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Evaluation of the effective temperature change in Gd-based composite wires assessed by static and pulsed-field magnetic measurements

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ABSTRACT

Gd cladded in a seamless 316L austenitic steel tube has been swaged into wires by the powder-in-tube (PIT) technology, resulting in an outer diameter of 1 mm, a wall thickness of approx. 100 μm and a filling factor of around 62 vol%. Such wires provide an advantageous geometry for heat exchangers and have the benefit to protect the Gadolinium, i.e. from corrosion when being in contact with a heat transfer fluid. The magnetocaloric composite has been studied by static and pulsed magnetic-field measurements in order to evaluate the performance of Gd as a core material. By the analysis of magnetization and heat capacity data, the influences of deformation-induced defects on Gadolinium are presented. The subsequent heat treatment at 773 K for 1 h in Ar atmosphere allowed restoring the magnetic properties of the wire after deformation.

Data of the pulsed magnetic-field measurements on the Gd-filled PIT-wires and a Gd–core separated from the jacket are presented, with an achievable temperature change of 1.2 K for the wire and 5.2 K for the Gd in 2 T, respectively. A comparison to previously studied La(Fe, Co, Si)$_{13}$-filled composite wires is included. It indicates that performance losses due to the passive matrix material cannot be overcome only by an increased adiabatic temperature change of the core material, but instead the wire components need to be chosen regarding an optimized heat capacity ratio, as well.

1. Introduction

Given the challenge of ever-increasing energy demand in the refrigeration and air-conditioning sector, the market needs for innovative cooling technologies are rising [1,2]. So far, the cooling market for refrigerators and climate systems is dominated by conventional vapor compression [3], and magnetocaloric cooling is seen as one of a few promising substitutes to this technology [3,4]. Magnetocaloric cooling, or magnetic refrigeration, is a solid-state refrigeration technology based on the magnetocaloric effect that occurs near the magnetic phase transition of a material. To enhance the efficiency of magnetic refrigeration systems, significant progress is necessary in materials development, shaping and systems engineering [3,5–9]. In addition, requirements for fluid flow structure, surface quality and ease of series production must be fulfilled, just as functionally and mechanically stable cyclic operation in an active magnetic regenerator [8]. Shaping of magnetocaloric materials and the optimization of flow-channel design in solid regenerators have been the subject of extensive research [10–17]. One difficulty is the trade-off between effective heat exchange and a low pressure drop over the working length.

First published in 2018 [18], composite wires containing a magnetocaloric La(Fe, Co, Si)$_{13}$-core cladded by an austenitic steel can be fabricated by utilizing the powder–in–tube (PIT) process. These wires can be combined to a pin-like heat-exchanger geometry with optimized hydraulic and thermal exchange performance. Additionally, the passive austenitic steel jacket ensures that the active magnetocaloric material does not corrode or disintegrate during magnetic cycling. The La(Fe, Co, Si)$_{13}$-filled composite wires in [18] had a diameter of 1 mm, with a resulting steel jacket thickness of approx. 0.1 mm and a filling factor as large as 58 vol%. To evaluate the performance of these wires, pulsed-field measurements were performed and are presented in [19]. The findings of these experiments led to the assumption that a magnetocaloric material with a larger adiabatic temperature change ΔT_{ad} should be established to compensate losses due to the passive steel surrounding.

In this work, we present a magnetocaloric composite PIT-wire with Gd as core material cladded by the identical austenitic 316L steel. Gd is

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often used as benchmark for magnetocaloric materials, therefore, these Gd-filled wires can also serve as a benchmark for composites prepared by the PIT-process. More importantly, Gadolinium provides a temperature change of 4.7 K in a magnetic field change of 2 T, which is the larger ΔT_{ad} (compared to the used La(Fe, Co, Si)$_{13}$ in [18]) aimed for in [19] to compensate the losses by the steel jacket of the composite [20]. First, the influence of the mechanical deformation on the magnetocaloric properties of the composite wire during the PIT-process is discussed. Secondly, measurements of the adiabatic temperature change of both, the Gd-core up to 10 T and the resulting effective temperature change ΔT_{eff} at the steel jacket for magnetic-field pulses of 2 T, in a comparison to La(Fe, Co, Si)$_{13}$-filled composite wires, are presented. With these results, new demands for magnetocaloric PIT-wires are indicated.

