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Influence of the annealing temperature of the shape memory alloy actuator on its thermal characteristics

Krzysztof Kuś[1*] (*orcid id: 0000-0001-9811-5831*)
Adam Frączyk[1] (*orcid id: 0000-0002-3056-3141*)
[1] University of Warmia and Mazury in Olsztyn, Poland

Abstract: Shape memory alloys (SMA) are used in different areas of engineering and science thanks to their unique properties. They also continue to be an innovative material for the sustainable construction industry. In this study, a commercial helical-type SMA spring actuator was investigated by subjecting it to annealing at various parameters. The thermal shape memory properties were evaluated by means of the DSC method. In most cases, the higher the annealing temperatures for the material were in the range up to 595°C, the lower the transformation temperatures. As the DSC runs showed, a different character of the changes especially in characteristic temperatures, was observed for annealing temperatures above 600°C. The results showed that the different annealing temperatures, and even the method of cooling, provide a wide range of possibilities to control the SMA spring reaction – transformation behaviour and temperatures. Such treatment can be a simple technical procedure used for the preparation of the selected SMA functional properties if required. This means that the same SMA element can be reused without having to source a new one. This may be desirable from the point of view of sustainability.

Keywords: modern materials, SMA, actuator, annealing, thermal properties, reusing

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Introduction

Contemporary technology relies on actuators that are small in size and capable of generating high output forces simultaneously. In many applications, shape memory alloys (SMA), as the main representatives of the smart materials group, can be of great use especially when it comes to this type of combination (Cederström &

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[*] Corresponding author: krzysztof.kus@uwm.edu.pl

Humbeeck, 1995; Follador et al., 2012; Mavroidis, 2002). SMA actuators can have the shapes of wires or helical springs. In the case of the latter, their main advantage is they generate significant displacements with relatively small microscopic material deformations. There are also other advantages of spring actuators, particularly if they are made of Nitinol alloy (Ni-Ti) (Degeratu et al., 2008). Spring actuators based on SMAs, which are also components of other devices, can be used in many fields of engineering and other disciplines, such as medicine. Also, due to other properties like the superelasticity and high capacity of energy dissipation, SMAs are applied in civil engineering including buildings and other infrastructure (Alam et al., 2005; Chang & Araki, 2016; Fang et al., 2023; Sadashiva et al., 2021). Of great interest today is the development and implementation of smart systems, especially for sustainable civil engineering. Such systems are being built, for example, using SMAs that can be integrated into structures providing functions such as sensing, actuation and information processes essential to monitoring, self-adapting and even healing of structures.

SMAs allow the direct conversion of thermal energy into mechanical energy. When used as actuators, these types of devices bear little resemblance to the actuators previously used, whether hydraulic or electric. In addition, they allow for significant design simplification, improved structure properties, longer equipment life, miniaturisation and lower manufacturing costs. All they need to operate is electricity, water, an air stream or other heating and cooling medium. It is also important to note that they are safe, quiet and do not pollute the environment with exhaust fumes or other waste products. They are able to use practically any heat source for their operation, e.g. from flue gases or waste water, or that resulting from natural temperature differences. As is known, these features and opportunities are important for the realisation of sustainability. SMAs, especially Ni-Ti, are distinguished by their multifunctionality, i.e. the juxtaposition of different features within just one material structure. Up to now, if there were a need for multiple functions this was achieved using different materials with single functions (e.g. separate material for the sensor, others for the actuator or the vibration damper). The multifunctionality of SMAs may also be relevant from the point of view of sustainability.

The one-way shape memory effect (OWSME) is the basis of most SMA actuators. In this regard, there are three basic types of actuators: one-way actuator (gravity), biased actuator (spring, bias actuator), two-way actuator (antagonistic SMA, differential actuator, two-way actuator with one-way materials) (Hu et al., 2021; Huang, 2002; Mansourizadeh et al., 2021; Mavroidis, 2002; Weirich & Kuhlenkötter, 2019). Examples and solutions of applications of the above-mentioned modes are presented in the papers (Hattori et al., 2014; Hu et al., 2021; Mohd Jani et al., 2014; Sharma et al., 2018; Stoeckel, 1995).

