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Getting magnetocaloric materials into good shape: Cold-working of La(Fe, Co, Si)$_{13}$ by powder-in-tube-processing

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ABSTRACT

The powder-in-tube (PIT) technology was applied to La(Fe, Co, Si)$_{13}$ powder cladded by a thin seamless austenitic steel jacket. Wires appear to be promising in the search for alternative regenerator geometries, since they offer various possibilities of arrangements allowing to optimise heat transfer and pressure loss within the boundaries set by parallel plate and sphere beds. Here, pre-alloyed La(Fe, Co, Si)$_{13}$ powder was filled in a AISI 316L austenitic steel tube and swaged to wires with an outer diameter of 1 mm. By mechanical deformation, the steel jacket thickness was reduced to about 100 μm surrounding the magnetocaloric core. A post-annealing of only 10 min at 1050 °C is sufficient to form the magnetocaloric NaZn$_{13}$-type phase resulting in an entropy change close to the value of a pure reference sample. The presented technology is not limited to La(Fe, Co, Si)$_{13}$/steel combination but can be extended to material pairs involving wire core materials with a first order transition, such as Fe$_2$P-type or Heusler alloys.

1. Introduction

Magnetic refrigeration has significantly headed forward towards application during the last years. More and more research groups bundle their activities on the construction of prototypes and test stands to evaluate different materials under equal conditions on one hand, but also on the development of shaping technologies for the production of thermally effective regenerator beds on the other hand [1–8]. These regenerator beds consist of the magnetocaloric material and voids that allow a heat-exchanger fluid to flow through. Up to now, efforts were mainly focused on the production of packed particle beds and plane plates that can be stacked and separated in the regenerator bed by spacers to provide channels for the heat-exchanger fluid. Easy to fabricate, advantageous on the first sight, both configurations inhere processing difficulties that reduce their theoretical performance drastically once assembled and operated in a test device. Packed beds, although stabilised by tiny amounts of polymer [9,10] tend to segregate and sediment after many cycles leading to a maldistribution of the heat-exchanger fluid. Moreover, mechanical degradation of the particles results in a rise of the pressure drop and more severely, to flushing the small fragments into the surrounding heat-exchange system. For parallel plate beds on the other hand, a convenient stacking technology leading to parallel plate beds that satisfy the strict geometric requirements of the provided channel geometry is still lacking: From theoretical considerations, extremely small spacing between the plates (0.05–0.075 mm [11,12]) and plate thicknesses of <0.1 mm are favoured to provide large thermal efficiency of the regenerator beds [11–13]. Further, these geometric dimensions should vary only negligibly across the length of the regenerator bed. With common preparation methods, such as laser welding [2], or gluing spacers [14], these requirements can hardly be realised.

Moreover, due to the brittleness of the relevant materials for room-temperature applications, namely-MnFePSi- and La(Fe, Si)$_{13}$-based alloys, plates are usually composites composed of magnetocaloric powder and a non-magnetocaloric metal or polymer binder. The binder reduces the amount of active material in the plates and largest amount of active material up to now is about 70 vol% [6]. It is to consider that this amount will be reduced further in the regenerator depending on the spacing between the stacked plates. In plate regenerators produced by wire Electrical Discharge Machining (EDM) from bulk material, an overall amount of voids for the heat-exchange fluid of 30 vol% was reported [2]. However, EDM is both time and material consuming.

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Within the quest for an optimal trade-off between heat-transfer and pressure loss, plate and particle or sphere beds represent only the upper and lower boundaries [15]. Various alternative geometries have been suggested and for conventional regenerators, materials can be used that allow for facile manufacturing [16–18]. However, the amount of publications with successful preparation of more complex geometries for magnetocaloric regenerators for room-temperature application, is rather modest. Moore et al. [19] have shown, that fin-like structures can be prepared by additive manufacturing and double-sided comb-like structures have been prepared by extrusion by Wieland and Petzoldt [4]. Such structures are likely to fail during magnetic cycling, either by the mechanic torque generated by the magnetic field change or heat-exchanger fluid or due to corrosion damage.

