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# Austrian Academy of Sciences Space Research Institute Department of Experimental Space Research

## **ROSETTA-MIDAS**

## **Flight Spare Summary**

Version 1.0 22/10/2018



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### 1. Introduction

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The MIDAS (Micro-Imaging Dust Analysis System) flight spare (FS), located at the European Space Agencies European Space Research and Technology Centre, was fitted with a set of reference materials and was used extensively to learn more about the operation of MIDAS. The MIDAS FS data is available in the datasets named RO-CAL-MIDAS-3-GRND-REF-year-version\_number. This document contains a description of the samples mounted on the flight spare, as well images of the cantilevers before and after use. Individual experiments were carried out over many years by many scientists who have long since left the MIDAS project. The following is compiled from available reports and notes, and while the information may be limited, it is hoped that the information available proves useful to any future scientists wishing to use the FS datasets.



### 2. Description of Targets Mounted on the Flight Spare

### 2.1. Table of Targets Mounted on the Flight Spare

Name of the sample [purpose]	Origin	Segment	Position on wheel	Page
Position calibration facet [position calibration]	made at ESTEC	0	0	6
Height calibration facet [Z- calibration]	NT-MDT	16	1	7
Square/Block calibration facet [X/Y- calibration]	NT-MDT	32	2	8
Spiked calibration facet [control of the tip shape]	NT-MDT	48	3	9
Clean facet (sol gel coated) [flat reference sample]		64	4	11
Calcite [performance on sharp edges]	AIU Jena	80	5	12
30nm gold spheres [sensitivity test]	Pelco & ESTEC 1	96	6	14
<b>No Sample</b> (was 15nm gold spheres [sensitivity test])	Pelco & ESTEC 1	112	7	16
5nm gold spheres [sensitivity test]	Pelco & ESTEC 1	128	8	16
No samples (was ForC83)	N/A	144	9	17
Clean facet with ink [phase shift test]	made at ESTEC	160	10	18
Harddisk [magnetic mode test]	Cut at ESTEC	176	11	20
8GB HDD magnetic sample 1	FELMI	480	30	22
8GB HDD magnetic sample 2	FELMI	496	31	22
Flat SiO2 sample 1	FELMI	512	32	22
Flat SiO2 sample 2	FELMI	528	33	22
Structure and logo sample 1	FELMI	544	34	23
Structure and logo sample 2	FELMI	560	35	23
Albite (SH1)	GIADA	576	36	28
Albite (SH2)	GIADA	592	37	29
Enstatite (<2 microns), low concentration (ENLC)	GIADA	608	38	30
Enstatite (<2 microns), high concentration (ENHC)	GIADA	624	39	31
Fluffy amorphous carbon aggregates (C)	GIADA	640	40	32
Forsterite (<2 microns) (OL)	GIADA	656	41	33
			1	
Hematite	COSIMA	672	42	-
Pyrrhotite	COSIMA	688	43	-



Fayalite	COSIMA	704	44	-
Fo90 laser irradiated	M.S. Bentley	720	45	-
PtSiO2 fume aggregate	J.K. Hillier	736	46	-
Serpentine	J.K. Hillier	752	47	-
M1	L. Ellerbroek	864	54	34
M2	L. Ellerbroek	880	55	35
M3	L. Ellerbroek	896	56	36
M4	L. Ellerbroek	912	57	37
M5	L. Ellerbroek	928	58	38
M6	L. Ellerbroek	944	59	39
M7	L. Ellerbroek	960	60	40

The test samples mounted on the dust collector wheel have been inspected by means of optical and atomic force microscopy (AFM). Further information on the COSIMA targets, Fo90 laser irradiated, PtSiO2 fume aggregate, and Serpentine, could not be found.



### 2.2 Targets 0-11

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### Target 0:

This facet is located on position 0 of the dust collector wheel. It serves as position calibration of the cantilever array with respect to the facet. No anomalies are visible. The facet is slightly contaminated by some dust grains of unknown origin.



Figure 2.1 - Facet 0 (290  $\mu$ m x 210  $\mu$ m).



Target 1:

This sample is located on position 1. It has a bar structure used for height calibration measurements. There are unambiguous traces of collision at the lower edge (towards facet 2) of the facet. Particles are sprayed all over the lower half of the facet. The upper half is sufficiently clean.

