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STUDY OF THE EFFECT OF COOLING/HEATING RATE ON THE THERMAL PROPERTIES OF NI-TI ALLOY (SMA) AFTER ANNEALING AT DIFFERENT PARAMETERS

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K e y w o r d s: Ni-Ti SMA, Annealing, DSC cooling/heating rate, Transformation parameters.

A b s t r a c t

This research focused on evaluation of the effect of different cooling/heating rates by differential scanning calorimetry (DSC) on changes in the phase transformation parameters of Ni-Ti shape memory alloy (SMA) following annealing at different temperatures. Samples for DSC testing were made of a spring SMA actuator. The initial study included identification of the chemical composition of the material. All the phase transformation temperatures were estimated from DSC curves using the tangent intersection method, as a commonly accepted way.

Following annealing at various selected temperatures, changes in the shapes of the calorimetric profiles recorded during heating and cooling of the material were obtained. Therefore, it is clear that annealing promotes modification of the SMA microstructure. In general, it was shown that there is influence of the cooling/heating rate performed in the DSC on the evaluated thermal characteristics of the tested material. This influence is perhaps not so pronounced if one takes the states of the material after annealing conducted at different temperatures. Regarding to the individual anneals, some of the characteristic transformation temperatures were sensitive to changes in cooling/heating rates, while others likely were not observed.

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List of symbols used in the paper

R_s, R_f, M_s, M_f – temperatures of the start and finish of the austenite (A) to R-phase (R) transformation and of the R-phase (R) to the martensite (M) transformation on cooling of the alloy, respectively

A_s, A_f – temperatures of the start and finish of the martensite (M) to the austenite (A) transformation on heating of the alloy

R_p, M_p, A_p – maximum temperature peak position of the particular transformation, respectively

R_{sheat} – temperature at which the transformation from martensite (M) to R-phase (R) transformation begins on heating of an alloy that exhibits two-stage transformation

R_{pheat} – temperature of the endothermic peak position on the DSC curve upon heating for the martensite (M) to R-phase (R) transformation of an alloy that exhibits two-stage transformation

A^*_f – temperature of the finish of the R-phase (R) to austenite (A) transformation on heating of an alloy that exhibits two-stage transformation

Introduction

Shape memory alloys (SMA), which have the shape memory effect (SMA), belong to the already numerous group of so-called smart materials. Some of more interesting functional and mechanical properties are demonstrated by a Nitinol SMA, which is based on a nearly equiatomic combination of Ni and Ti. The unique features of the material behaviour are associated with a reversible, thermo-elastic and diffusionless phase transformation (WANG 2013). These alloys typically show two stable phases between which a transformation can take place. Usually, it is referred to martensite and austenite. Under certain conditions, an intermediate phase, known as the R-phase, can occur. There are applications that SMAs can act simultaneously as a sensor and actuator or as an actuator itself performing cyclic displacement on demand. The SMA based actuators can be easily formed into various shapes (wires, ribbons, sheets, beams, torsion or helical springs).

As is known, one way to induce phase transformation in SMA can be carried out through thermal interaction by heating/cooling in a certain temperature range. The temperatures at which a phase transformation of SMA initiates or terminates are known as the characteristic (critical) transformation temperatures. According to the commonly accepted notation, they are expressed as A_s, A_f during heating and M_s, M_f during cooling. A refers to austenite and M to martensite phases, and s and f are referred to the start and finish temperatures of each transformation. Among the thermal parameters of transformation, the characteristic transformation temperatures are important parameters for almost any use of SMA.

