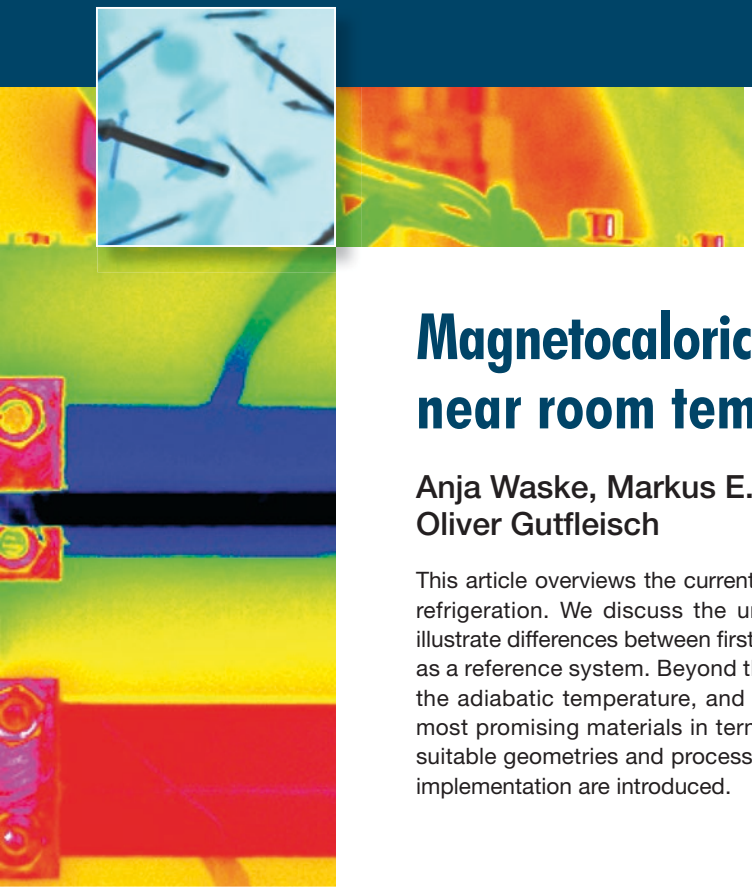


Magnetocaloric materials for refrigeration near room temperature

Anja Waske, Markus E. Gruner, Tino Gottschall, and Oliver Gutfleisch

This article overviews the current status of magnetocaloric materials for room-temperature refrigeration. We discuss the underlying mechanism of the magnetocaloric effect and illustrate differences between first- and second-order type materials starting with gadolinium as a reference system. Beyond the key functional properties of magnetocaloric materials, the adiabatic temperature, and entropy change, we briefly address the criticality of the most promising materials in terms of their supply risk. Looking at practical applications, suitable geometries and processing routes for magnetocaloric heat exchangers for device implementation are introduced.

Introduction

The magnetocaloric effect (MCE) is the reversible temperature change of a magnetic material upon application and removal of a magnetic field, as illustrated in **Figure 1**. The phenomenon was discovered in 1917 in nickel by Weiss and Picard.[1] Its potential for room-temperature refrigeration utilizing a regenerative cycle[2] was demonstrated with elemental gadolinium in 1976. Today, industrial prototypes for different cooling applications exist,[3–5] some of them utilizing materials with a "giant" MCE. In these materials, a first-order magnetic phase transition occurs jointly with a change in the structure, which was first observed in $Gd_5Si_2Ge_2$.[6] This finding triggered research that soon led to the discovery of other giant magnetocaloric materials, such as La-Fe-Si-based[7] and Mn-Fe-P-based alloys,[8] and NiMn-based Heusler alloys.[9]

Contributions of magnetism and structure to the MCE

Magnetocaloric materials release heat when an externally applied magnetic field induces a transition from a disordered to an ordered phase. This is expressed by an (isothermal) entropy change ΔS_T. In an adiabatic process, a corresponding temperature change, ΔT_{ad}, is observed. In a first approximation, the total entropy change, ΔS_T, may be decomposed into contributions arising from the relevant degrees of freedom—occupation of electronic bands, magnetic order, and lattice vibrations.[10] Apart from their high sensitivity to the magnetic field at the first-order transition, giant magnetocaloric materials may also benefit from the concomitant changes in the entropy contributions not directly related to magnetism.

