fig. 12), which serves as cooling bath for the diamond wire.
- Fill up the bath with a coolant (preferably methanol, the recycled methanol for Antarctic Meteorites is saved in a specific bottle, alternatively demineralized water can be used; Fig. 13). The lower part of the right wheel must be submerged 0.5 cm in the coolant. Add 1 cap of the 'Well' liquid to the coolant as lubricant (Fig. 14-16).



- Secure the meteorite to the sample holder. Two sample holders are available: one is flat and suitable for slices of material and the other is a ring for samples with irregular shape. At this stage the precise geometry of the cut is determined. In most cases fixing the sample will require the use of small wooden pieces between the screw and the rock. In this way, the sample is firmly fixed and damage or contamination from the screw is avoided.
- Fix the sample holder into the balancing rod, at a height between the two wires of the loop (Fig. 17 and 18); take care not to touch the diamond wire. Adjust the back-/forward position of the sample, first with two screws for rough positioning, then fine-tuning with the rotating ruler (Fig. 19; 1 turn = 0.5 mm; typically a slice of 3 to 4 mm thickness is used for polished section).
- Set the small weight of the balancing rod to 0. Adjust the big weight of the balancing rod so
  that it is horizontal (left and right sides are balanced and the sample does not touch the





wire). Adjust the small weight of the balancing rod so that the sample goes (from below) against the upper wire – typical starting setting is 80 (Fig. 20 and 21).

- Check if the instrument is switched off and the speed is set to 0 (Fig. 22). Turn on the starting key (on the left side) and press the "start" button (on the right side), while still manually holding the sample away from the wire (Fig. 23).
- Set the speed to 6-7 to start moving the wire. Carefully place the sample against the wire until a cut is formed (Fig. 21).
- Release the sample holder and set the speed to 8.5 (Fig. 24). Never work with speed higher than 9.

**Note**: Samples often drop coolant outside the bath. Please, place a tissue or a small red container to protect the engine and save bath liquid.



 Periodically look at the coolant level and adjust if not enough (methanol evaporates quickly). Wire must be wetted at all time while cutting.

**NOTE:** Do not change anything once the cutting process starts – do not touch the wire, sample, rod, or ruler, etc. If the cutting progress is slow, lower the speed to 0, carefully detach the sample from the wire and set the small weight to higher values (first to 90; never more than 100, to avoid

wire break). Set the speed to 6 and bring the wire and sample in contact again. Then set the speed to 8.5. A new wire cuts much faster than a used wire.

• When the cut is finished, the meteorite slice will fall into the bath. Stop the machine. Take the meteorite slice out of the bath with tweezers, rinse with methanol (or demineralized water followed by acetone if the bath was filled with water) and place in acetone in the ultrasonic bath (USB) for 5 min, in order to remove any remaining impurities due to the cutting process (Fig. 25 and 26). The sample slices are placed in small red flexible plastic bowls, filled with acetone (put some acetone first, than the sample face down), which are left to float in the bath, which is filled with demineralized water. At the end, take the slices out with tweezers and leave to dry on clean paper.





- Recuperate remaining methanol from the cooling bath of the diamond wire after use pass it through a coffee filter to remove most of the dust, and collect in a bottle for the next use of the saw. Do not recuperate deionized water if used as a coolant.
- The larger piece of the sample, on the sample holder, should be detached and rinsed with either methanol, or demineralized water followed by acetone, and left to dry on clean paper. For documentation, weigh both the dry sample and the slice and compare to the original weight.

#### 2.2 Maintenance

#### 2.2.1 Winding the diamond wire

Before starting the procedure, please make sure to have all supplied tools readily available including two pair of pliers (flat nose and wire cutters), Allen wrenches and retaining clamps (Fig. 27).

**IMPORTANT**: Treat the diamond wire with EXTREME care! Do not bend the wire if not required. If any kinks develop it is better to scrap that portion of the wire and begin again. Before proceeding, ensure that the hand crank is in the start position with the winding cylinder extended to its outermost position. The reference pin on the winding unit is properly aligned at its "start" position when it is at 3 o'clock position or, to describe it another way, the reference pin on the winding cylinder will be at its farthest position from the guide roller.



Fig.27

- Usually a wire with Ø 0.17 mm is used. Attention must be paid when the wire is raveled on the bobbin; keep the rolled wire with a thumb, otherwise the wire could unravel completely and thereby become unusable (Fig. 28).
- Place the wire bobbin containing the spare diamond wire on the wire bobbin receiver and secure with the washer and knurled screw in the center hole (Fig. 29). The bobbin must be placed on the wire bobbin receiver so that the wire runs parallel to the winding unit and is aligned with the channeled guide roller at the opposite end of the casing. With the wire bobbin in place, and the wire being held firmly by the tensioning roller, unwind approximately 8 to 12 inches of the diamond wire (leader) from the spare wire bobbin. (It may be necessary to rotate the spare wire bobbin by hand as the wire is being unwound).



