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# Superparamagnetic materials

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**Abstract**

Properties and applications of superparamagnetic materials, i.e. materials composed of very small magnetic grains, are briefly presented. After a short introduction, theory describing a single idealised grain is laid out and some magnetic properties of superparamagnets (FC and ZFC curves, $M(H)$ curve) are explained. There are many practical applications of superparamagnetic materials. For example ferrofluids are used for liquid seals, efficient heat transfer and damping. Even more importantly, superparamagnetic materials play a major role in the hard disk drive technology. Various challenges related to thermal stability and writability stem from the ever decreasing size of magnetic grains. Currently four cutting edge HDD technologies that either sidestep or solve problems related to magnetic grains are being developed: heat-assisted magnetic recording, bit-patterned recording, shingled magnetic recording and helium filled HDDs.

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# 1 Introduction

**Definition** A material is superparamagnetic if it is made of very small single-domain non-interacting magnetic[1] grains dispersed in some non-magnetic medium. How small is *very small* depends on properties of magnetic grains. Typical value of their diameter is on the order of 10 nm. Since properties of superparamagnetic materials depend crucially on the spontaneous magnetic moment of magnetic grains, it is implicitly understood that temperature is below the Curie or Néel temperature $T_C$ of magnetic grains. It doesn't make sense to talk about superparamagnetism above $T_C$.

**Origin of name** Above the so called blocking temperature $T_B$, which is material and experiment dependent (see Section 2.1), superparamagnet can be described by the same equations (details in Section 2.2.3) that are used for ordinary paramagnet (ensemble of non-interacting magnetic moments $\boldsymbol{\mu}$ with energy $E = -\boldsymbol{\mu}\boldsymbol{B}$ when put in magnetic field $\boldsymbol{B}$), the only difference being that magnetic moments of individual particles are much larger in the case of superparamagnet (single atoms vs. grains composed of 1000s of atoms). Therefore magnetization curve $M(H)$ of a superparamagnet is similar to that of a paramagnet, but much steeper (higher susceptibility). This is the reason for the name *super-paramagnet*. Below $T_B$ superparamagnet's magnetization curve $M(H)$ has hysteresis and is thus more similar to ferromagnet's magnetization curve.

**Occurrences and applications** Superparamagnetic materials play an important role in many areas of technology. In this seminar I will take a closer look at superparamagnetic materials in hard disk drives (HDDs) and at ferrofluids.

- In hard disk drives information is stored by magnetizing small pieces of magnetic material on the platter. The former is composed of magnetic nanograins.
- Ferrofluids, which are a colloidal liquid of nanoscale magnetic grains suspended in a carrier liquid, are used for liquid seals (e.g. in HDDs), efficient heat transfer (e.g. in loudspeakers), in suspension systems and in various medical applications. They also look nice when put into vicinity of a permanent magnet (Figure 4).

[1] In this seminar I use term magnetic in two different but related meanings. Sometimes I use it as a shorthand for "ferri- or ferro-magnetic" and sometimes I use it to signify a property of non-zero spontaneous magnetization. The first meaning is used when I talk about magnetic grains and the second one when I talk about magnetic material on HDD platter.

Superparamagnetic materials are also found in nature in various rocks and living organisms [1]. Some companies even sell magnetic nanograins extracted from horse spleen (that is not the primary way of production though) [2].

## 2 Properties

First we will take a look at the theory describing a single magnetic grain. Then some properties of superparamagnets will be presented and explained in terms of properties of a single grain.

### 2.1 A single grain

Let's for the sake of simplicity assume the following:

- Grain is composed of a single ferro- or ferri-magnetic domain and its Curie or Néel temperature $T_C$ is higher than temperature we are observing the grain at. Both conditions are rather mild since many materials with domain sizes on the order of µm $\gg$ 10 nm and with $T_C \gg T_{\mathrm{room}}$ exist.

- Grain has only uniaxial magnetic anisotropy, which means that there are two easy directions of magnetization[2] pointing in opposite directions. We need grain with some magnetic anisotropy, so that it can hold its magnetization, and uniaxial is the simplest one and it usually dominates [2].