Instead, the Japanese electronics company Fujikura has presented coiled Gd-wires with diameter from 0.25 to 0.5 mm as regenerator building blocks [20]. This approach is intriguing, since wires offer the possibility of a variety of arrangements (some or suggested in Fig. 1d) either in cross-flow or parallel-flow configuration. Such wire-regenerators can exceed the coefficient of performance of conventional plate or sphere beds with similar heat transfer area [18,21]. Moreover, amorphous Gd-based wires with extremely low diameter of about 50 μm were prepared by melt-extraction with transition temperatures around 100 K [22–25]. Optimised doping might lead to transition temperatures around room-temperature. Apart from magnetic cooling, wires with a magnetocaloric core can also be deployed in other energy applications, e. g. as active element for a magnetic flux switch in thermomagnetic generators.

The necessity of substituting Gd as regenerator material is widely accepted, but shaping first-order substitute materials into thin wires is still a challenge. Although $La(Fe, Si)_{13}$ wires have been prepared successfully by melt-extraction, there is a high risk that after an annealing treatment, necessary to establish the magnetocaloric phase, the wires will break apart easily and mounting in a regenerator will be impossible. Additionally, these wires should be cladded by a ductile jacket to prevent mechanical degradation resulting from the magneto-volume transition during magnetic cycling in a refrigeration device. Shao et al. were the first presenting Cu-cladded ribbons of amorphous Gd-based nanocomposite powder produced by cold-rolling. The ribbons obtained were of several 1–1.5 m length and had a cross section of about 10 × 0.5–1 mm^2 [26,27]. For optimal heat transfer a rotationally symmetric geometry is favoured. This is why we apply a different approach and present a way for producing thin magnetocaloric wires by the powder-in-tube (PIT) process. This technology has been developed initially for the production of superconducting electric wires (so-called "Kunzler wires") [28,29]. We extended this technology to magnetocaloric wires. In comparison to other recently applied technologies, all steps involved in PIT call back on already industrially established processes.

Here, we use $La(Fe, Si, Co)_{13}$-based powder cladded in an austenitic steel tube which was mechanically reduced in size down to 1 mm diameter. A maximum filling factor of $La(Fe, Si, Co)_{13}$-core within the tube of 0.58 and a thickness of surrounding steel jacket of 100 μm was achieved. The steel jacket tube is a low-cost semi-finished product and shows good cold-working capabilities. Considering an austenitic steel jacket is not only advantageous in terms of corrosion protection, but at the same time the steel can compensate mechanical stresses occurring from the magneto-volume effect appearing during the magnetic transition in the magnetocaloric core material. This prevents the core from mechanical degradation during magnetic field cycling.

2. Materials and methods

In the view of optimising heat transfer and magnetocaloric filling factor, the jacket of a PIT composite needs to meet two challenging features, *minimising* the outer diameter d_o while at the same time *maximising* the inner diameter d_i. The ratio of d_i^2/d_o^2 defines the filling factor of the magnetocaloric material but is limited at the same time by the capability of deformation of the core/jacket - material combination. Here, we applied a d_i^2/d_o^2 ratio of 0.69 which competes with the filling factor of recent composite plates. As explained earlier, the composite rods can be arranged as hexagonal bundles, with a theoretical packing density of 92 vol% (and 8 vol% voids = fluid flow channels), leading to an effective amount of MCE material inside the heat-exchanger chamber of 63 vol% and, thus, reduction of magnetocaloric material in the regenerator is marginal in comparison to parallel plate beds composed of composite plates and spacers.

