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In situ multi-frequency measurements of magnetic susceptibility as an indicator of planetary regolith maturity

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2	maturity
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#### 26 Abstract

27

28 Space weathering is now generally accepted to modify the optical and magnetic properties of airless 29 planetary regoliths such as those on the Moon and Mercury. Under micrometeorite and ion bombardment, 30 ferrous iron in such surfaces is reduced to metallic iron spheres, found in amorphous coatings on almost all 31 exposed regolith grains. The size and number distribution of these particles and their location in the regolith 32 all determine the nature and extent of the optical and magnetic changes. These parameters in turn reflect the 33 formation mechanisms, temperatures, and durations involved in the evolution of the regolith. Studying them 34 in situ is of intrinsic value to understanding the weathering process, and useful for determining the maturity of 35 the regolith and providing supporting data for interpreting remotely sensed mineralogy. Fine-grained metallic 36 iron has a number of properties that make it amenable to magnetic techniques, of which magnetic 37 susceptibility is the simplest and most robust. The magnetic properties of the lunar regolith and laboratory 38 regolith analogues are therefore reviewed and the theoretical basis for the frequency dependence of 39 magnetic susceptibility presented. Proposed here is then an instrument concept using multi-frequency 40 measurements of magnetic susceptibility to confirm the presence of fine grained magnetic material and 41 attempt to infer its quantity and size distribution. Such an instrument would be invaluable on a future mission 42 to an asteroid, the Moon, Mercury or other airless rocky Solar System body. 43 44

- Keywords: magnetic susceptibility, space weathering, planetary regolith, maturity 45 Accei
- 46

#### 47 **1. Overview of space weathering**

48 Amongst the first things to be noted on analysis of the Apollo and Luna samples brought to Earth was that 49 regolith samples had guite different magnetic and optical properties from rock samples, even those which 50 were chemically and mineralogically similar (Hapke et al., 1975). It became clear that these differences were 51 due to a surface maturation process operating in the upper few millimetres of the regolith. Various metrics 52 were defined to quantify the amount of time a particular regolith had been exposed in this way (Langevin and 53 Arnold, 1977). These all rely on the cumulative effects of solar wind bombardment, micrometeorite impacts 54 and cosmic rays. Such weathering agents, and their effects on the regolith, are collectively known as space 55 weathering. The most durable of these metrics was the ferromagnetic resonance (FMR) index, defined as 56  $I_{s}$ /FeO, where  $I_{s}$  is the magnitude of an observed resonance feature and FeO is the weight percentage of iron 57 oxide in the regolith (Morris, 1976).

58

This strong absorption feature, measured with an electron spin resonance (ESR) spectrometer, is believed to 59 60 originate from spherical metallic iron grains of between approximately 4 and 33 nm in diameter (Morris, 61 1976). These same grains are now understood to play a key role in the optical effects of space weathering, 62 which result in the reddening, darkening and a reduction in spectral contrast of visible and near-infrared 63 (VNIR) reflectance spectra. The theory that vapour-deposited coatings might be responsible for the optical 64 and magnetic properties of the regolith was first advanced during the initial analyses of lunar samples 65 (Hapke et al., 1975) but was not verified until modern laboratory techniques were applied (Keller and McKay, 66 1993). Theoretical modelling has also demonstrated that a vapour coating containing spherical metallic iron 67 grains can indeed replicate the optical effects of space weathering (Hapke, 2001). Various terms have been 68 used to describe this iron, in particular sub-microscopic metallic iron (SMFe) and nanophase iron (npFe<sup>0</sup>). As 69 this paper refers mainly to the magnetic properties of nanometre sized iron particles, the latter is perhaps 70 more appropriate here.

71

Several missions, including the MESSENGER and BepiColombo missions to Mercury, the Dawn asteroid mission and several lunar orbiters are currently scheduled to perform remote sensing of airless bodies, or more specifically their regoliths. Determining surface mineralogy from such measurements is greatly hindered by space weathering. The spectral alteration can in fact be so severe as to mask completely weak absorption features, making even mineral identification impossible, let alone quantitative comparisons.

77

78 Ideally, all remote sensing measurements would be supported by in situ, ground truth, data. If the same 79 weathering processes that occur on the Moon also play key roles on other airless bodies, ferromagnetic 80 resonance should be a useful measurement on those as well. ESR spectrometers work by applying a 81 magnetic field across a sample, causing unpaired electrons to align their magnetic moments either parallel or 82 anti-parallel to the field. These two states have different energy levels, with a separation determined by the 83 strength of the applied field and the gyromagnetic ratio (the "g-factor"), which is modified from the free 84 electron value by the electronic environment of the nucleus. A transition can be induced between these two 85 states by the absorption of electromagnetic radiation with the same photon energy as their separation. Thus 86 an absorption spectrum can be produced by varying either the frequency of applied radiation or the strength 87 of the magnetic field. The nature of such spectra can reveal much about the environment of the unpaired 88 electrons, which in inorganic materials are typically associated with transition metals. In ferromagnetic 89 materials, such as the metallic iron grains found in the lunar regolith, the resonance feature is orders of 90 magnitude stronger than that seen in a paramagnetic material, due to the coupling of spins. The frequency 91 and field strength at resonance are used to derive the g-factor, which varies from the free electron value of 92 approximately 2.0 according to the electronic structure; most lunar samples measured have values of 93 2.10 ± 0.03 (Manatt et al., 1970).