• If the drum onto which the wire is to be wound is attached to the winding unit, loosen the plastic knurled screw and remove the drum. Loosen both Allen bolts on the circumference of the drum approximately 1½ turn using the proper sized Allen wrench (Fig. 30). Feed the leader into the slot next to the Allen bolt where the locating pin is adjacent and wrap the wire one-half revolution around the threads of the Allen bolt (Fig. 31). While holding the wire in place, tighten the Allen bolt (Fig. 32). Trim any extraneous wire so as to prevent injury or any interference of the wire. The wire should now be in the slot adjacent to the pin and held firmly in place by the Allen bolt.



• Now place the drum on the winding cylinder. With the wire now protruding from and aligned to the slot, carefully hold the wire against the pin and direct the wire into the groove as close as possible to the pin in the direction of the guide roller (Fig. 33).

**<u>HINT</u>**: Applying pressure with a fingernail to the wire along the radius of the pin while guiding the wire into the groove with the other hand usually works best (Fig. 34). Insure that the wire follows the same groove of the drum as it is directed back towards the guide roller. The drum will fit only one way due to the locating pin.







 Guide the wire towards and into the channel of the guide roller, and then back towards the spare wire bobbin (Fig. 35). Once the wire is properly positioned on the drum and the guide roller, moderate tension can be maintained on the wire with one hand while any remaining slack can be taken up by turning the spare wire bobbin (Fig. 36). Now that tension on the wire is obtained, it is important that the hand crank not be moved in the opposite direction. If this happens the wire will unwind from the drum.

NOTE: At this point check to insure that the wire is:

1) firmly held against the pin;

2) filling the groove closest to the pin;

3) filling the same groove running directly back to the guide roller.

If so, and if there are no bends or kinks in the wire, proceed to the next step. If unwanted bends or kinks are in the wire, it is best to begin the process again.





• The winding of the wire onto the drum can now begin. Carefully and slowly turn the handle in a counter clockwise direction (Fig. 37), insuring that:

1) each groove of the cylinder is being filled and,

2) no overlap of wire occurs.

Proceed until the handle and reference pin reaches the "stop" position, which is the same 3 o'clock position as the "start" position.

It is now necessary to use both retaining clamps to hold the new wire in the drum (Fig. 38).
 Place one retaining clamp on either side of the drum by inserting the curved part of the clamp in the hole and the straight portion carefully over the wire (Fig. 39-40). Care must be taken so that damage does not occur to the wire and that wire is not pushed away from a grove.



- It is necessary to complete the winding process by hand. Detach the winding unit from the table and turn it, so that the drum faces up. With the retaining clamps holding the wire on the drum and the wire still being engaged by the guide roller, release enough wire from the bobbin to extend over the top of the drum plus approximately 6 inches. While holding the wire with the right hand momentarily remove the retaining clamp closest to the winding unit, and, with the left hand, feed the wire into the next available groove. Once the wire is in this groove, replace the retaining clamp immediately.
- Lead the wire into the groove making ¼ turn up to the slot near the Allen bolt. Feed the wire into the slot and wrap the wire around the Allen bolt one-half turn. Tighten the Allen bolt securely. Trim the wire with the supplied wire cutters as close to the Allen bolt as possible and tuck any remaining wire close to the Allen bolt so as to prevent accidental cutting of the hands. Make sure the wire on the bobbin is fed through the small hole on the side of the bobbin wall so that the remaining wire does not unwind from the bobbin (Fig. 41).
- Check if the number of wire tension weights installed in the wire saw is correct (see p.4 of the English manual of the wire saw). NOTE: there are mistakes in the French and English manuals, on pages 6 and 4, respectively, concerning the wire tension weights for wire Ø 0.17 mm in fact, we need only 1 (one) tension weight for wire Ø 0.17 mm.
- The drive unit of the wire saw must be rotated so that it is, as much as possible, inside the motor housing and the reference pin is located as far away from the guide roller as possible.
- The wound drum can now be removed from the winding unit. Never remove the drum without securing the wire on it with retaining clamps. Check that the drive unit and the guide roller of the wire saw are without casings. Loosen the knurled screw that secures the drum on the winding cylinder, and remove the drum with the retaining clamps still on it. Carefully remove the wire from the guide roller, while keeping it extended by hand, and exercise



great care to neither bend nor cause the wire to kink (Fig. 42). The drum is now ready to be mounted on the Diamond Wire Saw.



2.2.2 Mounting the Wire Drum



- Check that the drive unit is pushed as inside as possible, towards the engine. Place the drum on the receiving part and fix it with the central thumbscrew. The wire drum is designed to be fully seated on the receiving portion so it may be necessary to rock the drum back and forth as the screw is being tightened.

- Push the guide roller towards the wire drum, place the wire into the groove, and gradually allow the roller to return to its weighted position.



- At this point the wire tension should be taut. If so, the two retaining clamps can now be carefully removed from the drum. Inspect the wire in the drum to insure there are no void grooves or overlapping wire.