Energy of a single-domain magnetic grain with uniaxial anisotropy in external magnetic field $H$ is sum of its magnetic anisotropy and Zeeman energies [18],

$$E = KV \sin^2(\phi - \theta) - \mu_0 M_s V H \cos\phi, \quad (1)$$

where $V$ is the volume of a grain, $K$ is uniaxial anisotropy parameter and $M_s$ is saturation magnetization. External magnetic field $H$, grain's magnetization and easy axis of magnetization all lay in the same plane. $\phi$ is the angle between magnetization and magnetic field, while $\theta$ is the angle between easy direction of magnetization and magnetic field.

For the sake of simplicity lets make external field parallel to easy axis of magnetization, $\theta = 0$. Dependence of energy on angle $\phi$ between grain's magnetization and its easy direction of magnetization (which by our simplification equals direction of external magnetic field) is shown on Figure 1.

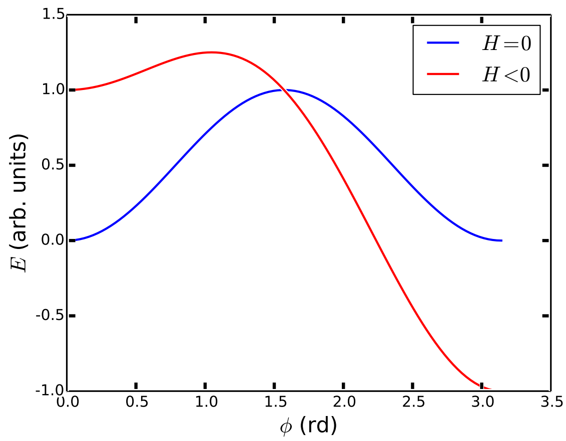


Figure 1: Schematic dependence of grain's energy on angle between its magnetization and easy direction of magnetization. Dependence is calculated from Equation (1). Source of figure: my own work.

We see that in absence of external magnetic field there are two energetically equally favorable directions (see blue curve on Figure 1).

[2]Easy direction of magnetization is an energetically favorable direction of spontaneous magnetization that is determined by the magnetic anisotropy [4]. When anisotropy is uniaxial, there are two easy directions of magnetization that point in opposite directions along the easy axis.

They are both parallel to easy axis of magnetization and between them is energy barrier $\delta E$ of size $KV$. If temperature is high enough, thermal energy $kT$ can overcome the barrier and grain's magnetization changes direction.

Non-zero magnetic field breaks the symmetry between the two magnetizations along the easy axis (see red curve on Figure 1). Energy of a grain is lower when magnetization points along the external magnetic field than when it points in the opposite direction. Also energy barrier for jumping from magnetization along the external field to magnetization opposite to external field is much bigger than for the reverse.

The thermal fluctuations of the magnetization direction between the easy directions are called *superparamagnetic (Néel) relaxation* and typical time between the flops $\tau$ is given by the Néel-Brown expression

$$\tau = \tau_0 \, e^{\frac{\delta E}{kT}},$$

where $\tau_0$ is usually between $10^{-12}$ s and $10^{-9}$ s. $\tau_0$ depends weakly on temperature and various material parameters such as magnetic anisotropy constant, particle volume and saturation magnetization. [2]

The measured magnetization of the grain will depend crucially on the timescale $t_{\mathrm{exp}}$ of the experiment. If $t_{\mathrm{exp}} \ll \tau$, the magnetization will appear static, while if $t_{\mathrm{exp}} \gg \tau$, the average value will be measured (in absence of external field this will be 0). In practice the two regimes are classified in terms of temperature instead of time. A *blocking temperature* $T_B$ is defined [2]; it is the temperature at which the relaxation time $\tau$ equals the experiment timescale $t_{\mathrm{exp}}$. Note that the blocking temperature is not uniquely defined for a given material, because it depends on timescale $t_{\mathrm{exp}}$ of the experimental technique used.