There are the four basic (working) temperatures if the transformations are carried out in one-stage sequences. The M_s and A_f temperatures establish a temperature hysteresis (OSHIDA, TOMINAGA 2020). The critical temperatures of SMA transformations can be determined by various methods using the thermo-mechanical properties. Among them is e.g. the bend and free recovery techniques (defined as austenite finish, A_f active temperature) characterized in (Memry Corporation… 2017). They have advantages and disadvantages. The transformation between phases in SMA

is most often generated by stress or by temperature. When transformations occur without any external stress, they are called the zero-stress or stress--free transformations. In this case, DSC is the appropriate technique used to measure the phase transformation temperatures and the latent heat. It should be noted that SMA are highly sensitive to variations in material composition and thermomechanical treatments. Intermediate transformations might occur, for example. Hence, the transformation peaks obtained in the DSC thermograms can vary, which results in changes in the characteristic temperatures.

This paper focuses on the effect of different DSC scanning rates of the cooling/heating process on the changes in the phase transformation parameters of Ni-Ti SMA following annealing at different temperatures. Although, there are several reports on the effect of different DSC cooling/heating rates on changes in the transformation characteristics of SMA (Wang et al. 2005, Nurveren et al. 2008, Ivanov et al. 2019, Kaya et al. 2019, Akgul et al. 2020), these studies usually took place after one, sometimes two selected heat treatments and involved alloys with a specific chemical composition in a single paper. As is known, the different chemical composition of SMA, even within the presence of the same constituent elements, influences its transformation behaviour and properties. Therefore, this issue needs to be completed and deepened, especially when it comes to investigating the effect of different cooling/heating rates on the thermal behaviour of SMA after annealing carried out at various parameters. Following the literature, it is also sometimes possible to see some discrepancies concerning direction of changes in the critical temperatures due to the various cooling/heating rates. In fact, it is common to find DSC tests of SMA conducted at different cooling/heating rates, and detailed explanations for this are not usually available.

Material and methods

The material prepared for the study was a SMA wire formed into a coil spring. It was a finished actuator with outer diameter of 7 mm. According to the supplier, the material of the spring should be a Nitinol SMA, but he had no information on the exact chemical composition or preceding treatments. As is known, products based on SMA are often available in the cold-worked or annealed conditions. In terms of functional properties, it was found that, in the vicinity of room temperature, the stretched spring showed shape memory and it returned to its original shape at temperatures well above the room temperature. It also happens that the information on the properties of SMA provided by the supplier and even by the manufacturers is not complete or deviates from the reality. Additional tests are then necessary. Therefore, for the material used in this study, it was decided to investigate its elemental composition as this has not

been sufficiently documented. The SEM with EDS analysis was used for this purpose (Quanta 3D FEG SEM/FIB microscopy).

The DSC method (DSC 204 F1 Phoenix®Netzsch apparatus) was used to obtain the characteristics of thermally induced transformations. Prior to these tests, the SMA spring actuator was annealed for a constant time of 15 min over temperature range of 485°C to 635°C with fixed intermediate values, followed by slow cooling in air. After each heat treatment, samples were taken from the spring for microcalorimetric testing. The samples weighed about a dozen mg. In order to ensure better thermal contact between the samples and the DSC pan, one side of the samples was grinded until a flat surface was achieved. DSC measurements were carried out in the temperature range from -100°C to 80°C at cooling/heating rates of 2.5; 5 and 10°C/min with nitrogen as the carrier gas. In turn, liquid nitrogen was used to cool the samples. The above temperature range was chosen so that the samples showed a complete phase transformation. For each DSC scan rate established, two DSC runs for the reverse transformation and one run for the forward transformation were performed. However, the first temperature run was not considered due to starting the measurements with heating from room temperature. Only these complete transformation cycles during cooling and heating were taken for the analysis. For sample stabilization during each individual DSC measurement an isothermal segments were inserted between the all calorimetric dynamic ones. With regard to Ni-Ti SMA samples annealed at different parameters, thermograms taken for three selected cooling/heating rates were used to trace phase transformations, including determining changes in their critical temperatures. The transformation temperatures were estimated as the intersection of the tangents from the baseline heat flow and onset and completion of the exo- and endothermic phase transformation peaks (see Ref. ASTM Standard F-2004-00 for details of the tangent method used to define start and finish phase transformation temperatures). The data collected was analysed using the DSC Proteus software.