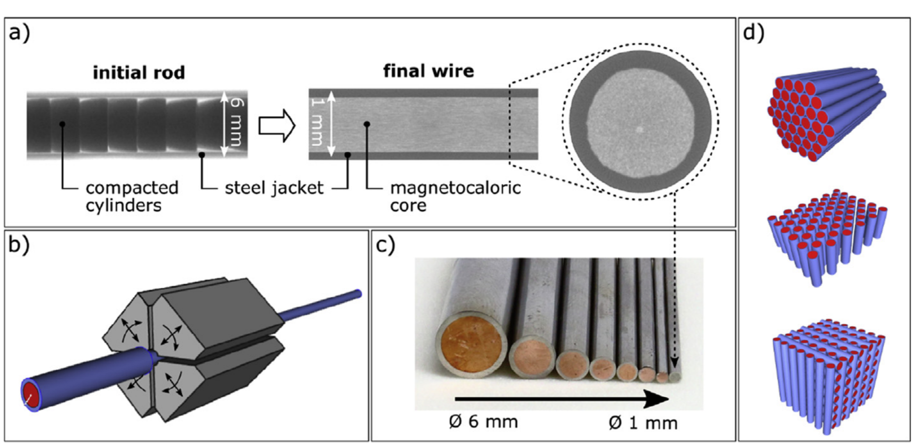

Fig. 1. Radiograph of the steel tube filled with compacted pre-alloyed $La(Fe, Co, Si)_{13}$ cylinders before swaging. Clearly, the conical shape of the powder cylinders can be distinguished (a, left). Tomographic slices of the swaged PIT wire with an outer diameter of 1 mm (a, right). Schematic illustration of the PIT swaging process (b) and photograph of PIT wires after several swaging steps (c). Copper cores are shown here to improve visibility. The right wire corresponds to the $La(Fe, Co, Si)_{13}$/steel combination discussed in this work. The sketches in (d) demonstrate possible designs of regenerators when wires are arranged differently.

2.1. Preparation of magnetocaloric PIT wires

An austenitic seamless AISI 316L steel tube with $d_o = 6$ mm and 0.5 mm wall thickness, i.e. $d_i = 5$ mm, was applied as outer jacket. The d_i^2/d_o^2 ratio = 0.69 defines the theoretical filling factor of 69 vol% of magnetocaloric core material. As core material a mixture of pre-alloyed La(Fe, Co, Si)$_{13}$ powder, described in Ref. [30] with a mean particle diameter < 20 μm, was provided by Vacuumschmelze GmbH & Co. KG. The powder was precompacted to cylinders with a $d = 5$ mm mould at $p = 1$ GPa in order to enhance the density of the core. After compaction the powder cylinders show a bale density of about 5.25 g cm^{-3} which was determined by XCT. The cylinders had to be manually grinded on their lateral surface in order to fit into the steel tube reducing the core volume slightly by about 10%. Fig. 1a, left shows the X-ray absorption image of the filled steel tube and the conic shape of the grinded, compacted La(Fe, Co, Si)$_{13}$ powder pellets. The filled tubes were plugged by Cu-cylinders on both sides. Cold work was applied by rotary swaging (HMP UR-2-4) to a final wire diameter of 1 mm. In total sixteen swaging steps with an areal reduction per pass of approx. 20% were applied. During processing (schematically shown in Fig. 1b) up to a total logarithmic degree of deformation of $\varphi = 3.58$ no necessity to apply an intermediate heat treatment was observed, thus, not performed. Fig. 1a, right shows the tomographic slice of the as-deformed wire with 1 mm diameter and a surrounding steel jacket width of only approx. 100 μm. By reducing the rods' diameter from 6 to 1 mm (exemplarily shown in Fig. 1c, the deformed Cu-plug is shown to enhance visibility), its length increased from 100 mm to 3600 mm and the density of the core with the pre-alloyed particles increased to an average of about 6.9 g cm^{-3} out of five samples. In order to form the magnetocaloric phase in the core and to release deformation stresses in the steel jacket, an annealing was performed after swaging. Wire pieces were wrapped in Ta-foil to getter residual oxygen during annealing. To investigate the influence of annealing time, heat treatments were performed at 1050 °C for 10, 30, 60, 120 and 240 min, respectively under Ar-flow and subsequent quenching in water. Two references for magnetic investigations were prepared. Firstly, a pure AISI 316L steel rod was swaged and annealed afterwards for 10 min in the same manner as the PIT wires and, secondary, a core was extracted from a 10 min annealed PIT wire by grinding off the surrounding steel jacket manually. In order to convert mass and volume related figures, a density of $\rho = 7.2$ g cm^{-3} of the magnetocaloric La(Fe, Co, Si)$_{13}$ core after heat treatment (from to [30]) and $\rho = 8.0$ g cm^{-3} of the AISI 316L steel tube (according to supplier's website [31]) was assumed.

