Magnetic susceptibility measurement of single iron/cobalt carbonyl microcrystal by atmospheric magnetophoresis

Masayori Suwa¹, Yuichiro Oshino¹, Hitoshi Watarai¹, Hiroshi Morita², Anzu Kasai³ and Jan Šubrt⁴

¹ Department of Chemistry, Graduate School of Science, Osaka University, 1-1 Machikaneyama, Toyonaka, Osaka 560-0043, Japan
² Graduate School of Advanced Integration Science, Chiba University, Yayoi-Cho, Inage-ku, Chiba 263-8522, Japan
³ Graduate School of Science and Technology, Chiba University, Yayoi-cho, Inage-ku, Chiba 263-8522, Japan
⁴ Institute of Inorganic Chemistry, Academy of Sciences of the Czech Republic, 25086 Řež near Prague, Czech Republic

E-mail: watarai@chem.sci.osaka-u.ac.jp

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Abstract
In this study, the use of an innovative atmospheric magnetophoresis, which enables us to measure the mass magnetic susceptibility and mass of a microparticle simultaneously, was demonstrated. Using this technique, we determined the magnetic susceptibility of a crystalline deposit of iron/cobalt carbonyl, mainly composed of Fe₂(CO)₉, which was prepared photochemically from a gaseous mixture of iron pentacarbonyl (Fe(CO)₅) and cobalt tricarbonyl nitrosyl (Co(CO)₃NO). The mass magnetic susceptibility and the characteristic relaxation time of the microcrystal were (7.0 ± 1.9) × 10⁻⁹ m³ kg⁻¹ and (5.6 ± 2.2) × 10⁻⁴ s, respectively. The observed magnetic susceptibility shows that the microparticle was paramagnetic. Assuming that the density was equal to that of Fe₂(CO)₉ (2.1 × 10³ kg m⁻³) and that the shape of the particle was spherical, a hydrodynamic radius of 4.7 μm and a mass of 0.91 ng were observed. It was suggested that Co was incorporated in Fe₂(CO)₉.

Keywords: magnetophoresis under atmosphere, magnetic susceptibility, gas phase photochemical reaction, iron pentacarbonyl, cobalt tricarbonyl nitrosyl

(Some figures in this article are in colour only in the electronic version)

1. Introduction
Many micrometer-sized particles exist in nature, such as cells, cell composites, DNA, environmental colloid particles, soil particles, and clay crystals. These are composed of many small ions and molecules, but each microparticle has characteristic functions as an individual. For instance, cells are unequivocally the basic building block of living organisms. Therefore, the separation and analysis of a single microparticle is necessary to elucidate the function of microparticles in real systems. To perform them, nondestructive and noninvasive measurement techniques for the properties of an ‘individual’ single microparticle are extensively required in different research fields, such as biology, environmental chemistry, and medical science [1–3].

Migration analyses in liquid have great potential for satisfying the above requirement. In liquid, the force acting on microparticles from an external field is instantly balanced by the viscous drag force, so that migration velocity is directly proportional to the force. The force must
be a function of the physical or chemical properties of microparticles closely related to external field. Therefore, the measurement of migration velocity under an external field allows one to estimate the properties of microparticles. For example, in electrophoresis, the surface ζ-potential of microparticles can be calculated from electrophoretic velocity, and during centrifugation, the specific gravity can be estimated. We have developed several kinds of migration technique employing various external fields over the past decade [4–32]. Dielectrophoresis [5–10], which is observed under a nonuniform electric field, enables the determination of the polarizability of microparticles. Laser photophoresis [11–16], driven by the radiation pressure of photons, allows us to estimate the reflectance, refractive index, and absorption coefficient and other optical properties of microparticles, and to separate them by utilizing the difference in such properties. Electromagnetophoresis [17–22], which is migration under a homogeneous magnetic field and an electric current, has great potential not only for characterizing microparticles by conductivity measurement, but also for measuring the adhesion force between microparticles in a liquid and the cell wall by measuring desorption current. Magnetophoresis [23–30] can be observed under an inhomogeneous magnetic field. Magnetic susceptibility can be determined from magnetophoretic velocity. Magnetic susceptibility is one of the most important properties in inorganic chemistry, material science, and other research fields, because it reflects the valence electron state and electron spin state of the component molecules.