94

95 Typical laboratory ESR spectrometers are necessarily massive and unwieldy pieces of equipment, using 96 large electromagnets to generate substantial magnetic fields (typically on the order of hundreds of milli-97 Tesla). A fixed monochromatic source of microwave energy is usually employed and the reduction in 98 received energy monitored as the magnetic field strength is scanned.

99

Miniaturised ESR/EPR spectrometers have been proposed for planetary missions (Kim et al., 2004). In such instruments, it is typically the microwave frequency that is scanned whilst the magnetic field strength is held constant. Two configurations were developed by Kim et al. (2004), one requiring sample ingestion and the other a "contact sensor" using a microstrip resonator. Such an instrument has been suggested to search for organic radicals on Mars and to identify radiation damage in minerals (Yen and Kim, 2004). This approach leads to a considerably lower mass and power than laboratory equipment; the field prototype had a mass of only 1.7 kg.

107

108 This reduction in mass is very promising and should be further developed. However, this paper proposes a

109 much more compact instrument concept which uses multi-frequency measurements of magnetic

susceptibility to infer the presence of sub-microscopic iron. Theoretically it could have a mass an order of magnitude less than the ESR instrument. Additional data that could be provided by such a sensor include basic magnetic mineralogy and electrical conductivity of the regolith.

#### 113 2. Magnetic properties of extraterrestrial regoliths

#### 114 2.1. The lunar regolith

115 Since most of our understanding of space weathering derives from the Moon, the case of the lunar regolith 116 will be discussed first. Metallic iron in lunar regoliths is thought to derive from several sources: direct 117 meteoritic input (e.g. from metal-bearing meteorites), reduction of hydrogen-rich regolith as a result of 118 micrometeorite impact heating (hydrogen having been implanted from the solar wind) and the production of 119 surface-correlated iron from the re-condensation of vapour produced in solar wind sputtering and 120 micrometeorite impacts. Subsequent gardening of the regolith can result in this iron being re-worked into the 121 regolith, for example becoming bound into agglutinates. One possible way of distinguishing these sources of 122 iron is by their grain size range and their location in the regolith (Morris, 1980), although thermal annealing 123 processes can increase grain sizes over time (Noble and Pieters, 2003) and so this approach is not uniquely 124 diagnostic. Determining the relative contributions of these processes on the Moon is important for 125 understanding their differing contributions throughout the Solar System, which should scale with factors including the heliocentric distance, the size of the body, presence of a magnetic field etc. 126 127 128 In terrestrial geology, magnetic susceptibility is primarily dominated by iron oxides such as the 129 titanomagnetite series (Carmichael, 1989). Magnetism on Mars is also believed to be dominated by iron 130 oxides and sulfides (Rochette et al., 2005). In the lunar (Fuller and Cisowski, 1987), and by analogy

131 Mercurian or asteroidal, environments such oxidised minerals are not readily formed. Extensive analysis of

132 lunar samples has shown that the main carriers of ferromagnetism on the Moon are metallic iron and iron-

133 nickel grains. As such, magnetic susceptibility ( $\chi$ ) should correlate well with the amount of metallic iron in the

regolith. It has in fact been shown for a limited set of samples that  $\chi$ /FeO correlates extremely well with

135 I<sub>s</sub>/FeO and so should make a good maturity index (Oder, 1992). This normalisation to the weight percentage

136 of *FeO* is required since the amount of metallic iron is related to both the degree of weathering and the

amount of iron oxide in the original, unweathered, material.

138

Although no such in situ extraterrestrial measurements have been made, low field magnetic susceptibility (that is, magnetic susceptibility measured at a field too low to produce irreversible changes in the sample magnetisation) has been measured in the laboratory for many samples of lunar regolith. The typical range of mass susceptibility for lunar regoliths is around  $1400 - 4400 \ 10^{-8} \text{ m}^3 \text{ kg}^{-1}$  (Carmichael, 1989), depending on the mineralogy and maturity of the sample.

144

Magnetic susceptibility is, of course, not a unique measurement of iron content. The majority of lunar minerals are paramagnetic and the total susceptibility also reflects their various contributions. However, the cumulative effects of weathering are understood to increase the ferromagnetic metallic iron content in the regolith as it matures. Although the susceptibility of this iron is a complicated function of temperature and grain size, it is always considerably larger than the paramagnetic susceptibility. Thus only a few wt% of metallic iron, as found in a mature lunar soil, can dominate the magnetisation of such a sample.

151

152 Ferromagnetic susceptibility is critically dependent on the magnetic domain state of the magnetic carrier. 153 Large iron grains will be multi-domain (MD), such that neighbouring domains are oriented with opposing 154 magnetic polarities in order to reduce magnetostatic energy. It is relatively easy to change the magnetisation 155 of such a material and hence the coercivity is low. Below a critical size, this energy saving is less than that 156 required to split a single domain into two, and hence the material will remain single domain (SD). In this 157 state, ferromagnetic materials are strongly resistant to magnetic change and hence have a higher coercivity. 158 It is magnetite grains of this type that allow the Earth's geomagnetic signature to be recorded and retained 159 over geological timescales. Even so, any acquired magnetism decays over time in an exponential fashion, 160 described by its relaxation time. As the grain size is decreased further, thermal effects become important. At 161 a critical temperature and size, thermal agitation overcomes the magnetic ordering and the atomic moments 162 of such particles are free to orient with an applied magnetic field. Such particles will behave like 163 paramagnets, but with a vastly greater magnetic moment and magnetic susceptibility, and hence are called 164 superparamagnetic (SPM). Usually, a material is considered SPM if its relaxation time is comparable to or 165 smaller than the measurement time in a typical experiment (i.e. if its decay is observed). The boundary 166 between SD and SPM grains in a given sample is therefore a function of the experiment being performed 167 and the temperature of the sample. Figure 1 shows a plot of the variation of susceptibility of metallic iron 168 grains at room temperature with size, based on Néel theory (Néel, 1949) and following Stephenson (1971a), 169 which demonstrates how SPM particles can have a magnetic susceptibility an order of magnitude larger than 170 SD grains; this figure will be discussed in more detail later.