When designing various actuators using SMA, many different factors must be taken into account. Among them are temperature-stress-strain characteristics, overheating, lifetime, maximum transformation speed, stabilization, transformation hysteresis and heating/cooling methods (Ashrafiuon & Elahinia, 2016; Rao et al., 2015; Reinaerts & Brussel, 1998). A very important shape memory property is the set of phase transformation temperatures (A_s, A_f, M_s, M_f) which it turns out can be changed, depending on the need of the target application.

Different scanning calorimetry (DSC) is one of several methods available for characterizing the thermal properties of SMAs. In short, this technique examines the energy effects during the cooling and heating of the sample (latent heat, enthalpy). Here, attention should be paid to the relevant conditions, including among others, the preparation of the sample for such tests (Abd-Elghany & Klapötke, 2018; Memry Corporation, 2017; Turabi et al., 2016). On the basis of DSC profiles it is possible to trace the transformations in SMAs and in particular, to determine the critical transformation temperatures. Moreover, a careful analysis of the obtained thermograms can be helpful in a rough assessment of the thermo-mechanical history of SMAs (Nespoli et al., 2015).

Most of the engineering applications based on SMAs (e.g. robotics, medicine, biomimetics, civil structures and automobile or domestic industry) require helical springs as actuators and proper design of such elements, including knowledge of thermal properties, is very important. It is often the case that helical-type SMA spring actuators, often without exact or even disclosed specifications, are offered by many vendors. The authors have experienced this state of affairs in practice. The main motivation of this work is to demonstrate, especially for practical purposes, that there are no obstacles to trying to modify the thermal properties of SMAs for other applications or to make changes to existing material characteristics. Besides the programming of characteristic temperatures, the shape to which the SMA is to return to during the reverse transformation can be modified. For this purpose, annealing with defined parameters is used (Kuś et al., 2019; Rao et al., 2015).

This paper aims to monitor the evolution of thermal characteristics of the SMA spring material under annealing at different parameters. In particular, the critical transformation temperatures can be tailored depending on the need of the target application. In terms of sustainability, for example, the same SMA component can be reused without the need to acquire new SMAs. This translates into a reduction of negative environmental impact during the manufacture of such materials, processing, storage or destruction.

1. Materials and methods

A commercial helical spring, made of Nitinol type wire and 0.75 mm in thickness, was used for the tests. According to the partial manufacturer's specification, the SMA spring tensioned should return to its original shape at about 40-45°C. Analysis of the element composition of the spring material was performed with use of the Laser-Induced Breakdown Spectroscopy (LIBS) method (Keyence laser-based elemental analyzer EA-300 with digital microscope VHX 7000). Before the test the spring section was ground down to reveal its inner layers. In particular, this was to remove the outer oxide layers, which could affect the test results. Measurements were taken at several points of the exposed surface (Fig. 1). In each of these places five laser pulses were used to collect data from layers below the top surface. The penetration depth of a single laser pulse was slightly over 3 μm. As revealed by the LIBS analysis, the basic chemical elements of the spring material are associated with Ti and Ni.