Although magnetophoretic migration analysis in liquid media has great potential for characterizing microparticles, it cannot be used in the case of measuring the magnetic susceptibility of microparticles grown in gaseous phase, which are not preferable to be dispersed in liquid, since the number of valence electrons or unpaired electron spins, which mainly governs the magnetic susceptibility of elements, varies with the redox potential of the solution, with crystal fields affected by the coordinating ligand, and even with the state of solvation. Recently, the magnetic field effect on the chemical composition of several microparticles grown in gaseous phase via photochemical reaction has been discovered [31–33]. When an external magnetic field was applied, the chemical composition and morphology of deposited microparticles varied [34]. The chemical composition could be determined by several techniques such as infrared absorption spectroscopy, x-ray photoelectron spectroscopy (XPS), and electron probe microanalysis. To explain the mechanism of the magnetic field effect, the measurement of magnetic susceptibility is very important. However, it is difficult to measure the magnetic susceptibility of individual microparticles formed in gaseous phase. Therefore, a new method of determining the magnetic susceptibility of these materials is necessary.

Nowadays, new techniques such as magnetic force microscopy (MFM) [35–37] and scanning superconducting quantum interference device (SQUID) microscopy [38–42] have been developed mainly to observe the magnetization state of the surface of high-density recording media. These methods are based on scanning probe microscopy (SPM). In MFM, the tip of the needle of atomic force microscopy (AFM) probe is coated with a magnetized thin film, and the magnetic force between a sample and the needle is measured. By this method, a magnetic momentum less than $10^{-16}$ A m$^2$ can be detected by measuring the force of the nN order very close to the sample (~5 nm) [43] with a spatial resolution less than 30 nm. The SPM utilizing micro-SQUID as a probe is called a scanning SQUID microscope, whose sensitivity makes it possible to measure the magnetic momentum of about $10^{-17}$ A m$^2$ [44]. However, these two methods have disadvantages in measuring the induced magnetic momentum of a microparticle. In a SQUID microscope, the probe should be cooled and strictly shielded from an environmental magnetic field [45]. Therefore, the measurement at room temperature with high resolution is difficult. No micrometer spatial resolution is achieved for room-temperature samples. The disadvantage of MFM is that a high magnetic field cannot be applied, because the magnetization of the thin film on the needle varies. Therefore, no induced magnetic momentum of a μm-sized paramagnetism or diamagnetism (less than $10^{-17}$ A m$^2$ under 10 T as mentioned above) can be measured. Hence, an alternative technique is necessary to characterize single microparticles with magnetism.

More recently, a novel type of magnetophoretic technique has been developed, which is analogous to the concept of ion mobility mass analysis [46, 47]. The magnetophoretic velocity in gaseous phase can include inertia, which contains mass information. Hence, from the magnetophoretic acceleration and velocity measurements under atmospheric conditions, the magnetic susceptibility and mass of a single microparticle could be determined independently and simultaneously. In this article, we have applied the new method to the measurement of the magnetic susceptibility of individual iron/cobalt carbonyl microparticles, which were synthesized by irradiating UV light on a gaseous mixture of iron pentacarbonyl (Fe(CO)$_5$) and cobalt tricarbonyl nitrosyl (Co(CO)$_3$NO) and observed to align perpendicularly to the applied magnetic field. This material was expected to show a very weak magnetism as measured by MFM or SQUID microscopy, because it was composed mainly of diamagnetic Fe$_2$(CO)$_9$.