171	
172	[Figure 1]
173	
174	Metallic iron with characteristic of all of these size ranges (SPM, SD and MD) has been detected via
175	magnetic measurements of lunar materials, although a large proportion of the Fe <sup>0</sup> in mature regolith is
176	superparamagnetic (Dunlop and Özdemir, 1997). Theoretical studies have shown that the range of sizes and
177	shapes possible for SD metallic iron are rather small, and almost non-existence for spherical grains (Butler
178	and Banerjee, 1975). These authors calculate that only 9% of the metallic iron the lunar regolith need be
179	single domain to reproduce the measured magnetic characteristics. Somewhat oblate spheroid grains, for
180	example with an elongation of only 10%, are sufficient to fulfil this criterion. The FMR signature, however,
181	arises from grains that are single domain and close to spherical (and hence have negligible shape
182	anisotropy). Thus it is likely that metallic iron grains dominated by both magnetocrystalline (spherical) and
183	shape (spheroid) anisotropy are present in the lunar regolith.
184	
185	It is, however, the presence of extensive SPM material in lunar regoliths which dominates their magnetic
186	susceptibility. The proportion of SPM grains is, in turn, a function of space weathering. It can be seen that
187	measuring the initial magnetic susceptibility of a sample in situ, coupled with measurements of bulk
188	composition from complementary instrumentation, should give a good first indication of the degree of regolith
189	maturity and hence the degree of spectral alteration that should be expected in, and ideally removed from,
190	remote reflectance spectra.
191	
192	Simple but effective models have shown that a given reflectance spectrum of an un-weathered material can
193	be numerically manipulated to simulate weathering by the linear addition of a volume and surface correlated
194	component of metallic iron (Hapke, 2001). In practice, the spectrum is inverted to obtain the single scattering
195	albedo using a simplified radiative transfer model. Once the complex refractive index of the measured
196	sample has been obtained, this is combined with the optical constants of metallic iron and the process
197	reversed. The size of the host grains, the weight percentage of fine grained iron and the distribution between
198	surface and volume correlated iron are all critical parameters in this model.
199	
200	Thus in situ measurement of the amount and size distribution of metallic iron could theoretically allow
201	weathered spectra to be inverted to yield the un-weathered properties. Unfortunately this is a non-trivial task
202	even in a terrestrial laboratory. Attempts have been made to measure the size distribution of metallic iron

spherules in a small subset of lunar samples by SEM and TEM (James et al., 2003), but this clearly is not
useful for in situ investigation.

#### 205 2.2. Other Solar System regoliths

The discussion of regoliths here applies mainly to airless bodies, where both solar and micrometeorites regularly impinge upon the surface. However, evidence from Martian meteorites also points to a mechanism for impact shock metamorphism of olivine to produce similar nanoparticles, probably during larger impacts and over a larger spatial scale (Van de Moortèle et al., 2007).

210

211 Mercury is also most likely a highly weathered surface, as its location in the inner Solar System results in a

high impact flux and greater average impact velocities than at the Moon (Cintala, 1992). However, the

213 intrinsic magnetic field discovered by Mariner 10 and verified recently by Messenger (Anderson et al., 2008)

is expected to stand off the solar wind, at least some of the time, limiting the role of solar wind sputtering, at

215 least at certain combinations of latitude, heliocentric distance and solar condition.

216

Asteroidal regoliths are also of great interest, and here it should be noted that the presence of a magnetic field can also play a role, again preventing access to the surface of a body. In the asteroid belt, where solar wind sputtering might be expected to dominate over impacts, weathering could potentially be inhibited (Vernazza et al., 2006).

221

#### 222 2.3. Laboratory regolith analogues

The motivation for this paper derived from laboratory experiments performed to simulate the space weathering process, with particular reference to Mercury. In these experiments (Bentley, 2004; Bentley et al., in preparation), powdered olivine samples were irradiated under vacuum with a pulsed IR laser to simulate the weathering process. As with previous experiments (e.g. Sasaki et al., 2001) this resulted in optical changes resembling those seen to have occurred in lunar samples. A variety of magnetic techniques was then used to characterise the changes, including ESR spectroscopy, vibrating sample magnetometry and multi-frequency measurement of the magnetic susceptibility.

230

In these latter experiments (Bentley et al., in preparation), a Bartington MS2-B sensor was used to record the
 magnetic susceptibility before and after irradiation. This instrument uses an AC technique with a field

strength of approximately 250  $\mu$ T (peak) and measurement frequencies of 0.465 kHz and 4.65 kHz. These frequencies are sufficiently low that any conductive losses should be minimal. Samples of San Carlos olivine, sieved to < 63  $\mu$ m and each with a mass of 2 g, were measured using the Bartington device before and after irradiation. The total bulk magnetic susceptibility of the samples typically increased several-fold after irradiation, as might be expected if paramagnetic ferrous iron is being reduced to metallic iron. Additional evidence from ESR spectroscopy and other magnetic methods support the hypothesis that metallic iron, some of it superparamagnetic, is produced.