Results and discussion

It was determined from the SEM/EDS analysis that the average percentage concentration (in wt. %) of Ni and Ti was 56.92 and 43.08, respectively. Results from the SEM/EDS study but outside the area of visible darker particles are shown in Figure 1. These particles were identified as the TiC phase, however, the EDS spectrum is not included here. According to the literature nomenclature, the term Nitinol is referred to a group of nearly equiatomic alloys composed of Ni and Ti. They can often contain other alloying elements. Inclusions, such as oxides and carbides, can also be found in these alloys.

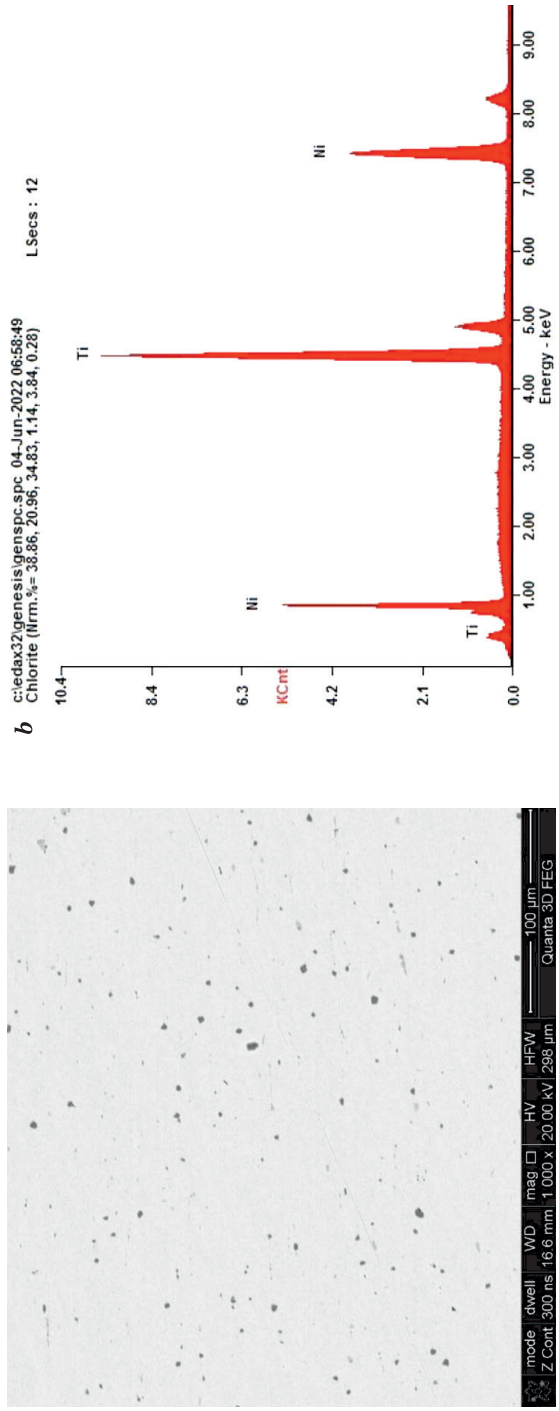

Fig. 1. SEM micrograph of the material used for EDS analysis (*a*) and EDS spectrum obtained to determine the elemental composition (*b*)

Nitinol alloys are best known for their shape memory properties. By changing the nickel content and adding other metals, the SME-related properties are influenced. These include decreasing or increasing the hysteresis, the transformation temperature and strengthening the matrix. Thus, on the basis of the SEM/EDS analysis, it was confirmed that the spring material purchased for the present study was Nitinol SMA. However it is a Ni-rich version of the equiatomic Ni-Ti alloy.