2. Materials and methods

A Gd bulk rod (2.4 mm diameter, 99.9% purity) as magnetocaloric core in a seamless austenitic (316L) steel tube with a starting diameter of 3 mm was stepwise swaged down to a diameter of 1 mm, similar to the PIT-process presented in [18]. In order to investigate the degree of filling of the composite wire, X-ray computed tomography (XCT) was performed with a GE Phoenix Nanotom M device. 1000 absorption images with an exposure time of t = 1000 ms where captured while scanning the sample with an acceleration voltage U = 125 kV and current I = 100 μA during a 360° rotation. The scan had a voxel size of 2 μm as resolution. Phoenix datosx 2.2 software was used for volume reconstruction and VG-studio max 2.2 image analysis software for the identification of the volume fractions of the inner magnetocaloric core and outer steel jacket.

After deformation, a heat treatment of the wire was performed in Argon-atmosphere at 773 K for 1 h, followed by water quenching at room temperature, according to [21,22]. To prevent oxidation, Ta-foil was wrapped around the composite wire.

The magnetic properties in static magnetic-fields were measured using a Physical Property Measurement System (PPMS) from Quantum Design. The magnetization was measured using a Vibrating Sample Magnetometer (VSM). Magnetic-field was applied along the longest axis to avoid demagnetization effects. For temperature depended magnetization curves M(T), samples were measured from 375 K down to 200 K in a magnetic-field of 1 T. From isothermal magnetization curves M(H) in a temperature range of 250…325 K and fields from 0 to 2 T, the entropy change Δs_T was calculated using the thermodynamic Maxwell relation:

$$\Delta s_T = \mu_0 \int_0^H \left(\frac{\partial M}{dT}\right)_H dH.$$

For the heat capacity (c_p) measurements of the deformed and heat-treated Gd-core, the material was separated by grinding off the surrounding steel jacket with abrasive SiC-paper. The heat capacity was determined by relaxation method [23] in fields of 0, 1, 2, 5 and 9 T. Using this data, the isothermal entropy changes Δs_T and the adiabatic temperature change ΔT_{ad} as a function of the magnetic-field and temperature were obtained by 3D interpolation. To set a reference for the composite wire, a piece of the as-cast Gd-bulk has been measured regarding of M(T) and Δs_T.

Furthermore, pulsed-field measurements were performed at the Helmholtz–Zentrum Dresden-Rossendorf (HZDR-EMFL) [24]. Two samples, i.e. a complete composite wire and a separated core, have been prepared. For the core sample, the steel jacket was removed by grinding it off, using abrasive SiC-paper, similar to the heat capacity samples. Type T thermocouples consisting of constantan and copper were used to measure the temperature difference between those samples and a PT100 reference [25,26]. Positioning and preparation of the experimental setup was taken from [19]. The core sample, serving as reference to the composite wires, has been equipped with one thermocouple in between two pieces of it. The temperature change of the wire was monitored by a thermocouple at the steel jacket. The positioning of these thermocouples was checked by X-ray computed tomography. The samples were aligned along their longest axis, minimizing demagnetization effects. Magnetic-field pulses of 2, 5 and 10 T were applied. Temperatures around the magnetic transition temperature (T_t) in the range between 261 and 321 K, using 5 K steps around T_t and 10 K steps further away, were measured. Two pulses - one in positive and one in negative field direction - were applied for each temperature and field step in order to average interfering signals.