240

241 The increase in bulk susceptibility can be used to make a first, coarse, estimate of the amount of metallic iron 242 that could cause such a change. As an example, one such experiment showed an increase in mass specific magnetic susceptibility after irradiation of 8.79 ± 0.09 × 10<sup>-8</sup> m<sup>3</sup> kg<sup>-1</sup>. If it is assumed that the increase derives 243 244 solely from the production of multi-domain metallic iron, which has a room temperature initial susceptibility of 3.77 × 10<sup>-4</sup> m<sup>3</sup> kg<sup>-1</sup> (Stephenson, 1971b), this corresponds to only 0.023 wt%, or substantially less if a 245 246 sizeable fraction is superparamagnetic, and hence has a susceptibility up to 66 times higher (Stephenson, 247 1971a)! Clearly this technique is highly sensitive to the changes attributed to space weathering, which can produce several wt% metallic iron in mature lunar regolith. 248

249

250 The Bartington device also allowed investigation of the frequency dependent nature of the sample. The un-251 irradiated samples showed an identical susceptibility at both frequencies, within experimental errors. The 252 same samples after irradiation, however, showed a coefficient of frequency dependence (CFD) of over 4%. 253 Typically a CFD of less than two percent is measured if the material contains no SPM grains, a value in the 254 range 10 – 14% is found for a sample containing virtually all SPM grains and a value in the middle has a 255 mixture of SPM and coarser grains (Dearing, 1999). This frequency dependence arises as a result of the two 256 measurement frequencies corresponding to two different observation times, each of which defines a different 257 SD/SPM threshold; the theoretical explanation for this behaviour will be discussed later.

258

There is hence a clear rationale for making in situ measurements of magnetic susceptibility; it can provide an estimate of the metallic iron content and detect the presence of SPM particles. The following section

describes how such a sensor might be developed for making a contact measurement on a planetary lander,

rover or penetrator and discusses the major hurdles and the operational and calibration difficulties arising.

#### 263 3. An *in situ* magnetic susceptibility instrument for planetary regolith maturity determination

#### 264 **3.1.** *Methods of measuring magnetic susceptibility*

265 Methods of measuring magnetic susceptibility can be divided into two categories. The first measures the 266 force experienced by a sample placed in a magnetic field gradient. In the laboratory, the Evans and Faraday 267 balances are examples of this type. The second uses electromagnetic induction. In the simplest setup, an 268 alternating current is applied through a coil. The sample to be measured is placed inside, or close to, the coil. 269 Magnetic susceptibility is directly related to magnetic permeability  $[\mu = \mu_0(1 + \chi)]$ , which in turn is related to 270 the inductance of a coil immersed in a medium. The required measurement is therefore one of inductance, 271 which can be performed quite simply by a variety of electrical circuits. Typically, AC bridges are used to 272 measure an unknown inductance, in which the resistive and reactive components of the inductance must be 273 separately balanced (Collinson, 1983). In this configuration, when the bridge is unbalanced by the presence 274 of a sample, either the off-balance current can be read, or the bridge can be balanced again to give a zero-275 current at the null detector. It should again be pointed out that both the real and imaginary parts of the 276 complex magnetic susceptibility are of interest here, and thus these should both be measured. Hence some 277 phase discrimination, or lock-in amplifier is needed to record both the signal in phase with the driving 278 alternating field and that in quadrature ( $\pi/2$  out of phase).

279

For a single coil configuration, the self inductance change due to the magnetic susceptibility of surrounding material is purely reactive, resulting in a change in the quadrature component of the coil impedance. In reality it is likely that the surrounding medium has a non-zero electrical conductivity. Induced currents flowing through the medium also modify the effective resistance of the coil and this must be accounted for. In such a configuration, the inductance change is usually measured by observing the change of frequency of an LCR circuit in which the inductor is coupled to the sample material.

#### 286 3.2. The benefit of multi-frequency AC measurements

The measurement of initial magnetic susceptibility at a single frequency can be very useful; the Apollo sample collection has been extensively characterised in this way, and more recently it has been used to classify stony meteorites (Rochette et al., 2008) – a technique that has also been proposed for in situ use (Rochette et al., 2004). The additional benefits gained from performing the measurement at several oscillator frequencies are discussed here.

292

The utility of multi frequency measurements can be explained with reference to the work of Néel (1949), who showed that the relaxation time ( $\tau$ ) for a collection of uniformly magnetised (i.e. SD), non-interacting uniaxial grains is related to the volume (v), temperature (T), saturation magnetisation ( $j_s$ ) and microscopic coercivity ( $h_c$ ) of these grains, by equation 1, where k is the Boltzmann constant and  $f_0$  is a constant, approximately  $10^9 \text{ s}^{-1}$  for iron.