The largest particles have been removed by hand in order to avoid unwanted contacts with the cantilever during future investigations.



Figure 2.2 - Facet 1 (710  $\mu$ m x 530  $\mu$ m).



Target 2:

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This sample is located on position 2. It has a block-like grating structure, which is used for the calibration of the horizontal XY plane of the scanner head. The facet shows a moderate amount of contamination.



Figure 2.3 - Facet 2 (290  $\mu m$  x 210  $\mu m$ ).



### Target 3:

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This sample is located on position 3. The needle-like structure is used for self-imaging of the cantilever tip. It does show an unusual contamination of unknown nature, not observed on other targets. The particle density of particles is very high. The upper corner of the facet is damaged.



Figure 2.4 - Facet 3 (left 710  $\mu$ m x 530  $\mu$ m scan, right 290  $\mu$ m x 210  $\mu$ m scan).



Figure 2.5 – Damage at top left corner of Facet 3.

An image obtained by a commercial AFM shows that the contamination is severe but the particle size is small. Meaningful images are still obtainable.

The particles in the image do not appear as simple flakes released by collisions between the scanner head and the sample facet. One particle appears with an elongated needle-like structure, others also show complex internal chain textures.

Surprisingly the pylons used for self-imaging of the tip show a flat top. This normally indicates that the tip is blunt. However, many fine-grained features in-between the pylons indicate that this is not related to the tip. Possibly the sample is not perfectly etched in this region.



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Figure 2.6 - AFM image of contamination on Facet 3.



Figure 2.8 - 3D image of a pylon on Facet 3.



### Target 4:

This sample is located on position 4. It is a clean coated sample. The surface is still very clean with the exception of one large particle of unknown origin located roughly in the centre of the facet.



Figure 2.9 - Facet 4 (710  $\mu$ m x 530  $\mu$ m).



### Target 5:

This sample is located on position 5. It contains small calcite crystals. It does not show obvious contamination or traces of any alteration processes. Figure 2.10 shows a centre region of the facet. A large number of particles are visible. The particles tend to build small clusters. They have been applied by a water droplet. This explains the concentric distribution of particle clusters.



Figure 2.10 - Facet 5 (710  $\mu$ m x 530  $\mu$ m).



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In the part of the facet next to facet 4 a small damaged region with two particles flaking off are visible. A collision between microscope and sample has to be assumed.



Figure 2.11 - Two particles flaking off the edge of facet 5 (710  $\mu$ m x 530  $\mu$ m).



### Target 6:

This sample is located at position 6. It contains a gold sphere 30 nm in diameter. The sample is intact despite one strip within which particles are removed.



Figure 2.12 - Area on which gold spheres were removed on facet 6 (710  $\mu$ m x 530  $\mu$ m). This region is exactly in the centre of the facet. Apart from this region the pattern looks nominal.



One AFM image, below, was taken from this sample. Height measurements confirm that many particles are in the order of 30 nm.



Figure 2.13 - AFM image of facet 6.



Target 7:

This sample should be located at position 7. It is coated with gold spheres of 15 nm in diameter. The facet appears to be missing. No remains of the facet can be observed in the gap of the collector wheel.

Target 8:

This sample is located on position 8. It is coated in gold spheres of 5 nm in diameter. It does not show any significant amount of contamination. The optical image does not give any indication of the applied gold particles.

The AFM image shows a large amount of small spheres. Their height measurement has an average between 15 and 20 nm, slightly above the anticipated value of 5nm. The "material" showing a ring-like structure around the individual particles is an image artefact.



Figure 2.14 - Optical micrograph of facet 8 (290  $\mu m$  x 210  $\mu m$ ).



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Figure 2.15 - AFM image (height mode) of facet 8.

### Target 9:

This sample should be located at position 9. A mixture of silicatic and carbonaceous material is condensed on the surface (ForC83). The facet is missing.





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Target 10:

This sample is located on position 10. It is a clean non-coated facet with a stripe ink. The ink stripe is scratched in order to visualize the material differences in the phase shift signal.



Figure 2.16 - Images of different magnification (A, 710  $\mu$ m x 530  $\mu$ m ; B, 290  $\mu$ m x 210  $\mu$ m) of facet 10. The black square in figure B marks the region of the AFM image.