3. Results and discussion

3.1. Magnetic characterization of Gd-filled wires in static magnetic-field

The Gd-filled steel tube was successfully swaged to a diameter of 1 mm. By X-ray computed tomography a filling factor of around 62 vol% and wall thicknesses of approx. 100 μm were determined, as can be seen in Fig. 1.

Therefore, the Gd-filled composite wires have comparable shape and aspects to the La(Fe, Co, Si)$_{13}$-filled composite wires of [18,19]. The diameter change from 3 mm to 1 mm directs to a degree of deformation ($\varphi_A = \ln(A_i/A_f)$, where A_i and A_f are the initial and final cross-sectional areas, respectively) of φ_A = -2.19. Mechanical deformation, as induced during the rotary swaging, can lead to microscopic defects and dislocations, which affect the magnetic properties in Gd [21]. Fig. 2 shows the temperature dependence of the magnetization for the as–cast Gd-bulk reference, a deformed composite wire and a deformed composite wire that has undergone heat treatment (annealed).

The inset of Fig. 2 displays the temperature derivate of the magnetization to determine the transition temperature (T_t) of the samples from their local minimum, resulting in values of 293 K for the as-cast, 291 K for the deformed and 294 K for the annealed wire. Compared to the bulk Gd reference, the magnetization of the deformed composite wire is reduced by nearly one half and T_t is lowered by 2 K. A similar behavior of a deformation–induced reduction in the magnetization and transition temperature caused by a random magnetic anisotropy has already been reported for plastically deformed Gd [21,27]. This anisotropy is the consequence of lattice disorders that occur due to the impact of the mechanical deformation. It has been proposed that such random magnetic anisotropy tends to play a decisive part in determining magnetic properties of deformed Gd samples [28–30]. A decrease of T_t, by 4 K, has also been reported in [21] for severe plastic deformed Gadolinium, stating that induced defects bring about internal stresses and thus exchange interactions are weakened.

A magnetic impact of the steel as in [18], where a low magnetization occurred, cannot be observed. This might be due to the lower degree of deformation φ_A which was applied here ($\varphi_A = -2.19$) when compared to the PIT-processing of La(Fe, Co, Si)$_{13}$ ($\varphi_A = -3.58$) [18].

To reduce the impact of mechanical defects on the magnetic properties of the Gadolinium, the composite wire has been annealed. As can be seen in Fig. 2, the annealed sample indeed displays an increased magnetization and increased T_t compared to the deformed wire. Still, the values are a bit more than 1/3 lower in comparison to the as-cast state. This can be attributed to the non-magnetic steel jacket surrounding the Gd-core, since the displayed values of magnetization are mass related. Scaling the magnetization change in the annealed composite wire by multiplying with the inverse mass fraction of the core to 100 wt% Gd and therefore, 100% magnetocaloric phase (since the steel jacket does not contribute to the magnetic properties) the curve overlaps with the as-cast state as shown in Fig. 2 by the dotted line.

The effect of the heat treatment on the deformed Gd-filled wire is presented in Fig. 3, displaying heat capacity measurements for separated cores of the deformed and the annealed state.

Both samples have been measured in 0, 1, 2, 5 and 9 T within the same temperature range and the comparison shows the following

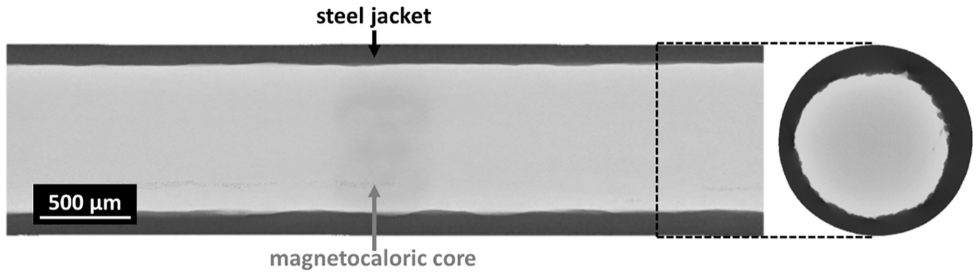

Fig. 1. The cross section of the swaged Gd-filled PIT-wire in lengthwise (left) and transverse (right) view, determined by XCT.