- Manually rotate the drum counter clockwise two-three turns while checking the concentricity. If the drum is not rotating concentrically rotate the drum back to the start position, replace the clamping pins and begin the drum mounting procedure again. Notice that if the big wheel is at an end position, the wire cannot start moving.

- carefully place the casing splashguards over the drum and guide roller by aligning the slots for the wire to move into. Fix them with the screws.

- With the motor velocity adjustment knob turned completely to the OFF position, turn the key that is located on the rear panel of the instrument to the ON position. Then slowly turn the velocity adjustment control knob clockwise so that the motor speed and wire begins rotating at a slow velocity. After two or three changes of direction the speed can be increased.

# 3. Labeling the Al ring

- Connect all parts of the carver PROXXON and assemble the pit.



- Carve firmly the name on the side of the ring. Please, use a combination of letter and numbers that cannot be readable on reverse.

# 4. Preparation of sample embedded with resin in an Al-ring

Three resins are available: araldite, EpoFix and a polymer resin. We do not treat here the latter; the choice between the first two depends on the planned analytical techniques. EpoFix might cause troubles with the high vacuum required for SIMS, while araldite might react with the silica gel (syton) used for the fine polishing required by EBSD analysis. On the other hand, EpoFix is easier to use, as it is less viscose, therefore almost no bubbles form or they remain in the far part of the resin (close to the ring) after solidification. Araldite generally evidenced form bubbles in the vicinity of along the sample surface that could then interfere with the polishing. (EpoFix is not currently available in the meteorite laboratory, but could be asked from Dr. Thierry Leduc in the Department of Mineralogy lab on the seventh floor).

The first steps of this procedure are identical with both resins.

- After samples were rinsed in acetone in USB, let them dry in an oven at 40°C overnight.

- Stick a 2-side sticking tape (e.g. Tesa) on a rectangular preparation glass (3 cm x 4.5 cm). Do not remove the protective layer of the second sticking side. (Generally the preparation glasses are ready for the next step).



- Stick another tape on the first and remove the protective layer. In this way the second tape will be easily removed when required.

- Choose which side of the meteorite will be studied, with a preference for the fresh cut surface. Take the sample carefully with tweezers and stick this side on the tape, as centrally as possible. Press it to stick firmly (otherwise some resin could flow <del>sneak</del> between the sample and the tape and jeopardise the sample quality). - Stick the corresponding AI ring on the tape, with the sample

at the center, with the sample name upside down (downward). Press it to stick firmly (otherwise the resin could leak out sneak between the ring and the tape).



#### 4.1 Embedding in araldite



- Prepare the resin as follows:

- set the hot plate to 80°C;
- use gloves and 2 different syringes to mix 4 mL resin Araldite with 1 mL hardener in a glass jar on the hot plate (this will be sufficient to fill 3 rings);
- place the jar on the hot plate and stir well (3-5 min) with a wooden stick. Try to remove all bubbles (they could interfere with later analysis).

#### 4.2 Embedding in Epofix

- Prepare the resin as follows:

- use gloves and 2 different syringes to mix 7.5 mL resin EpoFix with 1 mL hardener in a glass jar;
- stir well (3-5 min) at room temperature the mixture. Try to remove all bubbles (they could interfere with later analysis).

(in both cases)

- Pour the resin drop-wise on the sample, using the wooden stick. Add resin until the Al-ring is completely filled. Be very careful to avoid the formation of bubbles. If bubbles are formed, try to pry put them out with a needle.



- Wash the syringe for hardener with water and keep for next use. Discard the syringe for the resin. Mop up inside the glass jar with paper.

- Let the resin harden overnight at room temperature. In the case of araldite, overnight heating at 50° C in the oven is an alternative.

- On the following day, remove the 2<sup>nd</sup> stick tape (together with the ring) from the glass plate. The glass remains with the 1<sup>st</sup> tape and is ready for other rings/samples.



- Separate the ring from the 2<sup>nd</sup> tape. The sample is now ready for polishing – first by hand and second with a polishing machine by an electric polisher.



# 5. Sample polishing

5.1 Manual polishing

- Prepare round SiC papers with mesh size 180, 500, 800, 1200, 2400, and 4000 (mesh size 4000 corresponds to SiC grain size of 6  $\mu$ m).



- Start with mesh size paper 180. Place it on the table with the abrasive part on top and wet the surface with a sprinkle of methanol. Start to polish the sample surface by moving the ring only in one direction (scratches would result parallel) for 3-4 min. Add some Pour methanol whenever the paper is dry.

- Rinse the ring with methanol and observe the progress of polishing with the reflected light binocular microscope (<u>Tip</u>: observe the metallic grains pieces – they act are like a mirror while the light direction is changed). Switch to paper with mesh size 500 when all resin is removed from the meteorite surface; before doing so it, never forget to wash the previous SiC-paper with water and a toothbrush, rinse it with demineralized water and mop it up with a paper towel. At every new paper mesh size, rotate turn the polishing direction of 90°, so the new scratches will be perpendicular to the previous ones and you can check the progress of polishing under the binocular. Use in sequence the next SiC papers until mesh size 4000. Polish for ~2 min with each paper (it takes longer with mesh size 180).