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A selected area was imaged, below, by an AFM in the height (A) and phase shift (B) mode. The specific purpose of this test sample is to visualize material difference by the phase shift of the cantilever excitation signal. While the image shows a pronounced contrast at the edge between ink and ink-free region (see arrow) a distinct difference between ink and ink-free areas cannot be identified.

Probably the storage in a vacuum for more than 1.5 years has modified the material properties to such extent that both appear similar.



Figure 2.17 - AFM image of a selected area on facet 10. Figure A shows the height information, Figure B the displays phase shift of the cantilever signal. The arrow indicates a sharp edge between the ink layer and the clean substrate.



### Target 11:

This sample is located on position 11. It is a piece of a computer hard disc and serves as a test sample for investigation of magnetic properties. It does not show a significant amount of contamination. The clean sample surface shows a few fine scratches which are a result of the writing head of the hard disk.



Figure 2.18 Optical image of facet 11 (710  $\mu$ m x 530  $\mu$ m).



### CONCLUSION:

It can be concluded that during operations the sensor head had some collisions with the dust collector wheel. In some occasions it led to the damage of the cantilever array or sample.

It is important to note that the damage of a tip results in a tremendous particle spray around the tip on the cantilever but probably also on the sample at the location of collision.

The summary below shows (Table 1, Table 2) that approximately one half of all cantilevers have a broken tip or are missing. The way the cantilevers are affected, i.e. spray of particles, points towards a forced physical approach of the cantilever onto the sample surface.

### Table 1 Current state of cantilevers

	Tip (normal use)	Tip (broken)	Cantilever broken	Cantilever
				missing
Cantilever	B0/C4	B0/C0	B1/C7	B1/C2
no.	B0/C5	B0/C1		B1/C4
	B0/C6	B0/C2		
	B0/C7	B0/C3		
	B1/C0	B1/C5		
	B1/C1	B1/C6		
	B1/C3			

### Table 2 Current state of sample facets on the dust collector wheel

	nominal	contaminated	damaged	Missing+
Facet no.	0	3	1	7
	2		3	9
	4			
	5			
	6			
	8			
	10			
	11			

The reason for the missing facets remains unclear. It could be due to a collision event but also a nonsufficient bonding by glue cannot be excluded.

The type of contamination on facet 3 is unknown. The chain-like structures possibly point towards an organic origin.



### 2.3 FELMI Targets (positions 30-35)

The Austrian Centre for Electron Microscopy & Nanoanalysis prepared several samples with following properties.

8GB HDD magnetic samples 1&2:

- Precut by ultrasonic cut
- Grinded lateral dimension followed by vertical dimension
- Top layer protection: superglue
- Cleaned with Aceton and Isopropanol
- Air dried



### AFM morphology

MFM phase

Figure 2.3.1 – AFM measurements of magnetic sample 1 or 2 (unknown): Tapping mode + Lift mode; Tip: Veeco MESP, f=60-100kHz, k=1-5 N/m; rectangular Antimony (n) doped Silicon with Co/Cr coating; Lift height: 55nm.

### Samples 1&2: Ultraflat sample: SiO2:

Si-Waver with 60 nm SiO2 layer on top

- Ra: 0.67 nm
- Rms: 0.84 nm
- Precut: controlled breakage
- Grinded lateral dimension followed by vertical dimension
- Top layer protection: superglue
- Cleaned with Aceton and Isopropanol
- Air dried





AFM morphology



Structure and logo sample 1&2:

- Precut: controlled breakage
- Grinded lateral dimension followed by vertical dimension
- Top layer protection: superglue
- Cleaned with Aceton and Isopropanol
- Air dried prior to structuring
- no cleaning afterwards

### AFM Measurement:

Tapping mode; Tip: AC160TS Olympus, f=300kHz, k=42 N/m; rectangular Silicon Cantilever







AFM morphology

AFM morphology

Figure 2.3.4 – High resolution scans of the 250 nm, 500 nm and 1  $\mu$ m structures



Figure 2.3.5 – High resolution images of 5 µm structures









Figure 2.3.7 – High resolution scan (left) and line scan (right) of window structure.



platinum deposition



AFM morphology



milled serpentines

AFM morphology







AFM morphology

Figure 3.3.9 – Milled squares of different sizes.

milled serpentine



Figure 3.3.10 – High resolution image of the milled serpentine (left) and line scan (right) shown by the red line in the left hand figure.