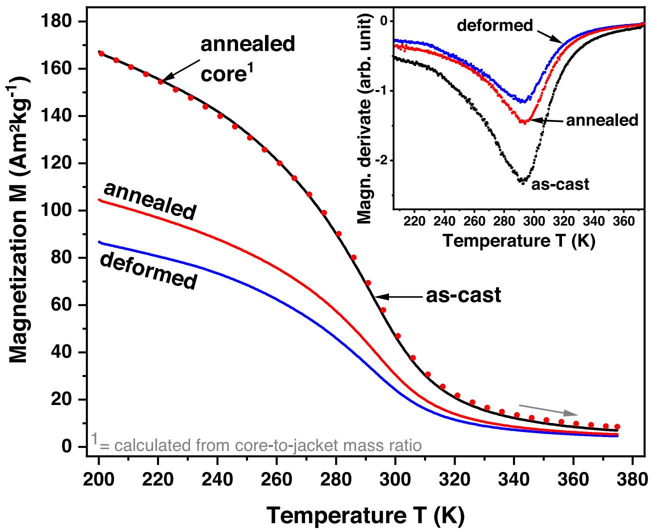

Fig. 2. Temperature dependencies of magnetization of the deformed wires and a Gd bulk reference in 1 T, Inset: Temperature derivate of the magnetization.

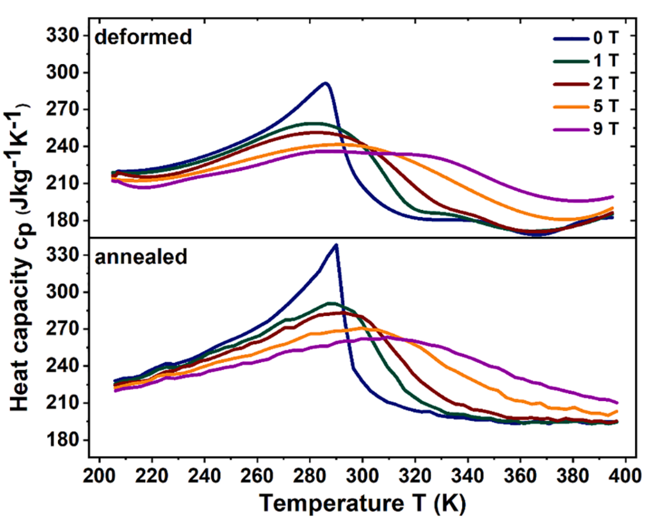

Fig. 3. Comparison of the specific heat of the deformed and the annealed separated Gd-cores in 0, 1, 2, 5 and 9 T.

features: A well-pronounced λ-type anomaly around the second order magnetic phase transition can be seen for the deformed, as well as the annealed core in the zero field curves. The peak for the annealed core is increased by roughly 15% and in the deformed core one can see a weaker suppression of the λ-type anomaly by the external magnetic field, as well as a smearing of the curves over the whole magnetic field range. This is due to the fact that the magnetic phase transition in the deformed state is broadened by the presence of dislocations and strains [31]. Furthermore, it was reported that deformed Gadolinium shows a phase separation, observed in a shift of the transition temperature seen in the heat capacity measurements [32]. While a decrease in the heat capacity after the deformation might be beneficial for the magnetocaloric effect, smearing of the magnetic transition weakens it, as discussed in [21]. As mentioned above, defects induced by the deformation process can be reduced by a heat treatment, resulting in an increased magnetization. After annealing, the broadening of the c_p-curves is less pronounced. With the increased magnetization, the magnetic contribution to the total heat capacity is increased, resulting in higher c_p-values for the annealed core. The temperature of the heat capacity maxima approximately describes T_t in zero-field [33]. This leads to 291 K for the deformed Gd-core and 293 K for the annealed Gd-core. These results are in good agreement with the values calculated by the temperature derivate of magnetization in Fig. 2. Generally, the specific heat of the annealed Gd-core agrees well with that reported earlier [31,33].