The electronic entropy, S_{el}, usually provides the smallest contribution, and its change, ΔS_{el}, is also small in absolute number. S_{el} is essentially proportional to the electronic density of states (DOS) at the Fermi energy, E_F. The highest occupied electronic energy levels at E_F are subject to thermal disorder. S_{el} can be determined by low-temperature calorimetry or photoemission spectroscopy, or alternatively, from first-principles calculations in the framework of density functional theory (DFT).[11,12] In magnetic materials, the electronic bands cease to be spin-degenerate, which leads to a spin-dependent occupation of the electronic orbitals and thus the appearance of magnetic moments. The sizes and orientations of the moments may differ from one atomic site to another and form ordered or disordered magnetic configurations in the crystal lattice. These determine the magnetic contribution to the entropy change.

In giant magnetocaloric materials, the coupling between magnetism and structure usually inhibits the direct determination of ΔS_{mag} from calorimetry, which yields ΔS_T instead.[13] However, ΔS_{mag} can be assessed indirectly by subtracting the electronic and lattice contributions from ΔS_T, or from theory,

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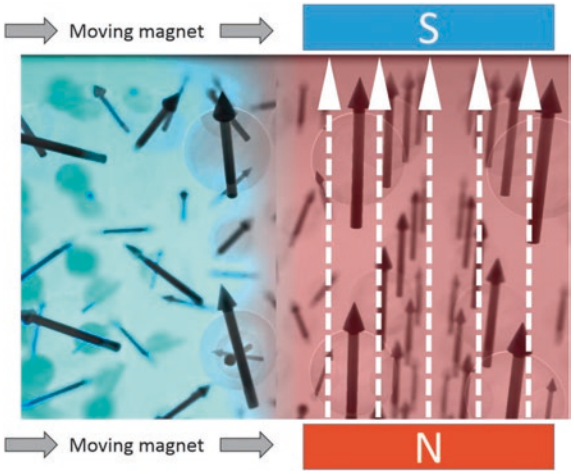


Figure 1. Illustration of magnetocaloric effect. When the magnet is removed from the (magnetized) magnetocaloric material adiabatically, the material cools down.

for instance, by employing a statistical model description of magnetism such as the Heisenberg model with parameters obtained from DFT calculations.[14] The third contribution—the change in lattice entropy ΔS_{lat}—can be large for magnetostructural phase transitions. It depends on the change in atomic volume as well as on the change in the elastic properties at the phase transition. ΔS_{lat} can be derived from the vibrational density of states (VDOS), which can be measured by inelastic neutron scattering or nuclear resonant inelastic x-ray scattering, and has also been calculated from first-principles.[15–18]

For $La(Fe_{1-x}Si_x)_{13}$, the three contributions to ΔS_T have the same sign,[17] whereas for NiMn-based Heusler alloys, the contributions from lattice and magnetism often compete.[19] However, the three contributions are not entirely independent. The itinerant electron metamagnet $La(Fe_{1-x}Si_x)_{13}$ is characterized by a competition of electronic states of Fe with different magnetic moments. Due to this, magnetic disorder leads to an increased DOS at E_F, which improves the electronic screening of atomic displacements.[20] This results in a softer lattice[17] and a favorable ΔS_{lat}. The itinerant magnetism of Fe is also at the heart of the caloric properties of Fe_2P-type materials, such as $Mn_xFe_{1.95-x}P_{1-y}Si_y$.[21]

In the following, we compare adiabatic temperature and entropy changes of different first-order materials with elemental polycrystalline gadolinium as a reference material.[22,23] Gadolinium undergoes a second-order magnetic ordering transition at a Curie temperature (T_C = 294 K) close to room temperature. Hence, we will compare it to first-order materials with similar magnetic transition temperatures (T_t = 269–297 K).