298

299 
$$au = \frac{1}{f_0} \exp\left(\frac{vh_c j_s}{2kT}\right)$$
 Equation 1

300

The coercivity depends on the dominant anisotropy in the material, which in metallic iron is either magnetocrystalline (spherical grains) or shape (non-spherical grains), and using an AC technique the measurement time can be taken as the inverse of the frequency. A spherical iron grain has a microscopic coercivity dominated by magnetocrystalline anisotropy, given by  $h_c = 2K_1 / j_s$ , where  $K_1$  is the uniaxial anisotropy constant. A value of  $4.8 \times 10^4$  J m<sup>-3</sup> will be used here (Dunlop and Özdemir, 1997), although considerably higher values have been reported for iron nanoparticles (Lacroix et al., 2008).

307

308 Knowing these parameters, it is possible to calculate the grain volume (the critical volume) at the boundary 309 between stable single-domain (SD) and superparamagnetic (SPM) grains, and hence the critical grain 310 diameter. For example, in a 1 kHz measurement of a collection of spherical metallic iron grains, this boundary occurs at a diameter 20.3 nm. In other words, grains smaller than this are SPM, whilst those larger 311 312 are SD. This is of course only strictly applicable for a material comprising SD grains of a single size. In reality 313 a range of grain sizes is likely to be present, resulting in a spectrum of decay times, blocking temperatures 314 and volumes. In addition, the saturation magnetisation depends on temperature, although can be considered 315 constant well below the Curie point (Stephenson, 1971a), and the coercivity depends on both grain size and 316 temperature. Nevertheless these approximations are sufficient to explain the fundamental behaviour of fine 317 particle magnetism with respect to frequency.

318

With re-arrangements of Equation 1, it can be seen that the critical volume is proportional to the logarithm of the measurement time, and so increasing the frequency (decreasing the measurement time) moves the boundary to smaller grain sizes. As a result, a material with a significant amount of very fine-grained ferromagnetic material will experience a decrease in total magnetic susceptibility with increasing frequency, as proportionally more grains become SD. As an example, the Bartington magnetic susceptibility instrument

324 referred to earlier uses measurement frequencies of 0.465 kHz and 4.65 kHz, corresponding to transition 325 grain diameters of 20.7 nm and 19.5 nm, respectively. Thus a decrease in the measured susceptibility 326 between these two frequencies can be explained by the presence of spherical iron grains in this grain size 327 range. This can be readily observed in Figure 1 by imagining a grain with diameter 21 nm, for example. In an 328 AC measurement at 1000 Hz this grain is single domain, however at 100 Hz this same grain is SPM and has 329 a magnetic susceptibility 14.5 times larger. 330 331 Thus direct measurements of magnetic susceptibility with frequency can be used to make inferences about 332 the size distribution of magnetic carriers, however the logarithmic nature of the frequency dependence 333 results in a rather limited iron particle size range. Figure 2 demonstrates this on a plot of SPM/SD transition 334 diameter for metallic iron at room temperature as a function of frequency. It still, however, provides a 335 mechanism for performing a limited degree of magnetic granulometry and at the least is a useful indicator of 336 the presence of superparamagnetic materials. Such a dual frequency measurement, if it had been carried 337 out on the NEAR spacecraft, could have provided additional vital evidence about the nature of space 338 weathering on asteroids. 339 340 [Figure 2 341 342 Since the blocking volume also depends on temperature, knowledge of the regolith temperature would also be required. If deployed as part of an instrumented sensor package such as the proposed HP<sup>3</sup> mole concept 343 344 (Spohn et al., 2001), such high precision temperature measurements would already be included.

345 **3.3**. Complementarity to other measurements

It is worth considering what instrument package might be deployed in a future mission, and hence what
synergy can be drawn from flying a magnetic susceptibility instrument alongside more traditional instruments.
This section therefore discusses typical in situ instruments that make complementary measurements, and
how these measurements might be related.

#### 350 3.3.1. Mössbauer

The most obvious complementary measurement is that of Mössbauer spectroscopy, which is specifically tailored to the study of iron and iron bearing materials. In nano-scale materials, such as superparamagnetic iron, the magnetic ordering of larger particles is not present. As such, the magnetisation in the absence of an

applied field orients randomly, leading to a net zero hyperfine magnetic field. The signature for SPM iron is
 therefore a singlet situated at zero velocity, while larger iron grains appear as a distinctive sextet.

356

As seen previously, the determination of whether a particle is SD or SPM is made by both the sample temperature and the so-called "observation time". In the case of a Mössbauer measurement, this time corresponds to the duration of a Larmor precession of the <sup>57</sup>Fe nucleus and is of order 10<sup>-8</sup> s. For spherical metallic iron room temperature this results in a blocking diameter of approximately 11 nm. As a result, much of the iron seen as SPM with typical AC susceptibility measurements will be SD in a Mössbauer measurement.

363

364 Measurements of lunar samples have shown that both SD and SPM iron can be detected with Mössbauer 365 spectroscopy (e.g. Morris et al., 1998). The remaining question is whether an in situ instrument would have 366 sufficient sensitivity to detect such metallic iron. For example the lunar regolith contains on average 367 approximately 0.5 wt% metallic iron, with 0.20 ± 0.10 wt% in the size range 0.4 – 30 nm (Morris et al., 1998) 368 The MIMOS II instrument operating on the Mars Exploration Rover (MER) vehicles is capable of detecting 369 magnetite and hematite at the 1-2% level (Klingelhöfer et al., 2003). Newer generations of the instrument 370 (e.g. MIMOS-IIa) are expected to show a significant improvement in sensitivity, however, thus making space-371 borne Mössbauer and multi-frequency magnetic susceptibility measurements complementary (i.e. probing 372 different SD/SPM thresholds).