Fig. 4 shows the comparison of Δs_T(T)-curves for the as-cast Gd-bulk reference, the deformed wire and the annealed wire. Additionally, curves of entropy change scaled up to 100 wt% Gd, by multiplying with the inverse mass fraction of the cores, are shown.

The Gd-bulk reference shows a maximum entropy change of 4.7 $Jkg^{-1}K^{-1}$ at 293 K and is, therefore, in good agreement with literature [20]. After deformation, the entropy change in the composite wire is reduced to roughly 2.2 $Jkg^{-1}K^{-1}$ at 290 K, again displaying the trend of shifting T_t to lower temperatures, as seen in Fig. 2. The reduced Δs_T can be attributed to the reduced magnetization by the induced defects. In the annealed wire the magnetization of the Gd-core was restored, hence, the entropy change should be increased as well. The annealed wire displays

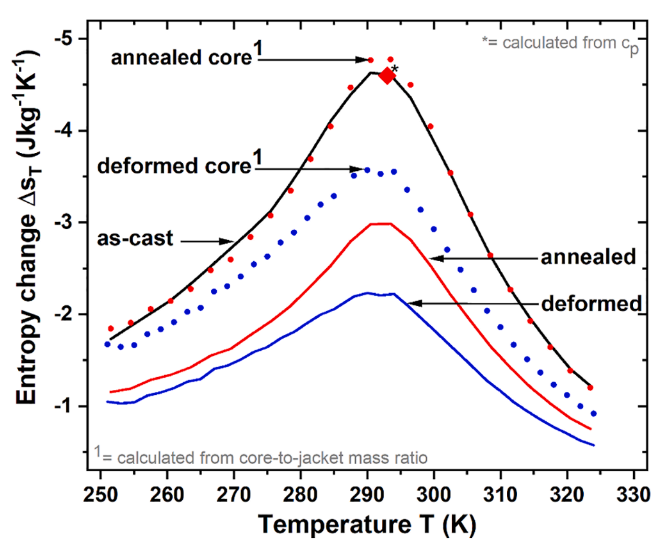

Fig. 4. Comparison of the entropy change of the Gd-bulk, the deformed wire and the annealed wire measured for 2 T, with addition of entropy change value for the annealed core calculated from c_p-measurements.

an increase of about 20% compared to the deformed wire, but with a maximum entropy change of 3.0 $Jkg^{-1}K^{-1}$, not the level of the as-cast reference. This reduction in Δs_T compared to the as-cast reference by a little more than 30% is reasonable when considering the core-to-jacket mass ratio. Hence, the reduction can be attributed to the passive steel jacket. The entropy values of the magnetocaloric Gd-cores, again scaled to 100 wt% for better comparison, are shown by dashed lines. An overlap of the scaled curve of the annealed wire with the reference can be seen, which confirms the restoration of the initial magnetic properties after annealing. The entropy change calculated from heat capacity measurements, shown as data point at 293 K, agrees to the values of the entropy change calculated from magnetization curves.

With regards to the static magnetic-field measurements, it can be stated that a heat treatment after the deformation is needed for Gd-filled composite wires. With the annealing at 773 K for 1 h in Ar, a sufficient way to restore the magnetic properties of the Gd-core to an as-cast level is presented. Still, the composite wires will display a reduced magnetocaloric performance due to the passive jacket material. To further evaluate the Gd-filled PIT-wires, especially in terms of achievable temperature change, pulsed magnetic-field measurements at HZDR-EMFL are presented in the next section.