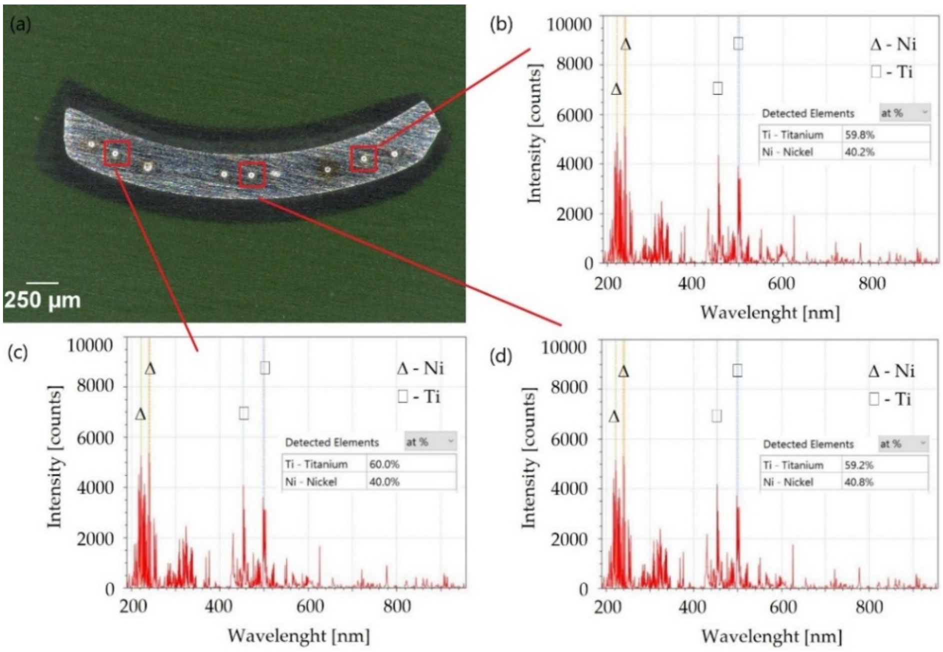

Fig. 1. The extracted spring section (a), and the measuring points (b-d) for the determination of the element composition by the LIBS analysis (*own research*)

The actuator was annealed in a muffle furnace in a fixture that ensured the sample was pre-compressed and restrained (Fig. 2). The annealing parameters are shown in Table 1. After each heat treatment, a small section of material was taken from the spring for DSC testing. The measurements were carried out on a differential scanning calorimeter, type DSC 204 F1 Phoenix® NETZSCH, which was equipped with a liquid nitrogen cooling system. The weight of the samples oscillated between 14 and 15 mg, cooling and heating in the measuring cycle for extreme values was performed at a temperature ranging from –100°C to +80°C. The measurements were carried out under a protective nitrogen atmosphere with a heating/cooling rate of 10°C/min.

Fig. 2. Fixture with the SMA spring subjected to annealing at different temperatures (*own research*)

Table 1. Annealing parameters of SMA helical spring (*own research*)

Temperature of annealing [°C]	Time of annealing [min]	Cooling type after annealing
485	15	air
500	15	air
525	15	air
555	15	air
585	15	air rapid quench in water (12.6°C)
595	15	air
610	15	air
635	15	air

The DSC profiles obtained were used to trace the phase transformations of the material and to determine the characteristic temperatures. Definitions of these temperature differences can be found in the studies (*Memry Corporation*, 2017; Reinaerts & Brussel, 1998). On this basis, the effect of the applied heat treatment carried out at different parameters on changes in the thermal properties of the SMA spring actuator were further evaluated.

2. Results and discussion

Based on the experimental DSC profiles, the estimated transformation temperatures obtained from the cooling curves were R_s, R_f and M_s, M_f. Those obtained from the heating curves represented A_s and A_f, but also under certain circumstances were R_s and R_f. The DSC results obtained during the cooling regime for the SMA spring annealed at temperatures of 485-635°C for 15 minutes are presented in Figure 3.

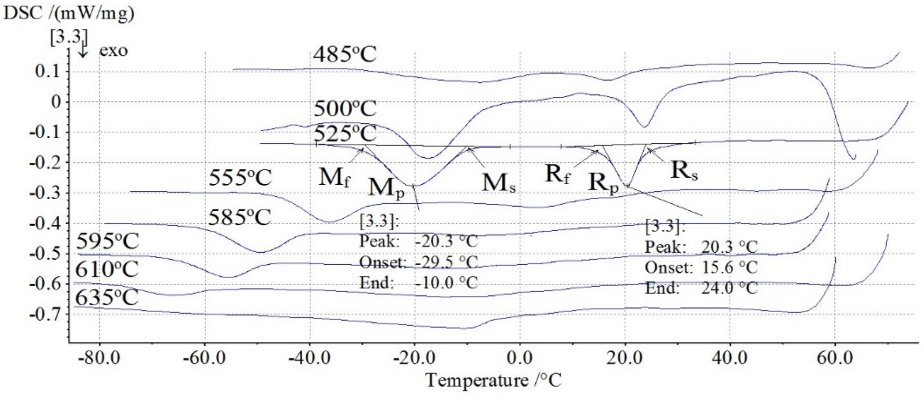

Fig. 3. Summary of DSC profiles obtained during the cooling segment for the SMA spring annealed at temperatures of 485-635°C (*own research*)

As can be seen that there are two distinct peaks, especially for treatment at 485, 500, 525 and 555°C, indicating the two-stage nature of the martensitic transformation. The first peak, viewed from the right, is associated with the transformation to the R-phase (rhombohedral phase) and the second one to martensite, respectively. The R-phase, being an intermediate one, can be observed in both cooling and heating, having its own characteristic temperatures. Its occurrence is influenced by a variety of factors, including chemical composition or thermo-mechanical processing. Thus, the R-phase appears on cooling before the transformation proceeds to martensite at lower temperatures.