As is known, the inimitable properties of Ni-Ti SMA are fundamentally determined by their transformation behaviour. In turn, the transformation behaviour can be influenced by a number of factors, such as alloy composition, cold working and post-annealing, aging treatment and thermo-mechanical cycling. Ni-Ti alloys with nickel content of more than 50.4,50.6 at. % are considered as Ni-rich alloys. Alloys of this group are sensitive to certain types of heat treatments and may develop a number of possible Ni-rich phases (precipitates), that are known to affect thermal characteristics (HABERLAND et al. 2016). These SMA can undergo a forward transformation (austenite to martensite) during cooling in two steps through an intermediate *R*-phase formation, known as rhombohedral phase (*R*). It is rather normal behaviour. When the *R*-phase is present, the R_s and R_f are the start and end temperatures for the formation of this transformation, respectively. This transformation can be manifested during both cooling and heating. In Ni-rich NiTi-based SMA, after appropriate heat treatment, a direct transformation sequence of $M \rightarrow A$ during heating is also possible (WU, CHANG 2019). The occurrence of the *R*-phase can be caused by different reasons such as aging, increased Ni content in alloying, thermo-mechanical treatment or thermal cycling. It is also the case that, under certain ageing condition, transformation can occur in three or more steps, which is considered to be unusual multiple step behaviour (DLOUHY et al. 2003, OSHIDA, TOMINAGA 2020). The multiple-stage phase transformations in Ni-Ti alloys associated with SME is depending on the thermal and/or mechanical history of the alloy, as reported in work (BRAZ FERNANDES et al. 2013).

The occurrence of three different phases as a function of temperature (*A*, *R* and *M*) is characterized on the DSC thermograms by different transformation peaks. The existence of an intermediate phase results in changes in the transformation path during cooling, but also in some cases during heating. Note only selected DSC runs will be included in this article. DSC results for material annealing temperatures of 500, 555, 585 and 610°C did not show significant qualitative differences from those discussed, so they will not be shown here. Nevertheless, the author addresses this issue further below. On the basis of the calorimetric measurements (Figs. 2-4), it can be seen that the forward transformation from austenite to martensite is exothermic (heat emitting reaction) whereas the reverse transformation from martensite to austenite is endothermic (heat absorbing reaction). For the sample annealed at 485°C (Fig. 2) the thermal

peaks were identified as two-stage transformation $A \rightarrow R$ followed by $R \rightarrow M$ on cooling. During heating, there are also sequences involving the R-phase transformation, but in this case the recorded peaks overlap.

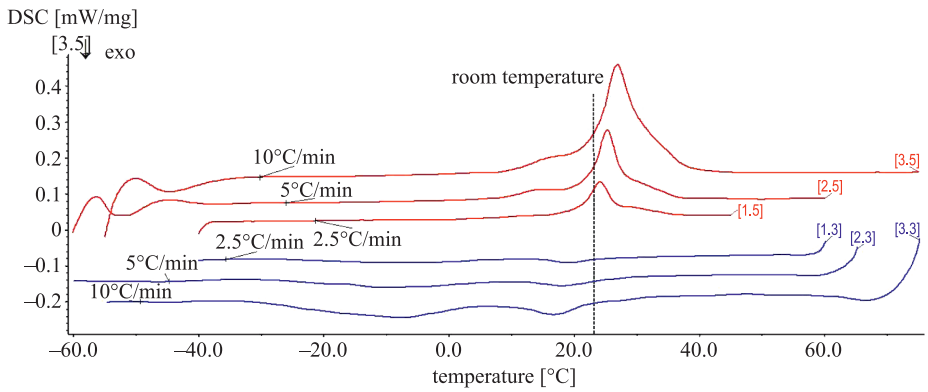

Fig. 2. DSC curves of the sample annealed at 485°C measured with different cooling/heating rate (curves for cooling are blue and those for heating are red)