## 2.2 A superparamagnet

In practice one usually deals with matter that is composed of a large number of magnetic grains. Let's investigate some properties of such systems.

### 2.2.1 ZFC and FC magnetization curves

These two curves are noteworthy because it is easy to estimate two important properties (blocking temperature and strength of interaction between particles) of a monodisperse sample just by quickly looking at the graphs of temperature dependence of susceptibility $\chi(T)$.

The dependence $\chi(T)$ in both ZFC (zero field cooled) and FC (field cooled) case is measured during heating of a sample in small (e.g. $10^4$ A/m) external magnetic field. Curves are different because samples have different initial state. In the ZFC case, the sample was prepared for measurement by being cooled from some high temperature ($T \gg T_B$) in absence of external field. In contrast, the FC sample was cooled in non-zero external magnetic field [2]. An example of FC and ZFC curves is shown in Figure 2.

At low temperatures, susceptibility measured in the ZFC case is small, because the timescale of the experiment is too short for the sample to reach thermal equilibrium value of magnetization determined by canonical distribution. At thermal equilibrium the ratio of particles with magnetic moment in direction of external field to particles with magnetic moment in direction opposite to external field (all other orientations are also possible, but it's easier to illustrate the idea by limiting to only two opposite directions) is proportional to $e^{\frac{\Delta E}{kT}}$, where $\Delta E$ is energy difference between the two orientations. At high temperatures susceptibility is low because the value of

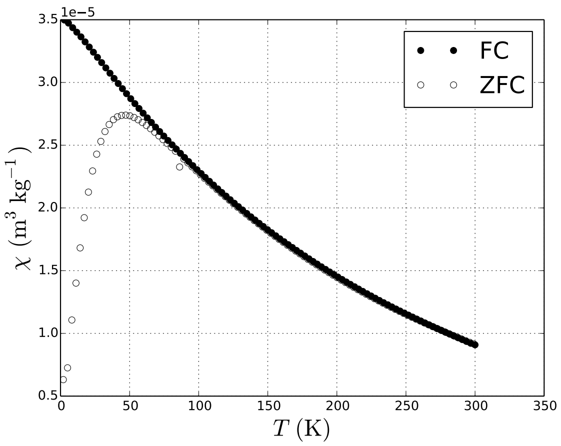


Figure 2: ZFC and FC curves for sample of maghemite ($\gamma\mathrm{-Fe_2O_3}$) magnetic grains with average diameter of 8 nm that are coated with oleic acid and dispersed in carnauba wax. The dependant variable on the plot is mass susceptibility, $\chi = \frac{\mu}{mH}$. Mass fraction of maghemite grains is 0.7 %. Blocking temperature can be estimated as the position of ZFC maximum — it's about 46 K. Source of figure: my own work.

magnetization in thermal equilibrium is small (again, proportional to $e^{\frac{\Delta E}{kT}}$). The peak is reached somewhere in between and it turns out that maximum for a monodisperse sample is at $T_B$.

The shape of FC curve is different from ZFC curve below $T_B$ as during the cooling process in magnetic field the system has enough time to reach equilibrium magnetization. Also its shape for $T < T_B$ tells us how much interaction there is between grains. If the interaction is negligible, susceptibility will be approximately proportional to $1/T$ (Curie's law) as in Figure 2, while if the interaction is large, susceptibility will saturate at low temperatures.

#### 2.2.2 Size distribution

In many applications, for example in HDDs, one wants grains as uniform in size as possible. There are several methods to measure size distribution of a sample, for example observing it under a transmission electron microscope or measuring some macroscopic property (e.g. magnetization curve $M(H)$) of a sample and then deriving the parameters of size distribution based on physical models.

It turns out [2] that the log-normal distribution,

$$f(V) = \frac{1}{\sqrt{2\pi}\sigma V} \exp\left(-\frac{\ln^2(V/V_m)}{2\sigma^2}\right), \quad (2)$$

is often a good model for magnetic grains. $V_m$ is median grain volume and $\sigma$ is standard deviation of $\ln V$. Sometimes log-normal distribution is used as volume-weighted distribution [3] and sometimes as number-weighted distribution [6][7]. In the first case $f(V)\,dV$ tells proportion of entire magnetic volume taken by grains of volumes between $V$ and $V+dV$, while in the second case $f(V)\,dV$ tells the ratio of number of grains with volumes between $V$ and $V+dV$ and number of all grains in the sample.