Annealing at higher temperatures resulted in the first peak being observed as increasingly fuzzy and flat, and at the highest annealing temperature used, 635°C, only a single, strongly broad peak in the DSC curve is visible. As the annealing temperatures increase, the DSC peaks tend to move to lower temperatures and to decrease in intensity. Unfortunately, this will further translate into difficulty in determining the characteristic temperatures of the transformations.

The DSC results obtained during heating segment for SMA spring annealed at temperatures of 485-635°C for 15 minutes are presented in Figure 4. In the case of annealing in the range of 485-585°C, there is only one peak suggesting that transformation has a one-step behaviour from martensite to austenite. However, for annealing at 485°C, there is a slight rise on the left side of the peak, which may indicate a different nature of the transformation. A similar situation occurs for annealing above 585°C, where the peaks become increasingly fuzzy and with lower intensities, which further, at even higher temperatures, can lead to their separation and transition to the two-step character of the reverse transformation. It can also be seen that as the annealing temperatures increase, the DSC peaks tend to move to lower temperatures. However, some anomaly occurs for annealing at 485°C and 500°C, where a shift of peaks to higher temperatures is observed.

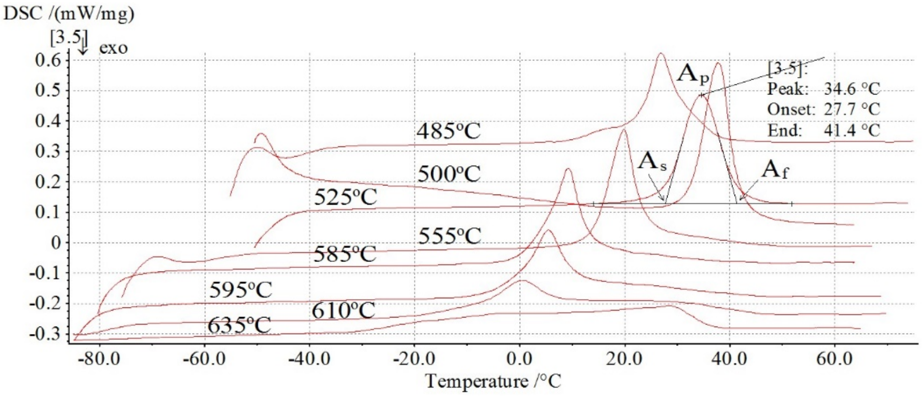

Fig. 4. Summary of DSC profiles obtained during heating segment for SMA spring annealed at temperatures of 485-635°C (*own research*)

The separate DSC peaks make it possible to directly determine the transformation temperatures. They were established using the generally excepted tangent method, as shown for profiles obtained after annealing at 525°C (Figs. 3 and 4). The collective results are presented in Figure 5, where the data obtained are supplemented with temperature values at peak maxima, denoted as R_p, M_p and A_p.

The annealing temperature is found to significantly affect the characteristic temperatures. Following annealing conducted in the temperature range of 485-500°C and above 610°C, an increase in characteristic temperatures is observed. However, this does not apply to M_s and M_p values at the lowest material processing temperatures. In contrast, in the range 500-610°C, there is a clear downward trend at almost all temperatures. Particular difficulty in analysing the transformations that occur in material annealed at 610°C, and especially at 635°C.