2. Experimental

2.1. Sample preparation

Using photochemical reactions of gaseous molecules, ultranine particles and crystalline deposits were produced. Iron pentacarbonyl (Fe(CO)$_5$) (Kanto, 95%) and cobalt tricarbonyl nitrosyl (Co(CO)$_3$NO) (Gelest, 95%) were degassed by freeze-pump-thaw cycles in the dark and purified by vacuum distillation immediately before use. To prepare a gaseous mixture, each vapor was introduced successively into a cross-shaped Pyrex cell having long (length 155 mm, inner diameter 35 mm) and short (length 80 mm, inner diameter 20 mm) arms or into a small cylindrical Pyrex cell (length 160 mm, inner diameter 20 mm, volume 50 cm$^3$) equipped with a couple of quartz windows through a vacuum line.
Figure 1. (a) Experimental setup for observation of magnetophoresis under atmospheric conditions, (b) configuration of magnets and pole pieces and (c) slit array mask.

equipped with a capacitance manometer (Edwards Barocel Type 600). The background pressure of the irradiation cell was less than $8 \times 10^{-5}$ Torr (1 Torr = 133.3 Pa). The partial pressures of Fe\(\text{CO}_5\) and Co\(\text{CO}_3\)NO in the irradiation cell were determined from the diagnostic band intensities of FT–IR spectra at 645 cm\(^{-1}\) for Fe\(\text{CO}_5\) and at 2108 cm\(^{-1}\) for Co\(\text{CO}_3\)NO. The gaseous samples were irradiated with a medium-pressure mercury lamp (Ushio UM-452, 450 W) through UV29 and UVD33S filters (energy, 5.4 mJ s\(^{-1}\) cm\(^{-2}\)) to excite both Fe\(\text{CO}_5\) and Co\(\text{CO}_3\)NO molecules at 313 and 365 nm, respectively. The crystalline deposits and sedimentary particles were deposited on a glass plate or a Cu substrate placed in the irradiation cell.

2.2. Experimental setup

The overview of the apparatus used in this study is shown in figure 1(a). An inhomogeneous magnetic field was generated by a pair of Nd–Fe–B permanent magnets and iron pole pieces 13 mm in length, 5 mm in width and 3 mm in thickness. The configuration around the observed region is illustrated in figure 1(b). The gap between the pole pieces was 0.8 mm. A large gradient of magnetic field was obtained at the edge of the pole pieces [26]. We defined the direction of gravity as the x-direction and the center of the pole piece as $x = 0$. The region where migration velocity and acceleration were measured was near the edge of pole pieces ($x = -1.7$ to $-1.3$ mm). The magnetic field strength of the observed region was measured using a Gauss meter. A single microparticle adhered on a cover glass was selectively desorbed using a Nd:YAG laser (New Wave Research, MiniLaseII, 532 nm, 5–7 ns pulse duration, 1.1 mJ pulse\(^{-1}\)) and was dropped into the gap of the pole pieces. The microparticle was irradiated with a continuous-wave He–Ne laser light (Melles Griot, 632.8 nm, 0.8 nm of beam waist, 15 mW). The laser beam was thinned down using a cylindrical lens to prevent stray scattering by the pole pieces. The direct laser beam was cut off by the back of the convex mirror, and then the scattered light was effectively collected by a reflective objective. The collected light was focused on a slit array mask (see figure 1(c)) on the image plane of the microscope. Only when the image was on a slit, the light passed through the mask and was detected by a photomultiplier tube. The photocurrent signals were observed as 13 spikes by a digital oscilloscope. From the spikes, the velocity and acceleration of the particle were measured. The measurement was carried out in a thermostatted room at 25 ± 1 °C.

2.3. Other measurements

Scanning electron microscopy (SEM) images of crystalline deposits were obtained with a JEOL JSM 6060 scanning electron microscope. EDX–SEM was performed using a Philips XL30 CP scanning electron microscope. The crystalline deposits and sedimentary particles were mixed with KBr powder to prepare KBr pellets, and the FT–IR spectra of the deposits embedded in the pellets were measured with a Nicolet NEXUS 470 FT–IR spectrometer. Magnetic field was applied by a helium-free superconducting magnet (Toshiba TM-5SP) during photochemical reactions of gaseous molecules.