3.2. Temperature change of Gd-filled wires in pulsed magnetic-fields

Fig. 5 shows the temperature and magnetic field dependent adiabatic temperature change ΔT_{ad} of the Gd-core that was separated from the annealed composite wire. Magnetic-field pulses of 2, 5 and 10 T have been applied around the transition temperature of Gd.

As expected, one can see the maxima of the adiabatic temperature change near the presence of the magnetic transition temperature at the 291 K measurement step. For the 2 T pulse a maximum ΔT_{ad} of 5.2 K, for the 5 T pulse a maximum of 9.7 K and for the 10 T pulse a maximum of 15.5 K are observed. These results for the annealed Gd-core are in good agreement with Gd samples measured earlier [20,26,34], i.e. this indicates that the annealing at 773 K successfully restored the magnetocaloric effect. For comparison, calculated ΔT_{ad} values from heat capacity measurements in static fields are pointed in Fig. 5. Showing 5.4 K and 8.7 K at 2 T and 5 T respectively, the values correspond well to the values directly measured in the pulsed-field.

These results for the Gd-core separated from the steel jacket can now serve as a reference to the field-induced temperature change observed in the Gd-filled composite wire, to investigate the influence of the surrounding steel jacket. Due to the thermal exchange between core and steel jacket, the field-induced temperature change of the core is no longer adiabatic. The steel jacket itself acts as passive heat sink, hence, ΔT rather than ΔT_{ad} will be used for core-related and ΔT_{eff} for jacket-related matters in the following discussion.

In Fig. 6 the comparison between Gd-filled and La(Fe, Co, $Si)_{13}$–filled PIT-wires is illustrated for fields of 2 T.

Fig. 6a displays the temperature change of the Gd-core and the resulting ΔT_{eff} at the steel jacket of a Gd-filled wire in a 2 T field pulse (at T = 291 K, the temperature step of ΔT_{max}) in comparison to that of the La(Fe, Co, $Si)_{13}$–filled PIT-wire of [19]. Both samples display the typical progression of temperature upon a magnetization pulse. Upon increasing of the magnetic-field, the cores exhibit the magnetocaloric effect and the core materials heat up by ΔT. In the composite wires the surrounding steel jacket is heated by this temperature change of the magnetocaloric cores, resulting in a temperature increase of ΔT_{eff}.

For the Gd-filled PIT-wire it is visible that the steel jacket exhibits only about 1/5 of the temperature change the core provides at field maximum of 2 T. The highest temperature change at the steel jacket is shifted towards the demagnetization step. This has already been reported in [19] and can be attributed to the time the heat of the core needs to be transported to the outside, since the steel jacket acts as a heat sink and is not exploiting a temperature change itself. This delay in temperature change due to heat transfer phenomena results in the displayed hysteresis of composite wire curves, where the measurement of the temperature change takes place at the outside of the steel jacket. The hysteresis of the Gd-core can be attributed to the time profile of the pulse, which can be seen as the blue curve in Fig. 6b. The time to reach the magnetic-field of 2 T is shorter than for the reduction of the field back to zero. Despite the high field-sweep rate, the thermocouple reacts quasi-instantaneously, indicating a sufficient thermal coupling between thermocouple and sample. Appreciable eddy-current heating is neglected, since this would have heated the samples while, both, increasing and decreasing the magnetic-field, leading to a higher final temperature than started, whereas closed loops are shown in Fig. 6.