Finding a substitute material for gadolinium

The MCE in response to a magnetic field of certain strength is characterized by two quantities, the adiabatic temperature change ΔT_{ad} and the entropy change ΔS_T. No commercially available equipment exists currently for measuring the adiabatic temperature change, and it is usually measured using purpose-built devices.[24] Furthermore, a general problem in determining ΔT_{ad} in giant magnetocaloric materials is potential mechanical failure and sample history dependence during the measurements, leading to lower repeatability of values observed after the first cycle.[25,26] Here, we will present values that have been measured with the same device and under field cycling.

Figure 2 shows the properties of the most important magnetocaloric materials along with gadolinium as the benchmark for a field change of about 2 T. Gadolinium yields a large cyclic adiabatic temperature change of 4.7 K.[23] $La(Fe, Mn, Si)_{13}H_x$ with T_t = 297 delivers 4.3 K,[27] while $Ni_{45.7}Mn_{36.6}In_{13.5}Co_{4.2}$ reaches 3.0 K and is to date the Heusler alloy[28] with the largest cyclic ΔT_{ad}. For Mn-Fe-P-based materials, with T_t close to room temperature, ΔT_{ad} values around 5 K have been reported from indirect measurements.[29] However, for a fair comparison, we use the cyclically obtained value of 1.9 K of a direct measurement[30] for a stoichiometry with T_t = 269 K.

The entropy change ΔS_T of the transition is proportional to the amount of heat that can be transferred by the material. ΔS_T is determined indirectly by magnetometry using adequate measurement protocols and calculations using the Maxwell relations.[31] Using the same stoichiometries and literature references as for ΔT_{ad}, entropy change values between 5.6 J kg^{-1} K^{-1} (gadolinium) and 17 J kg^{-1} K^{-1} (Mn-Fe-P-based) are reached. For first-order magnetocaloric materials, the existence of thermal hysteresis of different intrinsic and extrinsic origin[32,33] is known to have a detrimental effect on the available cooling power. For second-order gadolinium, no hysteresis exists,

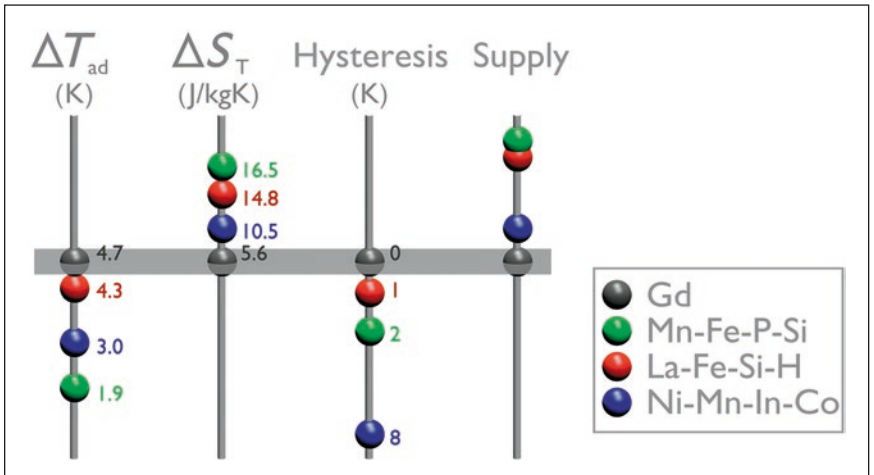


Figure 2. Comparison of the magnetocaloric properties of the three most relevant first-order-type magnetocaloric materials along with those of benchmark gadolinium. The values are given for the chemical compositions in the legend, with transition temperatures close to room temperature.