3. Results and discussion

3.1. Chemical component of sedimentary particles produced from gaseous Fe\(\text{CO}_5\)/Co\(\text{CO}_3\)NO mixture

Under UV light irradiation at 313 nm for 30 min, a gaseous mixture of Fe\(\text{CO}_5\) (2.3 Torr) and Co\(\text{CO}_3\)NO (1.7 Torr)
produced crystalline deposits with sizes of \( \sim 20 \mu m \) and \( \sim 5 \mu m \), respectively, in addition to a small number of spherical sedimentary particles with a mean diameter of 0.42 \( \mu m \), as shown in figure 2. The strong bands in the FT–IR spectrum of the crystalline deposits shown in figure 3(a) coincided with the bands of the deposits produced from pure Fe(CO)\(_5\) vapor (figure 3(b)), which are assigned to Fe\(_2\)(CO)\(_9\) [48, 49], showing that the main chemical component is Fe\(_2\)(CO)\(_9\). To study whether Co(CO)\(_3\)NO was incorporated into crystalline deposits, EDX–SEM was performed on the crystalline deposits produced from a gaseous mixture of Fe(CO)\(_5\) (2.4 Torr) and Co(CO)\(_3\)NO (1.3 Torr). The populations of Fe, Co, C, and O atoms are listed in table 1. The atomic ratio of Co atom to Fe atom was 1 : 13, showing that Co(CO)\(_3\)NO molecules are incorporated into crystalline deposits.

3.2. Magnetic alignment of crystalline deposits

A crystalline deposit with a size of \( \sim 20 \mu m \) was produced from a gaseous mixture of Fe(CO)\(_5\) (2.4 Torr) and Co(CO)\(_3\)NO (1.3 Torr) under the application of a magnetic field of 5 T during light irradiation. The crystalline deposit grew into a hexagonal shape and the long axis of the crystal aligned perpendicularly to the applied magnetic field, as shown in figure 4. This might be caused by the magnetic anisotropy of the deposit. As shown in table 1, the population of Co atoms increased with increasing magnetic field. In the preliminary XPS, Co 2p\(_{3/2}\) electrons were detected at a binding energy of 780.8 eV, indicating the incorporation of divalent paramagnetic Co atoms in the crystalline deposit.

3.3. Magnetic susceptibility measurement by atmospheric magnetophoresis

Magnetic susceptibility was measured for the crystalline deposits with a size of \( \sim 20 \mu m \), which was produced from a gaseous mixture of Fe(CO)\(_5\) (2.3 Torr) and Co(CO)\(_3\)NO (1.7 Torr) under light irradiation at 313 nm for 10 min. Figure 5 shows a typical oscillogram obtained...
in this experiment. Thirteen light spikes were detected by a photomultiplier tube for a single atmospheric magnetophoresis of a microparticle. The velocity, $v$, and acceleration, $a$, were calculated from the intervals of the spikes while taking into account the distance between the slits on the conjugated object plane (34 $\mu$m). The equation of motion under the inhomogeneous magnetic field can be written as

$$ma = mg + \frac{\chi_g}{\mu_0} m \frac{dB}{dx} - f v. \quad (1)$$

The first term on the right-hand side of equation (1) represents the gravity where $m$ is the mass and $g$ is the gravitational acceleration; the second term denotes magnetic force where $\chi_g$ is the mass magnetic susceptibility, $\mu_0$ the vacuum magnetic permeability, and $B$ the magnetic flux density; and the last term is the drag force where $f$ is the friction coefficient. Rearranging equation (1) yields

$$\frac{a-g}{B(\frac{dB}{dx})} = -\frac{1}{\tau} \left[ \frac{v}{B(\frac{dB}{dx})} \right] + \frac{\chi_g}{\mu_0}, \quad (2)$$

where $\tau$ is the relaxation time of translation motion, $\tau = m/f$. Figure 6 shows a linear relationship of $(a-g)/B(\frac{dB}{dx})$ versus $v/B(\frac{dB}{dx})$. From equation (2), the intercept afforded a mass magnetic susceptibility of the crystalline deposit of $(7.0 \pm 1.9) \times 10^{-9} \text{ m}^3 \text{ kg}^{-1}$. The crystalline deposit was slightly paramagnetic. Relaxation time was also calculated from the slope of the plots of $(5.6 \pm 2.2) \times 10^{-4} \text{ s}$. Assuming that the particle is a sphere, Stokes’ law can be applied on the drag force, that is, $f = 6\pi \eta r$, where $\eta$ is the viscosity of air $(1.8 \times 10^{-5} \text{ Pa s})$ and $r$ is the radius of a microparticle. Therefore, the radius of a microparticle can be expressed as