2.2. Characterisation of the core material and PIT wires

The inner constitution of the prepared PIT wires was studied by X-ray computed tomography (XCT) with a GE Phoenix Nanotom M device. The sample volume was irradiated with X-rays from a transmission tungsten target, using an acceleration voltage $U = 125$ kV and current $I = 100$ μA. While scanning the sample, 1200 absorption images were captured during a 360° rotation of the sample taken at discrete angular positions with an exposure time of $t = 0.75$ s. Phoenix datos|x 2.2 software was used for volume reconstruction with the standard FDK reconstruction algorithm [32] and VG-studio max 2.2 image analysis software for the determination of the volume fraction of the steel jacket and inner core. The microstructure was analysed by a Zeiss LEO 1530 GEMINI scanning electron microscopy equipped with field emission gun using a four quadrant backscattered electron detector. Chemical composition of the present phases was determined by energy dispersive X-ray spectroscopy (EDS) using a Bruker EDS-system. Magnetic measurements were performed in a magnetic property measurement system (Quantum Design MPMS-XL). Temperature dependent magnetisation in an external field of $\mu_0 H = 0.01$ T from 250 to 350 K was measured in 2 K steps at a temperature sweep rate of 2 K min^{-1}. The entropy change was calculated from field dependent magnetisation curves by applying the Maxwell relation. Isothermal $M(H)$-curves were measured at temperatures near transition temperature from 270 to 340 K differing in 5 K. The magnetic field was varied from 0 to 2 T for each temperature in oscillate mode and magnetisation was protocolled in 0.1 T settle point steps.

3. Results and discussion

3.1. Microstructure of deformed and heat treated PIT wire

As can be seen from Fig. 1a the magnetocaloric core is capsuled within an approx. 100 μm thick steel jacket and the core/jacket interface is free from macroscopic flaws. The precompacted cylinders are deformed uniformly forming a continuous core along the PIT wire. It is to mention, that the theoretical filling factor of 0.69 is now reduced to approx. 0.58 due to the loss of material by manual grinding the precompacted cylinders. This has been necessary to fit the La(Fe, Co, Si)$_{13}$ cylinders into the steel jacket. Dislocations and other microscopic defects are induced by the mechanical deformation during swaging, especially in the steel jacket, and the microstructure is refined in both, the steel and the core-powder (compare Fig. 2a and b). The interfacial energy in the La(Fe, Co, Si)$_{13}$/steel-compound is increased after the mechanical deformation. In Fig. 2 microscopic images of the as-deformed (b), 10 (c) and 240 min (d) annealed samples are shown exemplarily to illustrate the effect of annealing on phase formation and microstructure. In the as-deformed wire the core consists of the pre-alloyed powder (Fig. 2b) which is transformed into the magnetocaloric main-phase after 10 min annealing (Fig. 2c). Additionally, a diffusion fringe with the width of about 5 μm is visible. Increasing the annealing time does not lead to a significant increase of the magnetocaloric main phase, but the thickness of the diffusion fringe between core and steel is slightly increased (to about 20 μm after 240 min annealing). The change of chemical composition over the core/jacket interface for the as-deformed and the 240 min annealed samples is shown in Fig. 2e. The steep drop (from jacket to core) in Ni- and Cr-concentration at the interface in the as-deformed wire becomes smoothened due to diffusion after annealing. Further, EDS point analysis revealed that the fringe consists of Ni-enriched La-rich phases and Fe with Cr. Ni and Cr atoms might diffuse from the steel jacket into the magnetocaloric core, leading to a decomposition of the NaZn$_{13}$-type phase into the La(Ni)-rich and Fe(Cr)-rich equilibrium phases. To conclude, a 10 min annealing at 1050 °C is sufficient in a thin wire to form the magnetocaloric NaZn$_{13}$-type phase from a highly deformed powder and the diffusion fringe takes only < 5 vol% of the magnetocaloric core even after 240 min annealing.

3.2. Magnetic and magnetocaloric properties

Inducing microscopic defects by mechanical deformation affects the magnetic properties as observed in many material classes. In the as-deformed wire no clear magnetic transition is observed around room-temperature as expected since the core material is not magnetocaloric yet (Fig. 3a, top). However, the deformed steel reference shows a low magnetisation which can be attributed to structural changes (martensite) in the austenitic phase during deformation. After a heat treatment of 10 min at 1050 °C these defects are reduced and the steel has a neglectable magnetisation again.