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while for Heusler alloys with an austenite to martensite transition, thermal hysteresis on the order of 8 K is observed.[28] For Mn-Fe-P- and La-Fe-Si-based alloys, only a moderate hysteresis of 1–2 K is found.[34,35]

In addition to the physical properties of materials for magnetic refrigeration, it is crucial for this new technology to be based on abundant raw materials in order to decrease cost and be competitive on the refrigeration market. We discuss elements relevant for magnetocaloric cooling that are critical in terms of their demand-supply balance, which has a direct impact on short-term prices and long-term price stability of an emerging technology. Resource criticality is determined by several factors such as geological availability, environmental footprint, substitution and recycling potential, to name a few.[36,37] The most promising materials are based on La-Fe-Si and Mn-Fe-P, while Gd serves as the best reference material to date and finds efficient utilization in current prototypes. Additionally, Heusler compounds have emerged as a likely contender for magnetocaloric applications.

Finally, one should not forget the Nd-Fe-B permanent magnets when assessing the raw materials basis for MCE, which efficiently provide the strong external magnetic field necessary to drive the cooling cycle, and which for the time being have no alternatives. The global production of Gd is unlikely to be able to meet the demand forecasted for potential mass-market application in the mid- and long term.[38] Lanthanum is not critical in the context of the rare-earth balance,[39] and the same holds for Mn, Fe, and P. Nd in Nd-Fe-B magnets could be a bottleneck for magnetic cooling innovations as Nd price volatility and uncertainty in supply might be conceived as a risk for larger mass-market penetration of magnetic refrigeration technology. This risk, however, could be circumvented by, for example, a concerted approach securing a rare-earth supply chain outside China,[38] which is—with about 90–95% of global rare-earth oxide production—the main source for rare earths currently.

From materials to devices: The art of making a regenerator

We have seen that the adiabatic temperature change is actually small, generally less than 5 K in moderate magnetic fields of 2 T and below in magnetocaloric materials. For practical applications, it is necessary to increase the temperature difference between the hot and cold reservoirs by using active magnetic regeneration.[40] For this, the porous magnetocaloric regenerator is periodically cycled in and out of the magnetic field, while at the same time appropriate fluid flow through the regenerator collects the synchronously released heat and cold. Active magnetic regeneration can be further improved by using a combination of materials with different operation temperatures.[41] For gadolinium, this is achieved using binary Gd-based alloys[42] to shift T_C, such as $Gd_{1-x}R_x$ (R = Y, Dy, Tb). For first-order magnetocaloric materials, the transition temperature T_t can be adjusted[43] by doping or hydrogenation. With this set of materials, a porous body (regenerator) has to be formed, which allows the water reciprocating for heat transfer to be exposed to and removed from the magnetic field. This places stringent requirements on the design of the regenerator, such as maximizing the fraction of volume occupied by the magnetocaloric material, while simultaneously yielding a low pressure drop for the water flow. Due to the limited machinability of first-order magnetocaloric materials, which tend to be brittle, there are three types of geometry that are mainly used:[10,44] packed bed, parallel plate arrays—either of fully dense or composite material—and microchannel structures,[45,46] as shown in **Figure 3**.

Packed beds are composed of either irregularly shaped powder or spherical particles made of magnetocaloric materials. While the former can be produced by milling, the latter requires more elaborate techniques such as gas-atomization,[47] extrusion spheronization (which involves breaking or cutting an extruded powder mixture and then rounding it into spheres),[48] the rotating electrode process,[49] or drop-tube solidification.[50] Typical sizes for both particle types used in packed-bed regenerators[42] are in the range of d = 100–500 μm. Mechanical failure leading to cracking and subsequent washout of particle fragments can be counteracted by covering the particles with a thin layer of polymer, which holds the particles in position even after fragmentation.[51] Typical void fractions of packed-bed regenerators are 35–42 vol% for spheres, and 45–55 vol%