$$r = \sqrt{\frac{9\eta v}{2\mu}}. \quad (3)$$

A radius of $4.7 \pm 1.8 \mu$m and a mass of $0.91 \pm 0.35 \text{ ng}$ were estimated, provided that the density of the particle was equal to that of Fe$_2$(CO)$_9$ $(2.1 \times 10^3 \text{ kg m}^{-3})$ and the shape was spherical. The estimated radius seems smaller than that estimated from the SEM images. Two possible reasons can be considered; firstly, the microparticle may be broken by pulsed laser irradiation, if it is fragile. Secondly, the approximation of the sphere was too rough for these crystalline deposits, if the particle was not dense or spherical and was ellipsoidal, flat, or cubic. However, the size had no effect on the estimation of mass magnetic susceptibility, as noticed in equation (2). Therefore, the estimated value of mass magnetic susceptibility is reliable.

Finally, we estimated the effective magnetic momentum, $\mu_{\text{eff}}$ (B.M., which represents Bohr magneton), of this material, which can be calculated using

$$\mu_{\text{eff}} = \frac{3k_B T \chi_g \rho}{N_a M \beta^2}, \quad (4)$$

where $k_B$ is the Boltzmann constant, $T$ the temperature (298 K), $N_a$ Avogadro’s number, $M$ the molecular weight (Fe$_2$(CO)$_9$ = 363.8) and $\beta$ is the B.M. unit. The substitution of the experimental $\chi_g$ yields $\mu_{\text{eff}} = 1.0 \text{ B.M.}$, which is smaller than that of the mononuclear complex of a transition metal with 1 unpaired 3d electron spin (1.73 B.M.). Fe$_2$(CO)$_9$ is a typical molecule, to which the 18-electron rule can be applied. Therefore, this molecule must exhibit diamagnetism, since all its valence electrons should be paired. However, the present experimental results indicate that the examined particle was slightly paramagnetic, so that it can be concluded that the incorporation of Co atoms into the crystalline deposit of Fe$_2$(CO)$_9$ has a significant contribution to magnetic susceptibility.

4. Conclusion
An iron/cobalt carbonyl crystalline deposit was prepared from a gaseous mixture of iron pentacarbonyl and cobalt tricarbonyl.

Figure 5. Typical oscillogram obtained for single microparticle. The asterisks indicate the peak positions.

Figure 6. Plots of $(a-g)/B(\frac{dB}{dx})$ versus $v/B(\frac{dB}{dx})$. The solid line was obtained by the least-squares method.
nitrosyl by UV light irradiation. The magnetic field effect on the chemical composition of iron/cobalt carbonyl deposit was observed. The magnetic susceptibility of the crystalline deposit was measured by atmospheric magnetophoresis. The deposit was slightly paramagnetic. The major component of the deposit was determined to be Fe$_2$(CO)$_6$ from the FT-IR spectrum. The apparent radius was determined to be 4.7 μm, under the assumption that the deposit was spherical and that its density was equal to that of Fe$_2$(CO)$_6$. This value is reasonable, since theanalyte seemed to be easily broken from the electron microscope observation. Fe$_2$(CO)$_6$ should be diamagnetic, but the observed magnetic susceptibility of $(7.0 \pm 1.9) \times 10^{-6}$ m$^3$ kg$^{-1}$ indicated it to be paramagnetic, suggesting the paramagnetic contribution of the Co atoms incorporated into Fe$_2$(CO)$_6$.

In this study, the advantage of atmospheric magnetophoresis for the determination of an individual weak magnetic microcrystalline deposit, which could not be dispersed in liquid media, was demonstrated. To our best knowledge, no other method could perform magnetic susceptibility measurement for this material. The present technique will be applied to the analysis of aerosol particles, single microcrystals, and a few cell composites among others. Nanoparticles could also be measured using an improved sample introduction method.

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