- Put the meteorite samples under acetone in the USB for 5 min.

- Prepare a plastic box with label and fixing gum Blue Tack for each sample ring.





#### 5.2 Mechanical polishing (Struers DP10) Struers DP10 is located in the Mineralogy lab, Floor 7 of the main building)

- Two polishing operations are carried out, one with diamond particle size of 1  $\mu$ m, and one with 0.25  $\mu$ m (1/4  $\mu$ m). Use polishing clothes for the respective diamond suspension in methanol.



- Place the polishing cloth for 1 µm particles on the magnetic disk of Struers DP10.

- Lower the 6-place sample holder on the polishing cloth. Introduce the Al rings in each place and secure with the pins. Set the weights as they provide the required pressure on the rings.



- Dispense 2 volumes of 1  $\mu$ m particles suspension (DP-Suspension A) with a Pasteur pipette with a fine tip. Start the Struers DP10 at a speed of 125 rotations/minute and dispense methanol on the polishing cloth when it looks dry. Polish for 15 min.

- Clean each ring to remove the polishing suspension using clean paper with methanol or acetone.

- Repeat steps 2-5 with 0.25  $\mu m$  particles suspension and the respective polishing cloth. Polish for 15 minutes.

- Observe the samples with the reflected light microscope. All scratches from hand polishing should be gone.

- Wash the samples with acetone in USB bath for 5 min. Take them out with tweezers and leave to dry. Do not touch the polished surface with bare hands anymore. Weigh each sample and keep it in its labelled plastic box, pressing the lower side gently onto the Blue Tack gum.

- Wash the polishing clothes with methanol after finishing work, and leave to dry.

# Annex 6. Loan form

**MUSEUM** Geological collections

Curator of the meteorite collection Marleen DeCenkelarie Royal Belgian Institute of Natural Science marleen decenkelarie@maturalsciences.be To be sent to:

f

# Requested material:

| Sample name       Preferred weight       Afninum weight       Sample form       Sampling instructions         Image: Sample form         Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form         Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form         Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form         Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form         Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form         Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form         Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form         Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form         Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image:    |
|--|
| Preferred weight       Minimum weight       Sample form       Sampling instructions         Image: Sample form       Sample form       Sample form       Sampling instructions         Image: Sample form       Image: Sample form       Sample form       Sample form         Image: Sample form       Image: Sample form       Sample form       Sample form         Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form         Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form         Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form         Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form         Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form         Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form       Image: Sample form |
| Minimum weight Samplie form Sampling instructions  |
| Sample form Sampling instructions  |
| Sampling instructions  |
|  |

> This project will concern destructive analyses

The principal investigator will take the responsibility of returning the samples (in case of non-destructive analyses) immediately after the planned measurements

The principal investigator will take the responsibility of acknowledging the Royal Belgian Institute for Natural Sciences in any publication related to the samples requested in the present proposal. He/she will send a copy of the publication to the curator.

2

| Geological collections   | te material for analysis | nd phone number) <sup>1</sup>                    | names, e-mails)                                   |                              | d description of the methodology, including the   |  |
|--|--------------------------|--|---|------------------------------|---|--|
| To be sent to:<br>Currator of the meteorite collection<br>Marken DeCenkelaire<br>Noval Delegan Institute of Natural Science<br>andeen decentelaire@natural.sciences.be | Request of meteori       | Principal investigator (name, address, e-mail, a | List of the researchers involved in the project ( | Name of the project / thesis | Scientific justification of the sample request an<br>previous experience of the research team |  |

The principal investigator must hold a permanent position in a recognized scientific institution

# Annex 7. Reports of the Follow-up Committee meetings

# Belgian Antarctic Meteorites : curation and research (BELAM) project: Report on the Followup meeting of 10 October 2013

# **Participants**

# Members of the Follow-up Committee:

Brigitte Zanda, curatrice en chef collection metéorites, Muséum national d'Histoire naturelle, Paris Luigi Folco, prof. University of Pisa, Italy Christian Koeberl, prof. University of Vienna & general director Naturhistorisches Museum, Vienna

# **BELAM project:**

Maaike Vancauwenberghe, BELSPO project officer Vinciane Debaille, prof. Université libre de Bruxelles Philippe Claeys, prof. Vrije Universiteit Brussel Lidia Pittarello, postdoc researcher Vrije Universiteit Brussel Camille Pisani, general director Royal Belgian Institute of Natural Sciences Marleen De Ceukelaire, RBINS geosciences collection manager Thierry Leduc, RBINS, laboratory of mineralogy Michiel Dusar, RBINS, BELAM project coordinator (replacing Walter De Vos from October 1<sup>st</sup> onward)

Meeting place: RBINS Geological Survey building meeting room, Brussels

# **Comments by the follow-up committee**

# **On project organisation:**

The organisational set-up of the BELAM project is considered exemplary for combining Antarctic meteorite collecting and research and a very effective means of building a collection. Moreover, organisation of meteorite search expeditions is the only way to increase the collection of Antarctic meteorites. For the Belgian government on the other hand, the value of the collection is a return on investment for its considerable budgetary effort in building and maintaining a polar research station.