The reason for log-normal size distribution is not completely understood yet [8].

#### 2.2.3 Magnetization curve $M(H)$

Magnetization curves $M(H)$ below and above $T_B$ are qualitatively different. Here I will concentrate on curve for $T > T_B$, because it can be used to calculate parameters of grain size distribution.

- $T > T_B$: In external magnetic field energy due to magnetic anisotropy is often much smaller than Zeeman energy, so energy of a single grain (1) can be approximated as $E = -\boldsymbol{\mu}\boldsymbol{B} + K\,V\,\sin^2(\phi-\theta) \approx -\boldsymbol{\mu}\boldsymbol{B}$. Lets first assume that all grains have magnetic moments of the same size. Then we can use the same theory as for Langevin paramagnetism. Because magnetic moments of grains are much larger than magnetic moments of individual atoms, we use classical limit: Brillouin function can be replaced by Langevin function and the average magnetization is given by [2]

$$M = M_0\, L(x) = M_0 \left[\coth x - \frac{1}{x}\right],$$

where $x = \frac{\mu B}{kT}$. We see that the bigger the particles, the faster magnetization approaches its saturation value ($M \propto x \propto \mu \propto V$), which is schematically depicted on Figure 3.

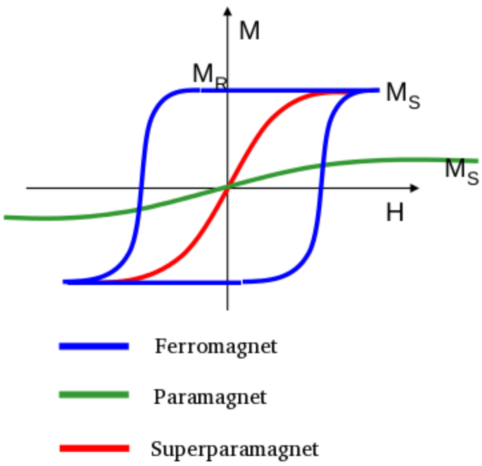


Figure 3: Schematic comparison of $M(H)$ curves of ferromagnetic, paramagnetic and superparamagnetic ($T > T_B$) materials. Source of figure: adapted from [19].

If $x \to 0$, we get Curie's law:

$$M = C\frac{B}{T}.$$

Note that above results are valid only when all particles have their magnetic moments $\boldsymbol{\mu}$ of the same size. In practice that is not the case, because particles differ in size. Magnetization of a sample with size distribution $f(V)$ is [2]

$$M = \int_0^\infty M(V) f(V)\, dV, \tag{3}$$

where $M(V)$ is magnetization of a monodisperse sample of particles of size $V$ and $f(V)\, dV$ is proportion of entire magnetic volume taken by grains of volume between $V$ and $V + dV$.

One can calculate $V_m$ and $\sigma$ parameters of log-normal distribution (2) by measuring $M(H)$ and solving Equation (3). More specifically, the log-normal distribution is put into Equation (3) which is solved in the limits $H \to 0$ and $H \to \infty$. Then $V_m$ and $\sigma$ can be expressed according to the shape of the $M(H)$ curve [3].