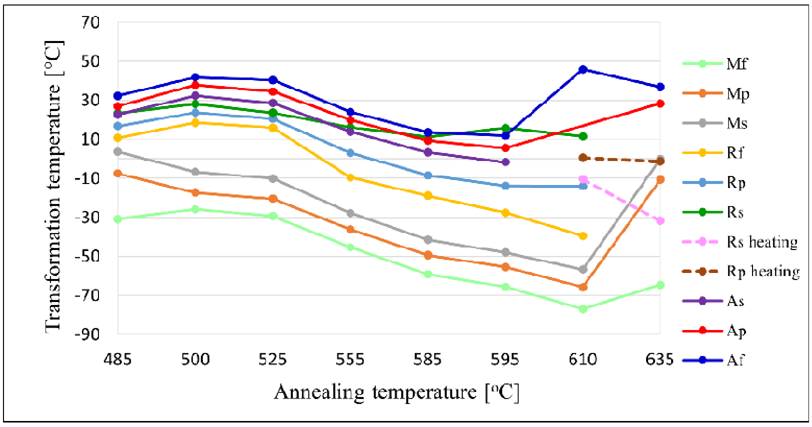

Fig. 5. Evolution of transformation temperatures of the NiTi spring material due to annealing at different temperatures (*own research*)

As can be observed, the shapes of the peaks in the case of cooling are extremely flat and broad (with a noticeable tendency to merge into one peak), while in the case of heating, two overlapping transformations can be observed. This situation results in the fact that not all the phase transformation temperatures can be determined. With cooling, for an annealing temperature of 635°C, one can only try to determine M_s and M_f. In heating, for annealing temperatures 610°C and 635°C, it becomes possible to identify R_s, R_f and A_f. The temperature of R_f, at heating, was not included in Figure 5 – its value was estimated to be about 11.2°C.

As shown, annealing in the temperature range 485-635°C with cooling in air immediately after removal from the furnace offers a wide range of possibilities to control the SMA spring transformation and critical temperatures. The experiments carried out included programming one of the critical temperatures in such a way that complete contraction of the previously stretched spring would occur under the heat of a human hand. This made it possible to anneal the material at 525°C for 15 minutes with cooling in free air. However, the behaviour of the spring immediately after it was received did not show this behaviour. It should be noted that this state of the material, i.e. using the patient's heat to react the material, can sometimes play an important function in biomedical applications. A Nitinol SMA is well suited for different medical areas (Oshida & Tominaga, 2020). A summary of the SMA spring behaviour is shown, including the observed experimentally phenomena, in Table 2.

Table 2. SMA spring behaviour after annealing at various parameters (*own research*)

Annealing parameters	Behaviour observed
500°C/15 min/air cooling	material shows no PE at room temperature* and at reduced temperature
525°C/15 min/air cooling	material shows no PE at room temperature* and at reduced temperature (OWSME already observed from temperature of 30-31°C, reaction at human body temperature; at room temperature** – PE behaviour)  about 36.6°C
585°C/15 min/water cooling	material shows PE at room temperature* and at reduced temperature
PE – pseudoelastic effect or superelasticity, definition is given in (*Memry Corporation*, 2017); OWSME – one-way shape memory effect; *at room temperature of 22.5-23°C; **at room temperature of 27°C.	

The influence of the heat treatment parameters on the behaviour of the material is also illustrated by the different methods of cooling to the ambient temperature. In the case of a spring annealed at 585°C/15 min and then cooled in air, an additional cooling in cold water was performed (Table 1). The calorimetric data obtained from these experiments are presented in Figure 6. It can be clearly seen that the same qualitative character of transformations occur in both cases. However, this is different for the characteristic temperatures. Compared to air, water cooling of the material causes them to shift towards lower values, both for the cooling and the heating transformation. The differences are significant and for example the A_s temperature may be estimated to be 20°C lower than the A_s temperature of the air cooled sample.

The experimental results show that annealing in the temperature range 485-635°C, as well as different cooling rates after such operations, provide a wide range of possibilities for controlling the course of SMA spring phase transformations and their critical temperatures. The changes are particularly well seen in the range of 500-595°C and there is no problem in estimating them, while in other ranges, the determination of critical temperatures becomes difficult due to the large evolution of thermograms (flat peaks, peak splitting, blurring). Similarly, cooling type applied from ‘slowly in air’ to ‘rapid using water’ provides the opportunity to change the transformation characteristics.