#### Staff:

The follow-up committee appreciates the efforts so far of establishing a repository – as a condition sine qua non for building and managing a collection for the scientific community – but insists on the importance of a <u>dedicated curator</u>, specialised in meteoritic research, for the full valorisation of the meteorite collection. Such a function is certainly necessary once the full Antarctic collection is available.

The follow-up committee questions therefore the distinction made between curation and research: museum and universities should cooperate more closely. The benefits of a full-time scientist at RBINS for the valorisation of the collection have been underestimated in the BELAM project. The follow up committee urges both RBINS and Belspo to find a stable, long-lasting, solution on the mid-term.

# **Repository**:

The number of meteorites collected during the successive field campaigns is high; therefore these campaigns are considered very successful. On the other hand, the follow-up committee has feeble understanding for the complicated process of sample transfer from Japan to Belgium [which may slow down the execution of further tasks of the BELAM project]. The classification system applied by NIPR is correct but slow, given the large number of meteorites to be classified. This means that meteorite

samples are available for research at a slow rate So it might be better to do some more classification in Belgium.

The follow-up committee insisted that investment for valorising of the meteorite collection should not be restricted to Antarctic meteorites but should be beneficial for all meteorites. This involves the management chain of the utilisation of the collection, the curator in charge, the loan system, the databasing. As an example, the Scientific Loan committee set up for the BELAM project (SLC) should function for all meteorites, not only for the Antarctic ones ('all meteorites treated equally').

### Belgian Antarctic Meteorites : curation and research (BELAM) project: Report on the Followup meeting of 3-4 2016

# **Participants**

#### Members of the Follow-up Committee:

Brigitte Zanda, curatrice en chef collection metéorites, Muséum national d'Histoire naturelle, Paris Luigi Folco, prof. University of Pisa, Italy Christian Koeberl, prof. University of Vienna & general director Naturhistorisches Museum, Vienna

#### **BELAM project:**

Vinciane Debaille, prof. Université libre de Bruxelles Philippe Claeys, prof. Vrije Universiteit Brussel Marleen De Ceukelaire, RBINS geosciences collection manager Thierry Leduc, RBINS, laboratory of mineralogy Sophie Decrée, RBINS, BELAM project coordinator (replacing Walter De Vos and Michiel Dusar)

#### **Invited experts:**

Cari Corrigan (USA-Smithsonian Institute) Jérome Gattacceca (France-CEREGE) Kevin Righter (USA-NASA-JSC) Caroline Smith (UK-Natural History Museum of London) Akira Yamaguchi (Japan-National Institute of Polar Research)

Meeting place: RBINS VIP meeting room, Brussels

#### General comment about the meeting

Partners of the BELAM projects have collected advices from the experts and members of the Followup Committee invited for this event.

After presentations about curation, conservation and research at the RBINS-VUB-ULB, the international experts gave presentations about conservation/curation in their own institutes. The second day, after a visit of the meteorite collection repository and meteorite laboratory, a roundtable discussion was organized to collect more comments from the Follow-up Committee, but also from the experts.

The main issues that were mentioned are the following (with their solutions):

# Problem 1: Classification of Antarctic meteorites should be largely completed at NIPR before shipment according to NIPR procedure. This was the rationale behind the slow arrival of samples in Belgium.

Solution: A quick classification procedure has been developed thanks to magnetic susceptibility and can be applied to a part of the samples.

#### Problem 2. Control of moisture content of ambient air in the repository.

This not only requires climatisation of the ambient air for stable temperature and low humidity but also monitoring and repair in case of malfunctioning. The air conditioning system did not function properly during the summer of 2014. A dehumidifier is now permanently installed in the repository, and humidity-temperature sensors are present in the room.

# Long-term continuation of the curation

Nomination of a curator: Vinciane Debaille was appointed on 15.10.2014 by RBINS as scientific collaborator – scientific curator to represent RBINS at international meetings of curators. In addition, high-level postdoc worker can help for curation at the RBINS. The most direct way for a federal scientific institute to get high-level postdoc worker is through the BRAIN programme (Belgian Research Action through Interdisciplinary Networks). Taking into account the recommendations of the follow-up committee, four BRAIN projects were formulated and approved for funding (AMUNDSEN, DIABASE, BAMM!, DESIRED). This should ensure help for curation 'til 2024.

# Annex 8. Report of the internship made by Thierry Leduc and Thomas Goovaerts (RBINS) at the NIPR

# Rapport de mission Thierry Leduc

# Tachikawa (Japon) du 26 novembre au 1er décembre 2016

#### **Objectifs**

Cette mission au Japon (Tokyo-Tachikawa) avait comme objectifs de:

- prendre part à la conférence internationale sur les sciences polaires qui se tenait durant la semaine du 28 novembre ;
- visiter les laboratoires et le conservatoire du NIPR (National Institute for Polar Research ) liés à la préparation et la conservation des météorites antarctiques ;
- prendre note des techniques de préparation des météorites et des conditions de conservation de manière à les comparer avec celles mises en place à l'IRSNB pour éventuellement y apporter des améliorations;
- renforcer les contacts avec les collègues du NIPR qui sont les partenaires privilégiés pour tout ce qui concerne les météorites antarctiques.