#### 373 3.3.2. Near-IR

Visible and near-IR reflectance spectroscopy are powerful tools to determine mineralogy remotely, typically from orbit. However, it is becoming more common for landed elements to also carry such an instrument, integrated, for example, into a microscope for close-up analysis. Comparison of the spectral properties of rocks and regolith on a small scale with their magnetic properties would be a useful combination to confirm the local effects of space weathering. These data could then be extrapolated to gain an understanding of global weathering properties from orbital remote sensing data.

380 3.3.3. XRS

381 X-ray spectroscopy (XRS) of asteroid 433 Eros has been used to argue in favour of a space weathered

382 surface, showing that the minor element ratios correspond well with those of ordinary chondrites (Foley et al.,

383 2006) and suggesting that this supports space weathering as the agent of sulphur depletion, rather than

partial melting. Space weathering preferentially removes the volatile components of the regolith, whilst leaving the abundances of rock forming minerals unchanged. Melting would, on the other hand, also change the ratios of other elements. Such indirect evidence for space weathering can therefore be measured with such techniques. However, the primary space weathering process (responsible for the major optical and magnetic effects) is a change of oxidation state, and the formation of metallic iron. This is more directly addressed by magnetic or Mössbauer measurements. Thus magnetic susceptibility and XRS are excellent complementary measurements for studying weathering and potential elemental depletion processes.

#### 391 3.4. Instrument design and development

Although no magnetic susceptibility sensor has yet been successfully deployed on a planetary surface, there is a rich history of proposed instruments and scientific rationales. In the pre-Apollo era, magnetic susceptibility was proposed as part of a surface and downhole instrument suite designed for characterising the lunar surface (Texaco, Inc., 1961). This suite was developed to breadboard level, but was of a similar size to terrestrial equipment.

397

Magnetic susceptibility instrumentation was then proposed as a future surface instrument after initial analysis of the lunar samples (Housley, 1977). Recent measurements of the terrestrial meteorite collection (Rochette et al., 2003 and 2008) have suggested the use of such a sensor on future asteroid (Rochette et al., 2004) and Mars (Rochette et al., 2006) missions. Finally, with attention turning to a human return to the Moon, magnetic susceptibility has been again proposed for identifying useful in situ resources; in fact the SPM metallic iron particles that are the key to space weathering could also allow the production of lunar "bricks" due to their absorption of microwave energy (Taylor and Meek, 2005).

405

A magnetic susceptibility instrument has been launched once previously, on the Phobos 2 mission. Carried onboard the PROP-F "hopping" lander (Kemurdzhian et al., 1988), it was a contact sensor using an AC bridge technique designed to make measurements of the magnetic susceptibility of Phobos at each hop (Dolginov et al., 1989). Unfortunately contact was lost with the orbiter before PROP-F was due to be deployed, so no measurements were made.

411

412 The utility of making multi-frequency magnetic susceptibility measurements should now be clear. However, 413 to describe a credible instrument concept, a set of top-level science and instrument requirements must be 414 defined. The range of expected values for bulk initial magnetic susceptibility must be chosen for the target

415 mineralogy; in lieu of detailed knowledge, one can baseline a range sufficient to cover typical lunar regolith 416 samples (1400 - 4400 10<sup>-8</sup> m<sup>3</sup> kg<sup>-1</sup> (Carmichael, 1989)). With laboratory instruments, a decade of frequency 417 difference is typically enough to establish the presence of SPM material, but this naturally depends on the 418 measurement accuracy of a field device. Extending this range will help, but will also enhance the ability of 419 such an instrument to perform magnetic granulometry. As noted, however, higher frequencies result in 420 additional energy dissipation in conductive samples due to the generation of eddy currents, but this can in 421 itself be useful for measuring electrical conductivity, if proper care is taken.

422

423 Of course this measurement cannot be made remotely and requires the sensor to be close to the regolith, 424 ensuring flux linkage with the material being measured. Several deployment mechanisms can be considered 425 for different mission profiles. As with all such sensors, mobility (horizontal or vertical) adds greatly to the 426 utility of the data and a magnetic susceptibility sensor could be deployed on a rover or even a sub-surface 427 penetrating mole. If deployed on a long-range rover, the opportunity is presented for recording maturity data 428 at several locations, for comparison with orbital data. This is vital for bodies other than the Moon where we 429 do not have the laboratory data to calibrate remote sensing techniques for separating compositional and 430 maturity variations, as has been performed for the Moon (Lucey et al., 2000). Alternatively, a mole-borne 431 sensor would be extremely useful for obtaining depth profiles and examining the stratigraphy of a single 432 location. Regolith cores extracted on the Moon show that there is a complex intermixing of layers of different 433 maturities at any given location, with a general trend towards decreasing maturity with depth (Basu and 434 McKay, 1995). Such a sensor would be able to examine this in situ for other bodies. In addition there is some 435 evidence for shock-produced fine-grained iron, which might be expected in a given layer with some lateral 436 extent, following a large impact (Cisowski et al., 1973). Layers of differing maturity could also be accounted 437 for by the creation of a palaeoregolith, in which layers of regolith with a given maturity are "sandwiched" 438 between lava flows of low susceptibility (Crawford et al., 2007).