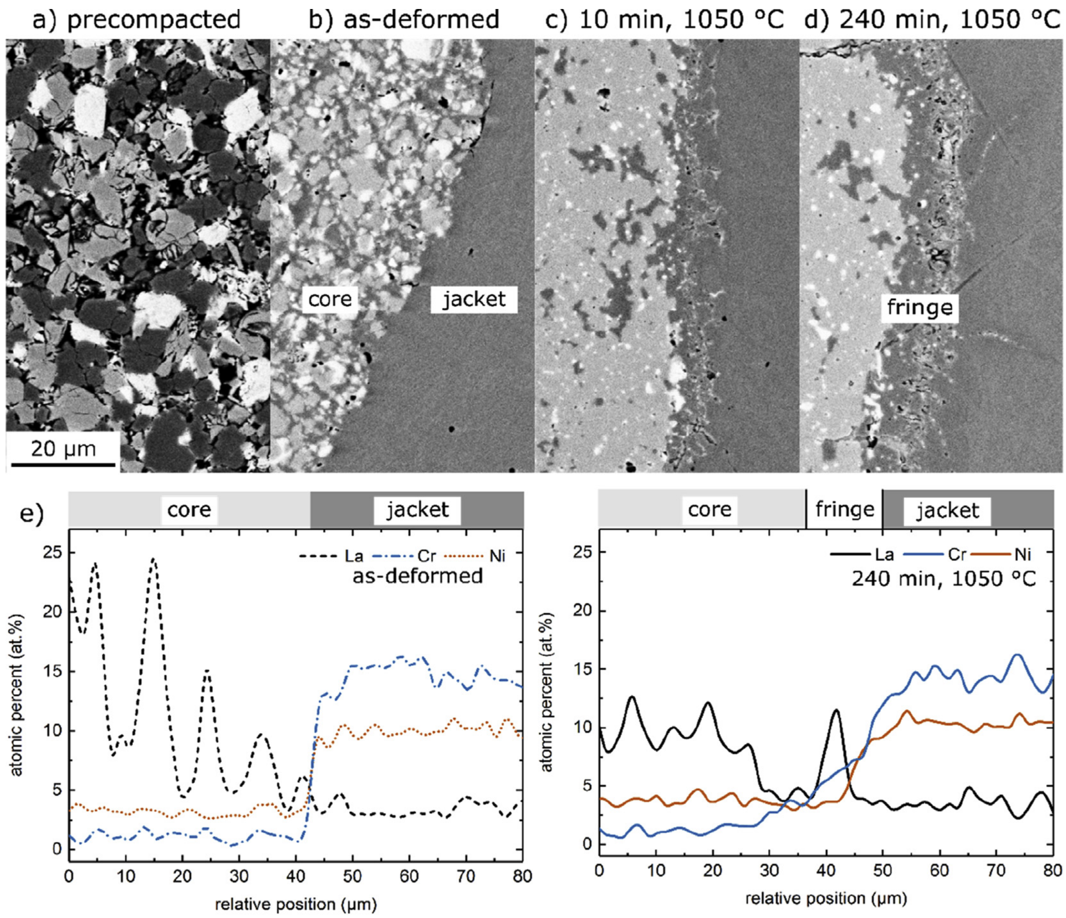

Fig. 2. Microstructure of the precompacted powder (a), as-deformed (b) and annealed wire for 10 min (c) and 240 min (d). EDS line-scan shows the change of La, Cr and Ni concentration over the range of the core/jacket interface in the as-deformed sample and the 240 min annealed sample (e).

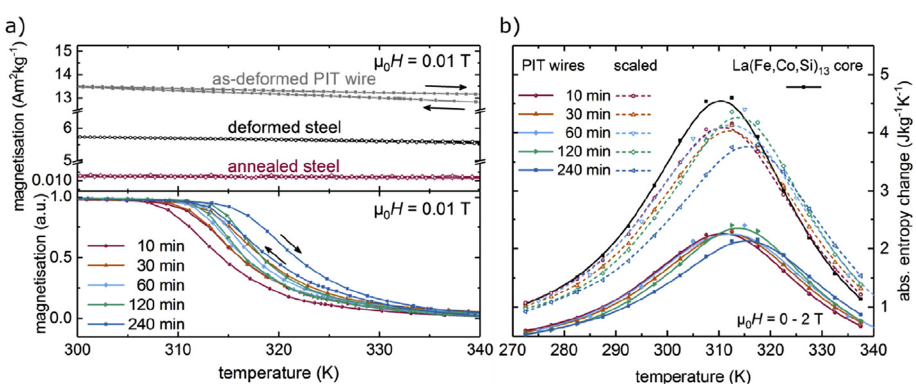

Fig. 3. Temperature dependent magnetisation of as-deformed wire and steel reference (a, top) and of annealed PIT wires (a, bottom). Entropy change of annealed PIT wires and reference core (b), the curves from the PIT wires were scaled (dashed) to a hypothetic amount of 100% magnetocaloric material in the wire for better comparison with the reference La(Fe, Co, Si)$_{13}$-core.