In comparison to the La(Fe, Co, $Si)_{13}$-filled PIT-wire, the Gd-filled PIT-wire shows a larger ΔT_{eff} at 2 T, however, in relation to the ΔT_{ad} that can ideally be transferred from the Gd this ΔT_{eff} is reduced largely. Although, the Gd-core provided more than twice the temperature change of that from La(Fe, Co, $Si)_{13}$, the temperature change ΔT_{eff} at the

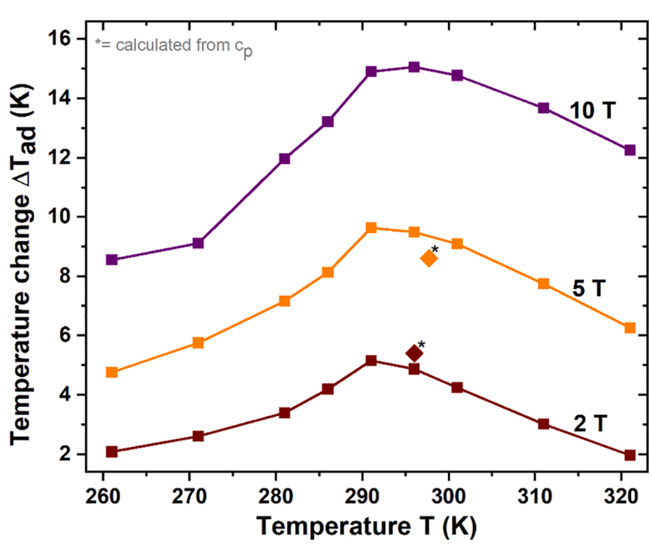

Fig. 5. Temperature change plotted against the initial temperature for the separated Gd-core of the annealed composite wire, with addition of temperature change values calculated from c_p-measurements.

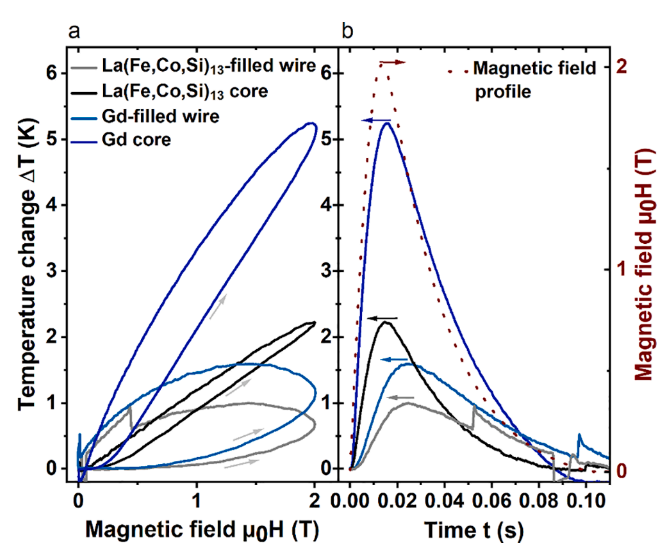

Fig. 6. a) Temperature change vs. the external magnetic-field for a Gd-filled and a La(Fe, Co, $Si)_{13}$-filled PIT-wire as well as separated cores of both composites, b) Time profiles of temperature change and applied magnetic-field pulse. Values were measured with pulsed magnetic-fields of 2 T in the vicinity of T_t of the respective magnetocaloric material.

steel jacket is only 31% at ΔT_{max} and even less at the field peak of 2 T. In the La(Fe, Co, Si)$_{13}$-filled PIT-wire 45% at ΔT_{max} and 31% of that ΔT_{ad} provided by the core at the field peak of 2 T are observed as the temperature change ΔT_{eff} at the steel jacket.