On the left, the large endothermic peaks show embedded humps, which appeared to be more defined when a higher heating rate was applied. To their right, shoulders are visible, which can be explained by the fact that there are some regions where martensite is directly transformed to austenite (characterized by a large peak), but there are also other regions where martensite is transformed to austenite via the R-phase (characterized by a shoulder) (TURABI et al. 2016). The complicated shapes of the transformation peaks during heating (upright peaks) made some characteristic temperatures unreadable. The lower the cooling rate was during the upright transformation (cooling cycle), the flatter the recorded curves became. When samples were annealed between 500-585°C, distinct two-stage transformations were observed on cooling. A significant decrease in the temperature interval of M-phase is observed between annealing conducted at 485 and 500°C, while it remains almost constant for higher annealing temperatures. A qualitative variation in phase transition behaviour was seen on heating when compared to all the previous DSC curves. In this case, the occurrence of only one well-defined peak indicates that the direct transformation $M \rightarrow A$ has become more favorable. Figure 3 shows, as an example, the course of the calorimetric curves for the annealing temperature of 525°C for the different cooling/heating rates. For annealing temperatures of 500, 555 and 585°C it was similar, hence thermograms are not presented here. The SMA material after annealing in the temperature range of 485 to 525°C is a mixed structure of austenite and R-phase at room temperature (23°C), but to varying proportions, as shown by DSC experiments.

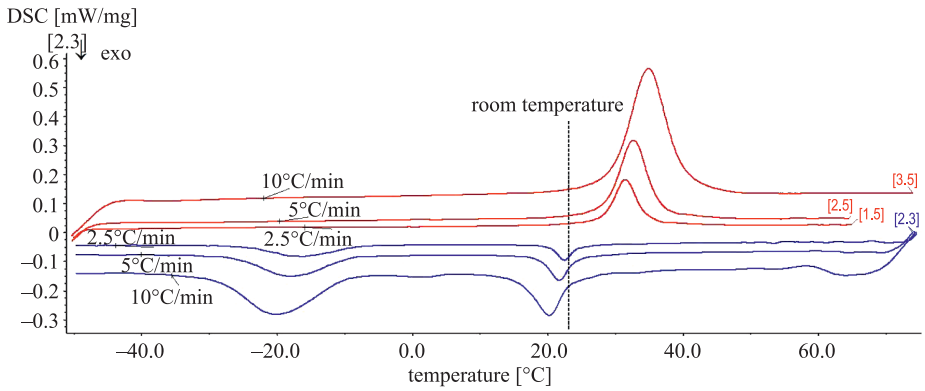

Fig. 3. DSC curves of the sample annealed at 525°C measured with different cooling/heating rate (curves for cooling are blue and those for heating are red)

During cooling, for samples annealed from 555°C up to 610°, the *R*-phase still remains which can be recognised by the presence of two separated peaks of the phase transformation. The peaks associated with the *R*-phase become more flattened and broadened, so identification of the critical temperatures is difficult on the basis of them. During the heating cycle, a pronounced shoulder develops as the annealing temperature increases, especially on the high-temperature side of the peaks (Fig. 4). It suggests that the endothermic peaks on heating represent a combined transformation of $M \rightarrow A$ and $R \rightarrow A$. After annealing at temperature raised to 635°C, the observed DSC charts on cooling and heating become too diffuse, which may indicate the multi-stage nature of the transformation (Fig. 5). While it is still possible to deduce something from the heating curves with regard to the changes in a few transformation parameters as the applied rate increases, this is hardly possible with the recorded cooling curves due to their extreme temperature expansion.

Figure 6 shows the evolution of phase transformation parameters for different cooling/heating rates after the Ni-Ti material was subjected to different annealing temperatures. After annealing at 485°C, rather all characteristic temperatures recorded during cooling show decreasing trend if the DSC cooling rate increases. It is only in the case of M_p that decrease is first observed, followed by increase in values as the scanning rate increases. During the reverse transformation, R_{sheat} decreases slightly between heating rates of 5 and 10°C/min. The A_s, A_p, A_f and A^*_f increase as the heating rate rises. Samples, which were annealed at temperatures in the range from 500 to 585°C, demonstrate decreasing of the M_s, M_p and M_f temperatures as the cooling rate increases. The R_s and R_p temperatures are almost constant at all scanning rates, except for the sample after annealing at 585°C – an increase in R_s temperature is already observed as the cooling rate increases. The finish temperatures of *R*-phase decrease with