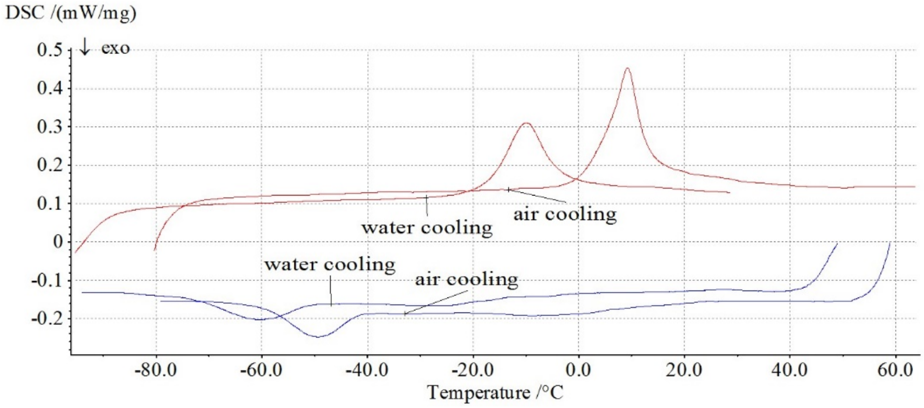

Fig. 6. DSC profiles obtained for SMA spring annealed at temperature of 585°C with two types of cooling (*own research*)

At this point it important to note that a manufacturer's specification of the received test material spring was not entirely known. Hence, before heat treatments were carried out, preliminary measurements revealed that the obtained material has low and mostly broad DSC peaks. This is related to the microstructural state. It can therefore be presumed that its thermo-mechanical history in the state of delivery is close to the cold-worked one followed by partial annealing. This condition is most commonly used in the trade of finished products with SMA. A structure free from defects would be characterised by well-defined DSC peaks with high transformation enthalpies. It is thought that annealing carried out at even higher temperatures, or at those used in experiments but over a longer period of time, would reveal just such a condition.

In summary, it is emphasised that annealing under different conditions causes microstructural changes in the Nitinol spring material reflected in its thermal properties. From the nature of the changes in the DSC curves obtained and based on the literature, it can be deduced that this is related to the lattice defects that undergo rearranging and, as a result, continuously disappear with increasing annealing temperature until recrystallization of the material. A variable trend in the critical temperatures was observed in the vicinity of the annealing temperature of 500°C followed by a continuous decrease. The higher the annealing temperature, the more the microstructural changes in the material are intensified. Annealing temperatures of the material below 500°C for 15 minutes may not yet be sufficient to introduce significant changes in microstructural features – although some of the characteristic transformation temperatures have shown a slight increase. It appears that a large degree of interference in the field of changes in thermal characteristics occurs in SMA material having the microstructure between the cold-worked and the fully annealed states. This can be a technical procedure used for tailoring of SMA functional properties for a specific, targeted application.

Conclusion

In the present study, a commercial helical-type SMA spring actuator was investigated by subjecting it to annealing at various parameters. These types of devices makes it possible to use them in a variety of technical solutions from biomedical to the construction industry. The thermal properties such as transformation behaviour and temperatures, related to shape memory properties, were evaluated by means of the DSC method. Based on the experimental results and other studies by the authors, the following was concluded:

1. The changes in characteristic temperatures can be seen particularly well in the annealing range up to 595°C, where their determination from DSC thermograms did not pose any major problems. In general, the higher the annealing temperatures in the range 500-600°C, the lower the characteristic temperatures of the tested material were observed. It was thus established that the microstructure of SMA spring actuator between the cold worked and the fully annealed states can be susceptible to annealing treatment and induces noticeable changes in thermal characteristics.
2. Although the study was performed for one selected annealing temperature of the spring material, the cooling type applied immediately after removal from the furnace, from ‘slowly in air’ to ‘rapid using cold water’, also provides the opportunity to change the transformation characteristics.
3. Based on the obtained results and the literature reports it was deduced that annealing carried out under different conditions influences the microstructural changes of the material reflected in the its thermal properties.
4. The annealing treatment performed can be an uncomplicated technique to prepare the selected SMA functional properties in a controlled way if needed. The results obtained for a commercially available SMA spring actuator can assist in the design and implementation for different systems including SMA such as nickel-titanium based alloys, and in the sense that a SMA element once acquired by a potential user can be successfully reused under other solutions without the need to source new material. The latter may be a desirable option from the perspective of sustainability.

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