AFM morphology

AFM morphology (inverted colorscale)

Figure 3.3.11 – High resolution scans of the milled logo.



### 2.4 GIADA Targets

### Sample SH1:

Albite grains less than 10 microns in diameter deposited on a MIDAS silicon target with a higher density of particles per unit area with respect to SH2.



Figure 2.4.1 - FESEM image of sample SH1: Albite grains with diameters < 10 microns deposited by aerosol dispersion technique on a silicon wafer (MIDAS target) high number of grains per unit area.



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### Sample SH2:

Albite grains less than 10 microns in diameter deposited on a MIDAS silicon target with a lower density of particles per unit area with respect to SH1.



Figure 2.4.2 FESEM image of sample SH2: Albite grains with diameters < 10 microns deposited by aerosol dispersion technique on a silicon wafer (MIDAS target) low number of grains per unit area.



### Sample ENLC:

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Enstatite grains less than 2 microns in diameter deposited on a MIDAS silicon target with a low concentration of particles per unit area.



Figure 2.4.3 - Low magnification FESEM image of sample ENLC to have an idea on how the Enstatite grains with diameters < 2 microns are dispersed on the MIDAS silicon target.



### Sample ENHC:

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Enstatite grains less than 2 microns in diameter deposited on a MIDAS silicon target with a higher concentration of particle per unit area with respect to ENLC sample.



Figure 2.4.4 - Low magnification FESEM image of sample ENHC to get an idea on the higher concentrations of Enstatite grains with diameters < 2 microns dispersed on the MIDAS silicon target.



### Sample (C):

Fluffy Amorphous Carbon aggregates deposited on a MIDAS silicon target by laser ablation technique.



Figure 2.4.5 - Low magnification FESEM image of sample C to get an idea on the distribution of fluffy amorphous carbon aggregates dispersed on the MIDAS silicon target.



Sample OL:

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Forsterite grains less than 2 microns in diameter deposited by means of the aerosol dispersion technique on a MIDAS silicon target.



Figure 2.4.6 - FESEM image of sample OL: Forsterite grains with diameters < 2 microns deposited by aerosol dispersion technique on a silicon wafer (MIDAS target).



### 2.5 Dust Targets (positions 54-60)

The following targets were produced in the same lab with a similar experimental procedure used as in Ellerbroek *et al.*, 2017 (The footprint of cometary dust analogues – I. Laboratory experiments of low-velocity impacts and comparison with *Rosetta* data, Monthly Notices of the Royal Astronomical Society, MNRAS 469, S204–S216).



Figure 2.5.1 – M1.



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Figure 2.5.2 – M2: 0.3  $\mu$ m particles fired at 3.6-3.9 m/s.

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Figure 2.5.3 – M3: 0.3  $\mu$ m particles fired at 1.1-1.6 m/s.



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Figure 2.5.4 – M4: 1.5  $\mu$ m particles fired at 2.7-3.5 m/s.

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Figure 2.5.5 – M5: 1.5  $\mu$ m particles fired at 0.2-1.4 m/s.



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Figure 2.5.6 – M6: 1.0  $\mu$ m particles fired at 2.5-3.6 m/s.





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Figure 2.5.7 – M7: 1.0  $\mu$ m particles fired at 0.8-1.3 m/s.



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### 3. Images of cantilevers before and after use

After significant usage, MIDAS scannerhead F15 was removed from the flight spare on the 2<sup>nd</sup> September 2013 and SEM images were acquired at two angles – normal (looking "down" on the tips), and from the side to allow us to compare with the original images made prior to being mounted on the flight spare, and estimate the tip shape/sharpness.

Template for following images:

Cantilever end prior to mounting	Tip image prior to mounting
Cantilever end after use	Tip image after use
Highest resolution cantilever image after use	Highest resolution tip image after use

MIDAS scannerhead F15 comprises the following chips:

W18-U, W19-G, W19-Q, W21-K

Cantilevers are numbered from 1-16, but care should be taken with the number of cantilevers on the chip – this corresponds to W18-U\_D, W18-U\_C, W18-U\_B, W18-U\_A, W19-G\_D etc.



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