Fig. 6b displays the time dependence of the temperature change for the cores and steel jackets of both materials and the time dependence of the 2 T magnetic-field pulse. The maxima of ΔT_{ad} and ΔT_{eff} are shifted, which shows that the heat transfer between core and jacket has not been completed during the magnetization pulse. Given the fact that the frequency in pulsed magnetic–fields is quite high, this can be overcome by increasing the time to achieve maximum field or in other words decreasing the frequency. However, in the presented comparison it is shown that the peak positions of the temperature changes agree well for both materials. This indicates that heat transfer phenomena, due to a slightly different thickness of the steel jacket or slight differences of the filling factors, do not contribute to the overall reduction of ΔT_{eff} in this comparison. This is rather a consequence of the different heat capacities of Gd, La(Fe, Co, Si)$_{13}$ and steel and more importantly their ratio when combined to a composite. While in the samples of [19], the ratio of c_p was 1:1 with approximately 500 $Jkg^{-1}K^{-1}$ for steel and La(Fe, Co, Si)$_{13}$, respectively, the heat capacity of the Gd-core is roughly half of that of the steel tube. As shown in the simulated data of [19], an increased heat capacity of the passive jacket material leads to a reduced effective temperature change.

Summarizing the magnetic-field pulse experiments, it is presented, that implementing a magnetocaloric core with a higher ΔT_{ad} cladded in the identical steel tube indeed increases the temperature change of the composite wire jacket. But, to overcome the losses due to the passive steel jacket and to utilize the highest possible amount of heat provided by the magnetocaloric core, it is necessary to tailor the ratio of the heat capacities of the active core and passive jacket.

4. Summary and conclusions

A Gd-filled composite wire has been successfully prepared with PIT-process, consisting of a 316L steel jacket and Gd-bulk. After swaging it a diameter of 1 mm, a wall thickness of approx. 100 μm and a filling factor of 62 vol% have been determined by XCT. Characterization in static magnetic-fields showed the influence of deformation-induced defects by means of a reduced magnetization to one half of the reference Gd-bulk. Annealing at 773 K for 1 h and in an Argon-atmosphere fully restored the magnetic properties of the deformed Gd-core to its as-cast level. It is shown that the existing reduction of the magnetocaloric effect is only due to the passive jacket material. Annealed Gd-filled composite wires displayed an entropy change of 3.0 $Jkg^{-1}K^{-1}$ at a transition temperature of 294 K. Therefore, the produced composite wires provide the advantage that Gd as magnetocaloric material can be used in a regenerator even in harmful environments (corrosive heat-transfer fluid), since it is protected by the surrounding steel jacket.

The field-induced temperature changes in the core ΔT_{ad} and ΔT_{eff} at the surrounding jacket are measured by pulsed-field measurements of a Gd-filled composite wire and a Gd-core separated from the jacket by grounding. The reduction of the ΔT_{ad} provided by the core to ΔT_{eff} of the steel jacket is greater than in La(Fe, Co, Si)$_{13}$-filled wires, resulting in a temperature change of 1.2 K at 2 T for the steel jacket, while the Gd-core provides 5.2 K at 2 T. The assumption that a magnetocaloric material with a larger ΔT_{ad} compensates losses due to the passive steel jacket can only be partially confirmed. The overall ΔT_{eff} of the composite wire is indeed increased compared to La(Fe, Co, Si)$_{13}$-filled wires of [19], but the heat capacity ratio pointed out to play an important role in terms of usability of the provided temperature change of the core. The full potential of the higher adiabatic temperature change of Gd cannot be used due to the large c_p of the steel jacket.

The findings affect the materials' choice for further magnetocaloric wire composites provided with the PIT-technology. To the property profile of the surrounding passive material, heat capacity has to be added as a crucial parameter in addition to mechanical properties and heat treatment temperatures.

CRediT authorship contribution statement

L. Beyer: . : Conceptualization, Visualization, Investigation, Methodology, Writing - original draft, Writing - review & editing. **B. Weise:** Investigation, Methodology, Writing - review & editing. **J. Freudenberger:** Resources, Writing - review & editing. **J. K. Hufenbach:** Resources, Supervision, Writing - review & editing. **T. Gottschall:** Investigation, Methodology, Resources, Supervision, Writing - review & editing. **M. Krautz:** Conceptualization, Methodology, Resources, Supervision, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability statement

The raw and processed data required to reproduce these findings will be made available upon reasonable request.

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