- $T < T_B$: Below the blocking temperature, the magnetization curves exhibit a hysteresis [2], because the timescale of the experiment is too short for the sample to reach thermal equilibrium value of magnetization

# 3 Applications

## 3.1 Ferrofluids

A ferrofluid is a colloidal liquid made of magnetic nanograins suspended in a carrier liquid. Particles are small enough that thermal fluctuations keep them evenly dispersed in the carrier liquid — there is no sedimentation. To prevent agglomeration grains have to be coated. In addition to Néel relaxation, there is a second mechanism that changes direction of grains' magnetic moments called Brownian relaxation — grains physically spin due to the collisions with surrounding particles. In the following paragraphs I briefly describe some practical applications of ferrofluids. [20][21]

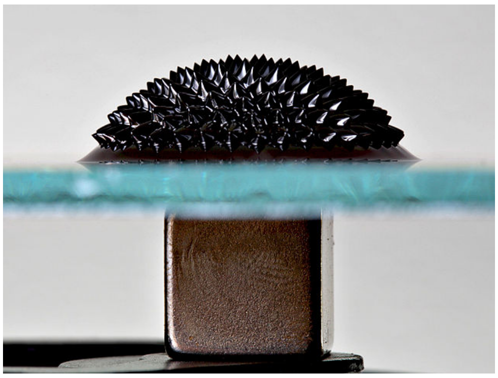

Figure 4: Ferrofluid above a permanent magnet. Source of figure: [20].

**Normal-field instability** This is actually a phenomenon, not an application, but it illustrates some physical properties that are important for practical applications. When ferrofluid is put in a strong vertical magnetic field, the surface forms spikes raised above the surrounding liquid (Figure 4). The effect is known as normal-field instability [20]. The observed shape minimizes the system's total energy, most important parts of which are the Zeeman energy of magnetic grains, the surface energy of the liquid and the gravitational energy of entire ferrofluid. Magnetic grains want to align their magnetic moments with field lines of external field and with fields of neighbouring grains, so they position themselves in lines along external magnetic field. If the field is strong enough these lines will raise above the initial surface of the liquid and pull the rest of the liquid with them (spikes). While the spikes lower magnetic energy, they increase surface and gravitational energy. The observed shape represents a configuration of least total energy.

#### 3.1.1 Liquid seals

Ferrofluids are used to hermetically seal space between rotary shaft (Figure 5) and surrounding pole pieces in rotary feedthroughs. They block debris from going from one side of rotary feedthrough to the other and can also sustain a big pressure difference between the sides. Ferrofluid is kept in place by magnetic field of permanent magnet, because grains position themselves along the field lines and pull the carrier liquid with them. The underlying physics is quite similar as in the case of normal-field instability — interplay of magnetic, surface and gravitational energy, with the addition of pressure gradient. Usually there are between 10 and 20 ferrofluidic rings (on top of "cuts" in shaft seen on Figure 5), each of them capable of sustaining a pressure of $2 \times 10^4$ Pa [22]. Because there is very little friction present when ferrofluidic seals are used, they have very little wear and thus long service life, they allow very fast rotation and they don't produce any dirt as a consequence of friction between solid parts. Ferrofluidic seals are widely used, among other things in HDDs. [20][22][23]

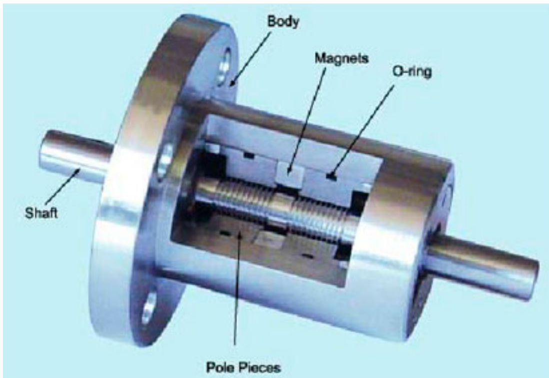


Figure 5: Rotary feedthrough that uses ferrofluid for sealing. Source of figure: [22].