439

Low mass and power magnetic susceptibility instruments already exist commercially for terrestrial applications, and have a suitable range and resolution for the measurements described here. Of course these are not space qualified and substantial re-development would be required before flight. Some of the potential issues that would arise are discussed here.

444

#### 445 **3.4.1. Coil geometry**

446 Of fundamental importance to any magnetic susceptibility instrument is the design of the coils. A choice of a 447 single coil (operating by self inductance) or multiple coils (mutual inductance) must first be made. There are 448 several advantages to a multi-coil design. Temperature variations affect both coils equally and so 449 temperature drifts are minimised. In addition, the geometry of the magnetic field can be optimised to focus 450 the region of investigation, resulting in finer spatial resolution, important for investigating layered structures in 451 a regolith, for example. The volume of material probed also depends on the spacing of the coils. Finally, 452 compensating coils can be used to minimise direct coupling between the transmitter and receiver. Once this decision has been made, the coil geometry must be optimised for the configuration and application. The aim 453 454 is to ensure maximum flux linkage with the medium in guestion; however additional constraints such as the 455 available volume and geometry must be taken into account.

456

457 Several coil geometries could be considered, in particular a planar configuration in which the transmitter and 458 receiver coils are arranged for a contact measurement, for example in the foot of a lander, rover wheel 459 (measuring once per revolution, and allowing a free-space calibration in between) or at the end of a robotic 460 arm. Alternatively a 3-coil linear arrangement, which could be included in an instrumented mole, can be 461 considered. The central coil can either be a transmitter or receiver, with a matched pair playing the opposite 462 role. However, other configurations could be considered, for example incorporating the coils into tracks on a 463 flexible substrate mounted on the inner wall of the mole and avoiding the difficulty of passing signal-carrying 464 wires through the coil.

#### 465 3.4.2. Calibration

Since measurement of susceptibility using an AC bridge is a relative technique, some method of calibration is required before each measurement. On the Earth this is performed by making a measurement away from any magnetic material, essentially giving a "zero". In the confines of a lander, and in particular during the penetration of a subsurface mole, this may be impractical.

470

Aside from zeroing in free space, the only realistic option that does not involve deployment booms, arms or other mechanisms, is to measure a known material when in close proximity to the sensor. If deployed in a mole, the hammering mechanism could offer a ready solution to this problem by manufacturing the mechanism from a magnetic alloy with stable properties. During each cycle a zeroing measurement could be made when the hammer was close to the sensor. However, a suitable material must be found that has

476 properties that do not change over time, with temperature, and with the energising frequency etc.

477 Alternatively a single-point calibration can be performed by incorporating a calibration material into the

478 housing of the instrument deployment mechanism.

479

A secondary issue is calibration of the volume over which the sensor integrates. Measurements of volume susceptibility rely on the sample filling the volume over which the sensor is sensitive. Even so, density changes in a material can be mistakenly recorded as a difference in magnetic susceptibility. For this reason laboratory measurements are often made using mass specific susceptibility, which is normalised by the sample mass (and hence density). Since in situ measurements cannot easily determine the sample mass, additional steps must be taken to ensure consistent values.

486

487 Gattacceca et al. (2004) have shown a method for determining the integration volume of an AC magnetic 488 susceptibility sensor based on measurements alone. This relies on taking measurements at different 489 distances from the sample, such that a different fraction of the integration volume is filled (and recorded) at 490 each distance. This method is suitable for deployment on a rover or robotic arm, but not on a mole. In this 491 case measurement of the bulk density surrounding the instrument could be used to ensure inter-492 comparability between measurements. Such a sensor, using gamma backscatter densitometry, has already been proposed and built to a breadboard level in a prototype of the HP<sup>3</sup> mole-borne instrument package 493 494 (Ambrosi et al., 2006; Spohn et al., 2001).

495

A final point to note is that such a sensor would be rather sensitive to the presence of metal in the instrument housing, or indeed the host spacecraft itself. This can already be seen from laboratory measurements where care has to be taken over the location of the instrument; conductive losses reduce the measured magnetic susceptibility. There are therefore some additional restrictions placed on where and how a magnetic susceptibility sensor can be mounted.

501 3.4.3. Thermal and mechanical stability

The value of inductance of a coil is strongly affected by the mechanical stability of the coil windings. Careful choice of the (electrically insulating) former would have to be made to ensure a consistent inductance over the expected temperature range. Continuous measurement of temperature and also "blank" measurements of a calibration material before and after measurement of the actual sample can both be used to remove this drift from the measured value.

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508 While temperature stability of the coil is vital, additional science is possible if the sample temperature can be

509 varied, either actively within the instrument, or passively during, for example, a diurnal cycle. An ideal

510 instrument design would allow the sample to be heated whilst insulating the coil itself from such changes,

511 maintaining the stable oscillator frequency. This is discussed further in section 3.5.

512 3.4.4. Choice of frequency

513 One of the critical parameters in making an AC susceptibility measurement is the frequency at which the 514 oscillator is energised. In typical magnetic susceptibility meters, a low frequency is used to prevent 515 conductive losses in the sample medium. However, this must be traded against the desire to increase the 516 frequency range (and hence the iron particle size range) available for magnetic granulometry of 517 superparamagnetic grains.