#### Calendrier de la visite

#### Lundi 28 novembre

- **10h** : accueil de la délégation belge (Sophie Decrée, Vinciane Debaille, Thomas Goovaerts et Thierry Leduc) au NIPR par Akira Yamaguchi ;
- 10h-12h : réunion concernant les divers projets actuels et futurs mis en place par l'Institut et l'ULB pour l'étude des météorites antarctiques. Etaient également présents : Hideyasu Kojima , Naoya Imae et Geneviève Hublet. Sophie a fait un bref rappel de l'organisation de l'Institut en ce qui concerne la gestion technique et muséologique des météorites antarctiques. Elle en a profité pour présenter Thomas et moi-même aux différentes personnes présentes. Les trois projets exposés sont :
  - BELAM (2012-2017) est une collaboration entre l'IRSNB, l'ULB, la VUB et le NIPR (Japon). Il devrait se terminer l'année prochaine après une dernière mission Antarctique. Cette dernière devait avoir lieu en cette fin d'année mais a du être annulée pour des raisons de conflit politique entre le Gouvernement belge et Alain Hubert.
  - AMUNDSEN (2016-2018), coopération entre l'IRSNB, l'ULB et la VUB, est lié à la curation des météorites antarctiques et surtout sur la mise en place de méthodes préventives. Ce projet comprend aussi une expédition en Antarctique pour investiguer certaines moraines pour y trouver des météorites.
  - BAMM "Belgian Antarctic Meteorites and Micrometeorites" (2017-2021). Le projet est une collaboration de l'IRSNB, l'ULB, la VUB et le CEREGE (France) dote d'un budget de 400.000€. Il portera sur l'étude des météorites antarctiques et des

micrométéorites. Dans le cadre de ce projet, deux missions en Antarctique sont également prévues en 2017-2018 et 2019-2020.

Les problèmes de déplacement en Antarctique sont débattus de même que la possibilité pour les scientifiques belges de se joindre à une expédition scientifique japonaise vers « YAMATO Mountains ». Les problèmes logistiques ont été débattus : le type de logement, les types de transport,...

 14-17h : visite des laboratoires du NIPR, labo de sciage, labo de réalisation de sections polies et lames minces, de la microsonde, du RAMAN, de la DRX et du conservatoire.



#### Mardi 29 novembre

- 9h30-16h40 : conférence internationale « Antarctic meteorites/Hayabusa » ;
- 16h40-18h30: discussion avec Mr Imae (NIPR) sur les techniques de préparation et de conservation des météorites antarctiques dans leur institut.

#### Mercredi 30 novembre

- 9h30-16h40 : conférence internationale « Antarctic meteorites/Hayabusa » ;
- 16h30-18h00 : session de posters

#### Jeudi 01 décembre

9h30-17h00 : conférence internationale « Antarctic meteorites/Hayabusa »

#### Vendredi 02 décembre

- 9h30-12h00 : conférence internationale « Antarctic meteorites/Hayabusa » ;
- 14h-17h : dernière visite des laboratoires et dernières discussions avec la technicienne responsable du sciage des échantillons (MIle Ojima).

# Rapport sur les techniques de préparation des météorites antarctiques et sur les conditions de conservation

#### Le labo de sciage

Le labo de sciage est composé essentiellement de trois scies à fil de différentes tailles (images) construites sur demande du NIPR qui permettent de scier des échantillons jusqu'à un diamètre de 20 cm avec la plus grande.



Chaque scie se trouve dans un « caisson » semiétanche, les deux plus petits en plexiglass et le plus grand dans un plastique souple. Les deux premiers sont munis de portes battantes et la plus grande d'une tirette. Chaque caisson est relié à un aspirateur qui permet d'évacuer les fines poussières. Un tissu à l'entrée du tuyau évite des morceaux plus importants de la météorite d'être aspirés (image). Pour être sciées, les météorites sont coincées entre deux planchettes de bois à l'intérieur d'un étau en métal (image). Le système est extrêmement aisé à utiliser