### 3.1.2 Heat transfer

Susceptibility of a ferrofluid depends on temperature (as seen from ZFC and FC curves) — above $T_B$ the susceptibility falls approximately as $1/T$. As a consequence a magnet will attract cool ferrofluid more strongly than a hot one. This effect is used in a loudspeaker for cooling of voice coil. Voice coil is submersed in a ferrofluid. As it heats, so does the ferrofluid around it which creates a temperature and susceptibility gradient. Magnetically induced (thermomagnetic) convection appears.[20]

### 3.1.3 Damping

As already mentioned, if put in external magnetic field, magnetic grains position themselves along the magnetic field lines. These lines of grains hinder liquid flow perpendicular to the field and thus increase viscosity. The effect is called magnetorheological effect.[24]

## 3.2 HDD

### 3.2.1 Basic operation

Main parts of a HDD are shown on Figure 6. The data (bits) are stored on platters as a sequence of changes in the magnetization direction (Figure 7). Each platter is divided into concentric tracks along which the magnetization is read or written with read and write heads, that are moved around by the head arm. In this context reading means translating bits from magnetic representation (magnetization) into electric representation (voltage), while writing goes in reverse.

In the past a single read-write head was used for both reading and writing. The head was just a C-shaped high-permeability magnetic material with a wire wrapped around it (Figure 7). Reading and writing was based on electromagnetic induction:

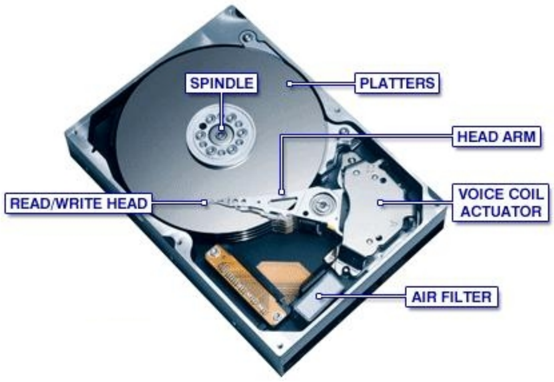


Figure 6: Main parts of a HDD. Source of figure: [25].

- Reading: When head moved over a piece of platter where magnetization changed, magnetic flux through the coil changed which resulted in voltage spike.
- Writing: Current through coil induced magnetic field that set magnetization of the part of the platter beneath the head.

Eventually single read-write head was separated into two heads, one specialized for reading and the other for writing. Today write heads are still based on induction, while read heads rely on the effect of tunnel magnetoresistance (TMR). Over the last 20 years read heads have used many different magnetoresistive phenomena (i.e. phenomena where electrical resistance of a material depends on magnetic field) — from anisotropic MR (AMR) to current-in-plane giant MR (CIP GMR) to the TMR [27]. Lately current-perpendicular-to-the-plane GMR head is being developed as the next step in the evolution of read heads.

### 3.2.2 Details

**Platter speed** The platters are usually spun at 5400 or 7200 rpm. Short calculation shows that the outer track of a 3.5 in disk (platter diameter is 3.74 in) spinning at 7200 rpm moves at

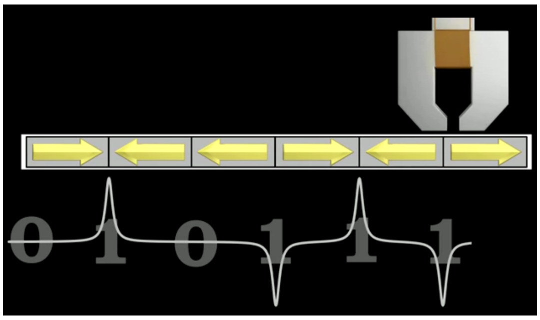

Figure 7: Change in magnetization direction means bit 1, no change means bit 0. This figure depicts an old recording technique where magnetizations were aligned along the tracks (longitudinal recording). Since about 2006 only the technique where magnetizations are perpendicular to the platter surface (perpendicular recording, see Figure 8) is used. Source of figure: [26].

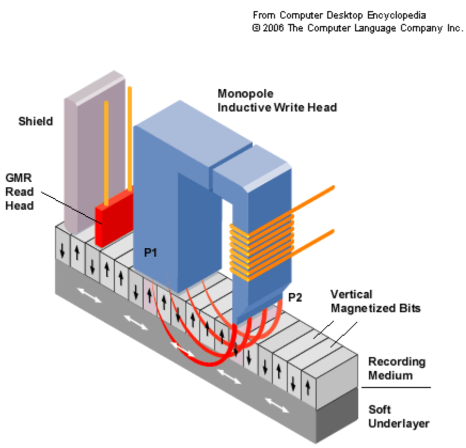


Figure 8: Read and write heads for perpendicular recording. Read head in picture still uses the older CIP GMR technology, currently TMR is used. Source of figure: [28].

the amazing velocity of 130 km/h. No wonder HDDs are not quite as quiet as SSDs. [10]

**Head clearance** Read head floats less than 10 nm above the platter surface [26]. It has to be that close so that it can measure or change just the magnetization of a given bit, not also its neighbours.