As shown in the microscopic images, the $NaZn_{13}$-type phase is formed after 10 min annealing. However, magnetic transitions near room-temperature are observed in all annealed samples (Fig. 3a, bottom). It is to note, that the transition in the shortest and longest annealed samples mark a transition temperature window in which all intermediate annealed samples transform. Since the formation of the $NaZn_{13}$-type phase is driven by diffusion, a certain time should be provided for annealing in order to establish chemical equilibrium of the magnetocaloric phase. After 30–60 min this equilibrium seems to be achieved. Moreover, an annealing time of 30 or 60 min appears to be practically feasible, in the light of reproducibility, rather than a short 10 min annealing. The shift of transition temperature of about 5 K from 10 to 240 min appears is rather slight. Increasing diffusion has been observed when increasing the annealing time, however change in chemical composition of the $NaZn_{13}$-phase is only expected in the area of the appearing fringe. Moreover, a broader distribution of chemical composition would result in a broader magnetic transition as compared to shorter annealed samples, which was not observed here. This might be also due to the fact, that the Co-content used for the experiments here, causes a broadening of the magnetic transition compared to ternary La(Fe, Si)$_{13}$, making it hard to distinguish from broadening induced by chemical inhomogeneities. Fig. 3b shows the comparison of entropy change of the core reference material and the annealed PIT wires in mass related units. The reference shows a maximum entropy change of about 4.5 J kg^{-1} K^{-1} which is in good agreement to a similar Co-doped alloy used in Ref. [19]. In comparison to the magnetocaloric core reference sample, the entropy change in the PIT wires is reduced by a factor 0.5, which corresponds to the amount of surrounding steel which is about 45 m% when converted from 42 vol% (since the volume of the core is about 58 vol%). Scaling the specific entropy change in the wires to 100% magnetocaloric phase by multiplying with the inverse mass fraction of the core, the values of the scaled entropy change (dashed lines) in the annealed samples are similar to that of the reference sample and lie within the error of the method. This clearly shows, that the magnetocaloric effect can be established in mechanically deformed La(Fe, Co, Si)$_{13}$ PIT wires by post-annealing.

4. Conclusions

In this work, the powder-in-tube processing technology was successfully applied for shaping magnetocaloric wires. To summarise, the following can be stated:

– Composites consisting of an AISI 316L steel jacket and La(Fe, Si, Co)$_{13}$-based powder can successfully be deformed by rotary swaging to a logarithmic deformation strain of 3.58 without intermediate heat treatment
– After deformation the steel jacket thickness was about 100 μm and fraction of the magnetocaloric core was about 58 vol% (from XCT) which is close to that obtained in packed or parallel plate beds
– Non-magnetocaloric powder was used for the production of thin wires avoiding long annealing treatment of the bulk alloy beforehand
– Annealing time to form the magnetocaloric NaZn$_{13}$-type phase throughout the whole core was drastically reduced to only 10 min at 1050 °C
– The reduction of annealing time can be attributed to the mechanical defects induced, refinement of the microstructure and the low wire diameter after swaging

5. Outlook

Up to now wires have not been considered to be applied as active regenerators due to time-consuming processing techniques and/or due to the brittleness of the wires consisting purely of magnetocaloric material after post-annealing. In this paper, composite wires with a brittle magnetocaloric core were produced for the first time. The presented results can be applied to other magnetocaloric material families, such as Fe$_2$P-based materials or Heusler alloys. Other ductile jacket materials, such as Cu-based alloys or Nb, can help to improve the thermal properties of the wire composite while serving as corrosion protection at the same time. Measures to surpass the presented filling factor of magnetocaloric material can be applied in future studies, such as using cold-isostatic compacted powder with enhanced bale density as core material filled in an outer jacket with reduced wall-thickness and outer diameter, in order to reduce the logarithmic degree of deformation required to achieve the final d_o of 1 mm and even below. Further shaping of the wires or direct mounting in a regenerator bed appears to be feasible since mechanical integrity after post annealing is warranted by the ductile jacket. In general, the PIT technology offers a vast playground for materials combinations and optimisation but more importantly proves to be a convenient way of shaping magnetocaloric composite wires as promising active building blocks for versatile components in energy applications. Being able to shape magnetocaloric materials into wires opens a huge number of prospect arrangements for novel regenerator geometries (as shown in Fig. 1d).

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Appendix A. Supplementary data

Supplementary data related to this article can be found at https://doi.org/10.1016/j.mtener.2018.05.009.

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