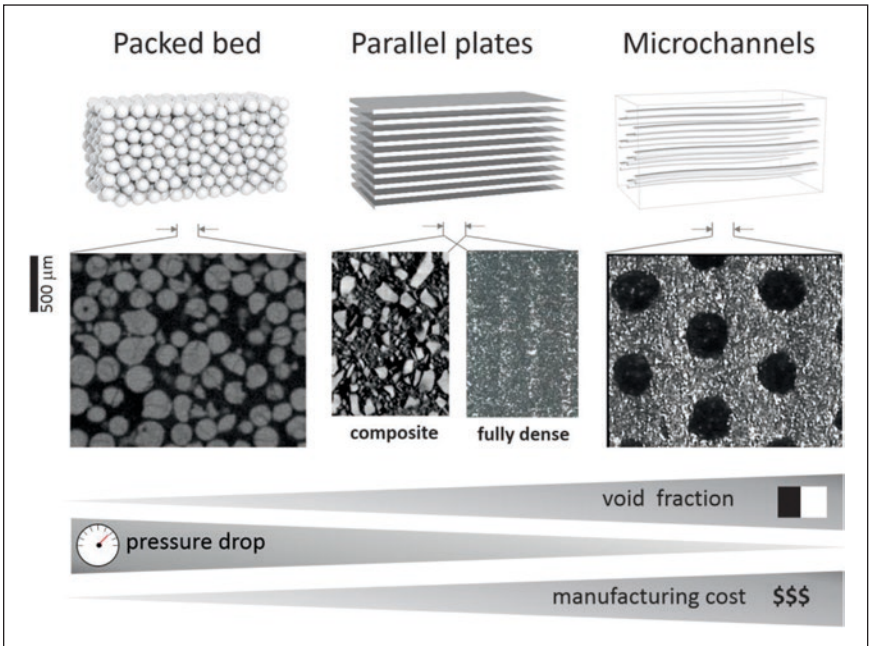


Figure 3. Typical magnetocaloric regenerator structures (top), their microstructures (middle), and important properties (bottom). A compromise is necessary between a low void fraction, which maximizes the amount of heat/cold supplied by the material, low pressure drop, which maximizes the flow of heat-transfer liquid, and manufacturing cost.

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for irregular particles, which together with the turbulent flow in packed beds leads to a large pressure drop.[42]

Parallel plate arrays are very well-defined geometries for regenerators. Typical plate thicknesses are around d_{plate} = 200–500 μm, with gap distances between the plates[42] in the range of d_{dist} = 200 μm, leading to void fractions[44] on the order of 0.36–0.64. The lower limit for the plate thickness is again set by the mechanical stability of the material, particularly for fully dense plates. One way out of this constraint is to form magnetocaloric composites of the active phase and a matrix of polymer,[52] amorphous,[53] or low melting temperature eutectic alloys.[27]

Microchannel geometries along the fluid flow direction with different cross-sectional shapes (circular, square) are another option to form a regenerator, offering a wide range of possible void fractions. Instead of drilling the channels, which wastes material, more sophisticated methods to produce this type of geometry are applied, such as three-dimensional selective laser melting[45] and extrusion.[46] Both methods require a heat-treatment procedure after shaping, either to retain the active magnetocaloric phase or to sinter the green body to yield a fully dense structure, respectively. In both cases, the mechanical properties of the finished regenerator are far from ideal. Economic considerations and easy up-scalability are likely to play an important role for a potential mass production of magnetocaloric regenerators.

Conclusion

Competitive magnetocaloric materials must score high in a large number of properties in order for this new technology to become commercially viable. One of the key challenges from a materials research standpoint is shaping the mostly brittle magnetocaloric materials into porous regenerators that allow for fast heat exchange. Solving this problem is likely to accelerate not only the field of magnetocaloric refrigeration, but also other applications of magnetocaloric materials, like thermomagnetic motors and power generation.

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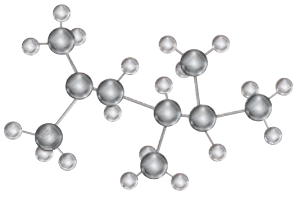
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