**Bit cell size** Bit cell size in 4 TB 3.5 inch drive is about $75 \times 13$nm [11][12]. The width is determined by the width of write head, while the length is determined by the distance the platter spins below the head between current switches.

**Transition noise** In a single bit cell there are a lot of magnetic grains. One of the reasons granular magnetic media is used instead of continuous is that granular media has shorter transition width between two magnetizations (Figure 9). In transition area of continuous media narrow stripes of opposite magnetization (Néel spikes) appear because that reduces magnetostatic energy. The reason for lower energy is that dipolar fields of different spikes cancel out the same way as fields of different domains in a non-magnetized bulk ferromagnet. Transition length in granular media is about the size of grain diameter which turns out to be less than in continuous media [14]. In order to increase areal density it is not enough to be able to magnetize a smaller piece of platter, it is necessary to also decrease the transition width, so that signal to noise ratio remains high enough [13]. Transition noise is decreased by decreasing the size of magnetic grains.

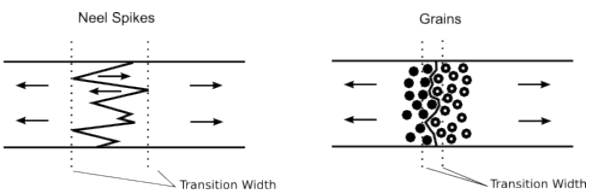


Figure 9: Granular media has shorter transition width than continuous media. Source of figure: [14].

**Bit stability** Magnetic grains must be able to hold magnetization that was given to them during writing for a very long time in order not to loose information. A typical value of energy barrier $\delta E = KV$ at room temperature is between $70\,kT$ and $100\,kT$ [13]. From first term in Equation (1) we can see that if energy barrier is to remain the same at decreasing grain volumes, anisotropy constant $K$ has to be increased. Engineers succeeded in producing grains with ever increasing anisotropy constants, but problems started to appear at the writing side. In order to switch magnetization direction of a grain, external magnetic field must remove the energy barrier and make current direction the maximum and desired direction the minimum of energy (1) (see Figure 1 to see effect of external magnetic field on energy). Field needed for this can be calculated by demanding a global maximum of energy $E$ at current magnetization direction, $\phi = 0$,

$$\frac{\partial E}{\partial \phi}\Big|_{\phi=0} = 0 \quad \text{and} \quad \frac{\partial^2 E}{\partial \phi^2}\Big|_{\phi=0} < 0.$$

One can roughly estimate the field needed to change the magnetization by equating Zeeman energy and energy barrier in absence of external field, $\mu_0 M_s V H = KV$. We get

$$H \approx \frac{K}{\mu_0 M_s}$$

As the anisotropy constant grows, so must the field. Recently it has become impossible to decrease the grain size because write heads can't produce magnetic fields strong enough to change the magnetization of these small and highly anisotropic grains. Some solutions and workarounds are described in Section 3.2.3.

**Grain size uniformity** It is important that magnetic grains are as uniform in size as possible. If size distribution is too wide then some grains will be too small to hold their magnetization due to thermal fluctuations, while others will be too big to sustain transitions between bit cells sharp enough [9].

### 3.2.3 Future

Apart from incremental improvements of all HDD components, four new technologies that change HDD in major ways are being developed and deployed. Two of them deal directly with the challenges related to grain stability and writability (*heat-assisted magnetic recording* and *bit patterned recording*) while the other two (*shingled magnetic recording* and *helium filled HDDs*) sidestep the issue.