contrairement à notre système de fixation à l'Institut. Pour faciliter la fixation des météorite sur notre support de sciage, l'utilisation de papier aluminium combiné au bois nous est conseillée. Il faut toutefois éviter de scier dans le papier aluminium car le fil perd alors une partie de ses capacités de coupe. Les échantillons sont généralement sciés à sec si ce n'est les météorites métalliques qui ont tendance à s'échauffer. Pour ces dernières, un peu d'éthanol est pulvérisé régulièrement sur l'échantillon pendant le sciage. A la fin du sciage, les échantillons issus de la coupe sont récupérés manuellement avant qu'ils ne tombent ou dans un réceptacle improvisé en papier aluminium. La petite et la grande scie coupe les échantillons verticalement en commençant par le dessus alors que la moyenne les coupe à partir du dessous ou horizontalement. Le fond des caissons est nettoyé régulièrement à l'aide de lingette et d'éthanol. L'inconvénient de ces machines est le système de poulie plus compliqué qui rend le remplacement du fil également plus compliqué. Les poulies doivent aussi être remplacées de temps en temps car le passage du fil finit par les user. Lors du remplacement, il faut s'assurer de l'alignement de toutes les poulies pour un fonctionnement adéquat et éviter une rupture prématurée du fil (diam. 0.18 et 0.35 mm). La vitesse de sciage est constante et la même quel que soit l'échantillon (env. 40% de 2000 feet/min → env. 250 m/min).



Le labo possède également des systèmes pour échantillonner les météorites sans les scier

(principalement pour analyses destructives). Les équipements sont utilisés dans un caisson pour réduire les problèmes de dispersion des fragments lors de l'utilisation d'un marteau par exemple. Il possède le même genre d'équipement que nous pour ce faire. Toutefois, il possède un système de burin sur pas-de-vis (image) permettant de couper un échantillon idéalement en deux sans trop de projection. Un système analogue mais plus archaïque existe chez nous pour réduire certaines roches. L'achat de ce type de matériel est à envisager.



Les échantillons sciés sont nettoyés avec une petite bombonne d'air comprimé (spray). Ils sont pesés mais aussi photographiés, y compris la pièce principale qui reste en collection. Après sciage, les deux échantillons avec leur numéros respectifs sont photographiés ensembles puis séparément. Les photos sont incorporées à la base de données. Les pièces utilisées pour les sections polies sont placées dans des sachets mini-grip en plastique avec le numéro de référence et le poids. Les masses principales qui retournent en collection sont de préférence emballées dans un sac en téflon puis dans un sac en plastique mini-grip avec dessus juste le numéro de référence. Les poids sont consignés dans un registre de laboratoire, dans un dossier papier et dans la base de données. Le registre du labo contient les informations suivantes : la date, le n° d'échantillon, le poids de départ, la technique utilisée, le numéro et le poids du nouvel échantillon, le nom du demandeur. Ces informations sont analogues à celles reprises dans notre registre de labo.

Cela ne nous concerne pas mais il est intéressant de savoir que dans les mini-chambres étanches utilisées pour décongeler les échantillons, le vide (-0.1 MPa) est réalisé avec une pompe à vide sans huile (pas de risque de contamination des échantillons) ce qui n'est malheureusement pas le cas pour la chambre à vide utilisée pour la réalisation des sections polies.

Toutes les manipulations sont réalisées avec des gants, de préférence en polyéthylène plutôt qu'en latex. Toutefois la largeur de ces gants rend la manipulations des très petits objets difficile.

#### Le labo de préparation de sections polies et de lames minces

Un labo est dédié à la réalisation de sections polies et de lames minces. La confection des lames minces ne sera pas abordée puisque l'IRSNB n'est pas équipé pour les réaliser. De plus, le technicien responsable des lames minces ne vient qu'un jour par semaine et nous n'avons pas eu l'occasion de le rencontrer.

L'enrobage des échantillons pour les sections polies se fait avec une résine à trois composants à la place de deux à

- 80% de résine

l'IRSNB :

- 10% de durcisseur
- 10% « extra »



L'échantillon fraîchement enrobé est placé une heure dans une cloche à vide, à une température de 45°C. Ensuite le vide est coupé et l'échantillon finit de sécher pendant la nuit. L'épaisseur des sections polies dépend de la grandeur de l'échantillon mais elles ont de préférence la même épaisseur (facile pour le stockage). Pour ce faire, des repères sont indiqués sur les moules.

Une scie à disque diamanté (image) munie d'un plateau coulissant permettant de « fixer » l'échantillon et de déterminer approximativement l'épaisseur de la coupe permet de réduire la taille des échantillons enrobés dans la résine et destinés principalement à la réalisation des lames minces.

Le labo de polissage est composé d'une polisseuse Struers pour les polissages fins alors que le polissage jusqu'à 4000 meshs est effectué manuellement avec de la poudre d'alumine et un peu d'eau de distribution sur une plaque de verre (400, 800, 2000, 4000). Contrairement à l'IRSNB, le SiC n'est utilisé que plus rarement. Ils utilisent ensuite sur la polisseuse des colloïdes de 6, 3 et ¼ µm. Chaque polissage dure de 4 à 7 minutes. Le technicienne utilise un support manuel qui permet de tenir l'échantillon plus facilement pendant le polissage automatique. Elle ne dispose pas d'un support mécanique comme celui dont nous disposons à l'IRSNB. Entre chaque étape de polissage, les échantillons sont nettoyés une dizaine de secondes dans un bain à ultrasons en utilisant toujours de l'eau de distribution.