#### 518 3.5. Additional science

519 Not only can such a sensor measure the bulk magnetic susceptibility and search for the superparamagnetic 520 iron indicative of space weathering, but it can also potentially measure electrical conductivity and magnetic 521 mineralogy. When recording magnetic susceptibility in a two-coil (transmitter and receiver) configuration, the 522 response of the coil is measured in phase with the driving current, but the quadrature component is also 523 useful, providing a measure of electrical conductivity. Indeed this technique is often used in terrestrial 524 borehole logging. The internal magnetic field generated by the movement of domain walls and the rotation of 525 domain magnetic moments corresponds to the magnetic susceptibility of the sample and this results in the 526 in-phase signal. The quadrature component represents losses in the sample, from both magnetic hysteresis 527 and eddy currents. Thus the magnetic susceptibility is written as a complex quantity when frequency is 528 considered. As well as being of intrinsic scientific interest, electrical conductivity is of particular interest at the 529 surface of Mercury, where it has been suggested that surface conductivity may play a role in the closure of 530 field aligned currents believed to be generated during magnetic substorms, first detected by Mariner 10 531 (Janhunen and Kallio, 2004).

532

Another common technique in terrestrial geomagnetic laboratories is thermomagnetic analysis, whereby the magnetic susceptibility is recorded as a function of temperature. Ferromagnetic materials will show a sudden drop in susceptibility at their Curie temperature. Hence measurements of a particular regolith sample over a range of temperature could aid in the identification of magnetic phases, as previously suggested by Rochette

537 et al. (2004). These temperature changes could either be natural, such as those due to diurnal oscillations, 538 or as a result of active heating. Building a coil around, for example, the oven of another instrument might be 539 a way of making magnetic measurements over a range of temperatures. In addition, Equation 1 540 demonstrated that temperature also controls the critical grain size, and thus heating a sample shifts the 541 superparamagnetic boundary to larger particle sizes, allowing a small degree of granulometry as seen in 542 Figure 3. Again, the dependence is weak, but nonetheless useful. In terrestrial laboratories samples 543 measured in this way inevitably partially oxidise, however much care is taken, but the vacuum environment 544 of an airless planetary body is ideal for such measurements. It has also been shown that the shape of the 545 thermomagnetic curve far below the Curie point is also diagnostic of the size distribution of a single domain 546 ensemble (Stephenson, 1971a). Griff

- 547
- 548

#### [Figure 3]

6

#### 549 4. Conclusions

550 Micrometeorite impacts and solar wind sputtering are now understood to produce nanophase metallic iron 551 from ferrous iron in airless planetary regoliths during the regolith maturation / space weathering process. 552 Metallic iron is ferromagnetic and hence amenable to a wide range of magnetic techniques.

553

554 Magnetic susceptibility is known to correlate well with the more frequently used ferromagnetic resonance 555 index used to describe regolith maturity and thus is a useful parameter for understanding the regolith 556 evolution at a given location. In addition, multi-frequency measurements of susceptibility can be used to 557 identify the very fine grained SPM iron particles resulting from space weathering. Definitive confirmation of 558 space weathering as the agent of spectral alteration on asteroids and other planetary surfaces besides the 559 Moon would be extremely useful.

560

561 Laboratory weathered regolith analogues have confirmed both an increase in initial susceptibility and the 562 introduction of a frequency dependence, along with lunar-like spectral alteration, for even low degrees of 563 alteration. Thus a multi-frequency magnetic susceptibility sensor has been proposed as a means of 564 identifying and potentially quantifying the effects of space weathering in a planetary regolith surface. Direct 565 granulometry with this technique has a rather limited range, but coupled with temperature dependent 566 measurements and complementary instrumentation inferences could nonetheless be made. Such data could

567 eventually be used with models of the spectral alteration process to remove some of the changes caused by 568 space weathering from remote spectra.

569

570 This measurement could be carried out most easily by an AC method in which the change in inductance of a

571 coil placed close to a sample provides a measure of magnetic susceptibility. Terrestrial instruments with low

572 mass and power exist, but the challenges in developing a space-borne instrument are not trivial. In

573 particular, the thermal stability of the instrument is key to achieving a high precision measurement.

574 Deployment as part of a mole-borne instrument suite including temperature and density measurements

575 would be extremely complementary, but the required high level of miniaturisation and integration and risk of

576 potential interference suggest that a surface contact instrument may be more feasible.

577

578 Additional properties that could be measured with such an instrument include mineralogy (through Curie

579 point identification if the sample temperature can be varied), and electrical conductivity. Measurements of the

580 susceptibility with temperature can also help to further constrain the metallic iron grain size distribution.

.n the

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#### 750 Figure captions

751

Figure 1. Theoretical calculation of the size dependence of volume magnetic susceptibility of spherical

753 metallic iron at room temperature. Several different AC measurement frequencies are shown, and the

susceptibility is scaled relative to that of SD grains for comparison. It can be seen that SPM grains can have

a susceptibility an order of magnitude higher than SD grains, and that the SPM/SD boundary shifts to smaller

756 grains as the measurement frequency increases.

757

Figure 2. The SD/SPM boundary for metallic iron grains at room temperature as a function of frequency, on a

759 logarithmic scale. It is clear that the nature of the dependence allows only a small degree of granulometry to

be performed. Realistically, measurements would likely only be performed at low frequencies where

761 dissipative effects can be avoided.

762

Figure 3. The SPM/SD boundary changes with temperature as well as frequency, although again the

dependence is relatively weak. In reality the curve is more complex since other parameters, for example the

765 saturation magnetisation, are temperature dependent.

Accepted