**Helium filled HDD** Replacing air in the cavity of the HDD with helium decreases wind turbulence and friction between the platters and the surrounding gas [29]. The former enables putting platters closer together (and consequently more platters inside a HDD) without disturbing the read and write heads with turbulence too much, while the latter means lower power consumption and less heat production. A company called HGST increased number of platters from 5 to 7 to achieve capacity of 6 TB (current record for a 3.5 inch drive) without increasing the areal density. HGST started shipping first commercially available helium filled HDDs at the end of 2013 [30].

**Shingled magnetic recording** Here areal density is increased by partially overwriting old tracks with new ones in a similar way that shingles are put one over another on the roof (see Figure 10) [17]. Because read head in a HDD is smaller than the write head, it doesn't need the entire width of a track to read information stored by write head. Therefore shingled recording doesn't reduce the read quality. It does make updating the information on the HDD much harder though, because it is no

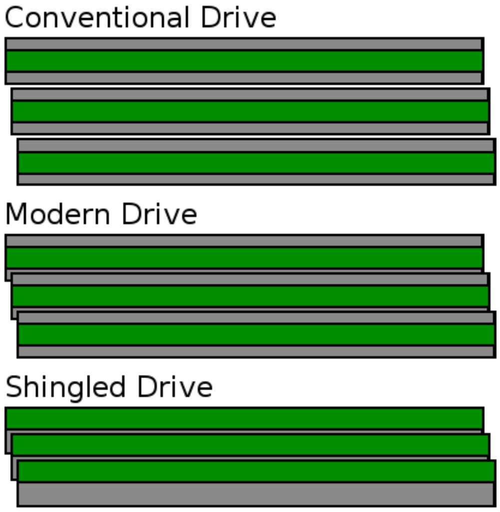


Figure 10: Tracks written by write head are gray, while parts of tracks needed by read head are green. Source of figure: [16].

longer possible to change just one single track. Write head is so wide that it always overwrites several trimmed tracks. When a trimmed track is overwritten with new information, all shingled tracks above it have to be rewritten also in order not to loose their existing information. To alleviate this problem tracks are combined in bands. Tracks from different bands don't overlap which limits the number of tracks that might need to be rewritten. A big weakness of SMR is longer write times, while its strength is that it offers big increase in areal density (25 % with first generation products) without the need for drastic changes in read and write heads or platter design. Looks like that first commercially available disks using SMR started shipping in the beginning of 2014 [15].

**Heat-assisted magnetic recording (HAMR)** Just before writing, grains get heated with a laser beam which both decreases their magnetic anisotropy and also effectively lowers energy barrier in units of $kT$ [31]. Seagate reached areal density of $1\,\mathrm{Tb/in^2}$ using HAMR in the beginning of 2012 [5], which is a 30% increase over what is possible with conventional recording. Currently there are no commercially available HDDs using HAMR.

**Bit-patterned recording (BPR)** In conventional recording media, bit cells touch each other and each of them is made of many small grains. Grains have to be small in order to reduce the transition noise and consequently increase signal to noise ratio. [32]

In bit patterned recording bit cells are lithographically patterned in ordered arrays with non-magnetic material between them. Because bit cells are separated there is no need to fill them with small grains that would make transition from one cell to the other as sharp as possible. Instead each bit cell is a single piece of magnet. These pieces are larger than grains in conventional HDDs and therefore thermally more stable, but they are still much smaller than bit cells in conventional HDDs which are build of many grains. In 2013 company HGST made prototypes with magnetic islands 10 nm in width [33].

The problem with BPR is that it is difficult to make ordered arrays of magnetic cells of such a small size. Cells must be positioned along the tracks very precisely or else problems with reading and writing appear. Currently there are no commercially available HDDs using BPR.

## 4 Conclusion

Superparamagnetic materials have unique properties because of the sizes of their magnetic building blocks. In some applications these properties are desired (e.g. in ferrofluids) and in others one tries to avoid them (e.g. in HDDs). In any case, there is a lot of research being done with the goal of optimizing superparamagnetic materials for the particular applications at hand.

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