#### Le conservatoire

Le conservatoire est maintenu à une température de 24°C et une humidité relative de maximum 30%. Ces valeurs sont maintenues constantes grâce à un système d'air conditionné commandé automatiquement. La pièce entière est climatisée. Le sol du conservatoire est enduit d'un revêtement adhésif permettant de capturer les poussières et de leur éviter de voler. Toute personne accédant au conservatoire doit retirer ses chaussures et enfiler des crocs en plastique. Malgré l'avantage certain d'un tel revêtement, le nettoyage pose problème. Le sol serait lavé à l'eau une fois par an. Est-ce efficace ? Cela n'entraîne-t-il pas d'augmentation temporaire de l'humidité relative ? Questions qui sont restées sans réponse.



Les échantillons sont placés dans des tiroirs en bois ouverts dans des armoires également en bois fermées par des portes, l'ensemble n'étant pas hermétique. Les échantillons sont classés par numéro et les sacs plastiques sont alignés debout côte à côte (image). Contrairement à ce qui nous a été conseillé notamment par la conservatrice du Muséum de Paris, les échantillons ne sont pas accompagnés d'une fiche reprenant toutes les manipulations effectuées précédemment. Il faut toutefois noter que contrairement à nous, les sachets ne sont pas placés dans des boîtes et qu'il serait donc moins facile d'y glisser une fiche. Le téflon très couteux (10 €/ sachet, 200€/flacon rigide)

n'est utilisé que pour les échantillons entiers et non traités (masses principales). Les sections polies et les lames minces sont rangées séparément dans le même type d'armoire mais avec des tiroirs adaptés aux différents types de matériel (images). Les sections polies sont soit enfermées dans des sacs en plastiques et pas en téflon soit placées dans des tiroirs adaptés (image). Lorsque plusieurs échantillons sont présents dans un même sac en plastique, ils sont séparés par un papier inerte spécial pour analyses (papier à l'huile de paraphine). Ce papier pourrait toutefois réagir à longue échéance avec des sulfates ou des sulfures. Les fragments issus des manipulations ou autres que la masse principale sont conservés ensembles mais à part de cette dernière. Lors d'une demande, la technicienne choisit d'abord, parmi ces fragments, le plus adapté en fonction du poids demandé, des analyses à effectuer et de la forme sous laquelle l'échantillon doit être livré. Si aucun fragment ne convient, la masse principale est sciée. Un minimum de 30% de la masse principale originellement présente à l'Institut doit être conservée à titre définitif.

C'est aussi dans le conservatoire que sont préparés, manipulés et conservés les micrométéorites. Ces dernières sont placées sur un support en carbone collant ou sur une lame de verre « coupelle ».

#### <u>Conclusion et améliorations éventuelles à apporter à notre laboratoire des</u> météorites

La mission s'est avérée très enrichissante que ce soit d'un point de vue scientifique ou technique. La visite des laboratoires et du conservatoire de la « section » météorite du NIPR nous a permis de comparer nos méthodes de travail avec celles d'une institution habituée depuis de nombreuses années à travailler avec ce type de matériel. Pour conclure ce rapport, je dirais que leurs méthodes de préparation des météorites et leurs moyens de conservation sont analogues aux nôtres. Toutefois, certaines améliorations ou suggestions sont susceptibles d'améliorer nos conditions de travail et de ce fait la qualité de nos échantillons :

- l'utilisation d'une plaque en verre pour faciliter le polissage manuel ;
- il faudrait étudier les avantages et désavantages liés aux différentes résines ;
- l'acquisition d'un système sous vide éventuellement chauffant pour la confection des sections polies;
- l'acquisition d'un spray d'air comprimé pour nettoyer les échantillons ;
- effectuer un sciage à sec et éviter totalement l'utilisation d'eau. Il est toutefois étonnant qu'ils scient leurs échantillons à sec ou avec de l'éthanol mais polissent à l'eau de distribution !!!
- l'utilisation de gants en polyéthylène plutôt qu'en latex ;
- l'utilisation d'une combinaison feuille d'aluminium/morceau de bois pour fixer les échantillons lors du sciage;
- l'utilisation des sacs en téflon pour conserver les masses principales dans le conservatoire plutôt que les sacs en plastique actuel ;
- effectuer les sciages et les prélèvements mécaniques dans un caisson idéalement muni d'une aspiration pour éviter d'une part les fines poussières de se répandre dans le laboratoire et d'autre part les fragments de s'éparpiller lors des manipulations ;

- le placement du laboratoire en surpression pour éviter les contaminants venant de l'extérieur ;
- L'acquisition d'une scie à fil pour les échantillons plus importants pour réduire l'utilisation de la scie à disque plus dangereuse et beaucoup moins pratique. De plus, cette dernière entraine une perte plus important de matériel;
- l'acquisition d'un « burin sur pas-de-vis » pour couper les échantillons ;
- photographier systématiquement les échantillons après manipulations pour la base de données.

Il est évident que si certaines améliorations peuvent être appliquées aisément et sans trop de frais, d'autres seront plus difficiles voir impossibles.