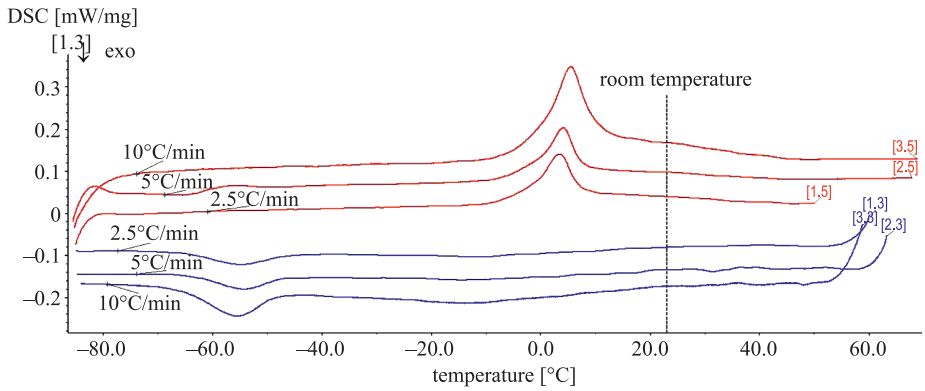

Fig. 4. DSC curves of the sample annealed at 595°C measured with different cooling/heating rate (curves for cooling are blue and those for heating are red)

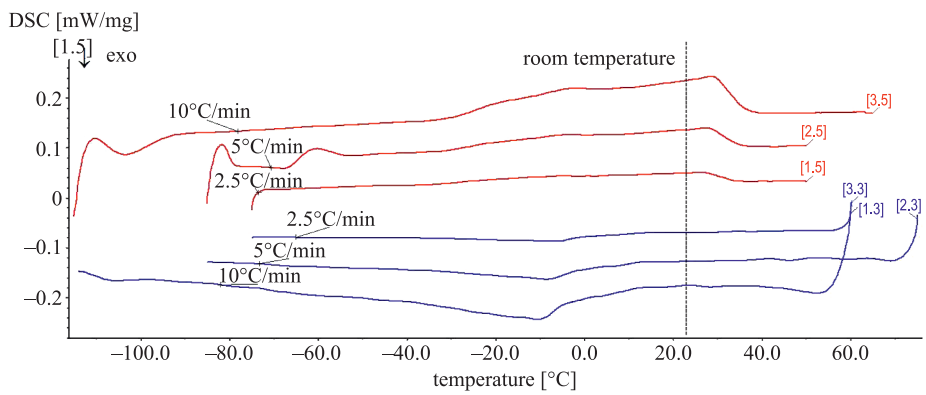

Fig. 5. DSC curves of the sample annealed at 635°C measured with different cooling/heating rate (curves for cooling are blue and those for heating are red)

increasing cooling rate. The exception is the sample annealed at 500°C, where the R_f temperature remains almost constant. After annealing at 500°C and 525°C, there is no effect of applied heating rate on the changes in A_s temperatures with respect to the reverse transformation, while at higher annealing temperatures, the higher the heating rate was, the higher the A_s temperatures were. With the heating rate from 2.5 to 10°C/min, there is an increase in A_p and A_f temperatures for samples annealed from 500°C to 585°C. The R_{sheat} temperature maintained almost the constant value as the DSC scanning rate of the heating process increased, which could be determined from the DSC curves for the sample after annealing at 585°C. For anneals carried out at 595-610°C, the M_s temperatures decrease, R_s, R_{sheat} and A_f increase, while R_p and A^*_f remain unchanged as the cooling/heating rate increases. After annealing at 595°C the temperatures of M_p, M_f, R_p, A_s and A_p remain rather constant, independently of the DSC scanning

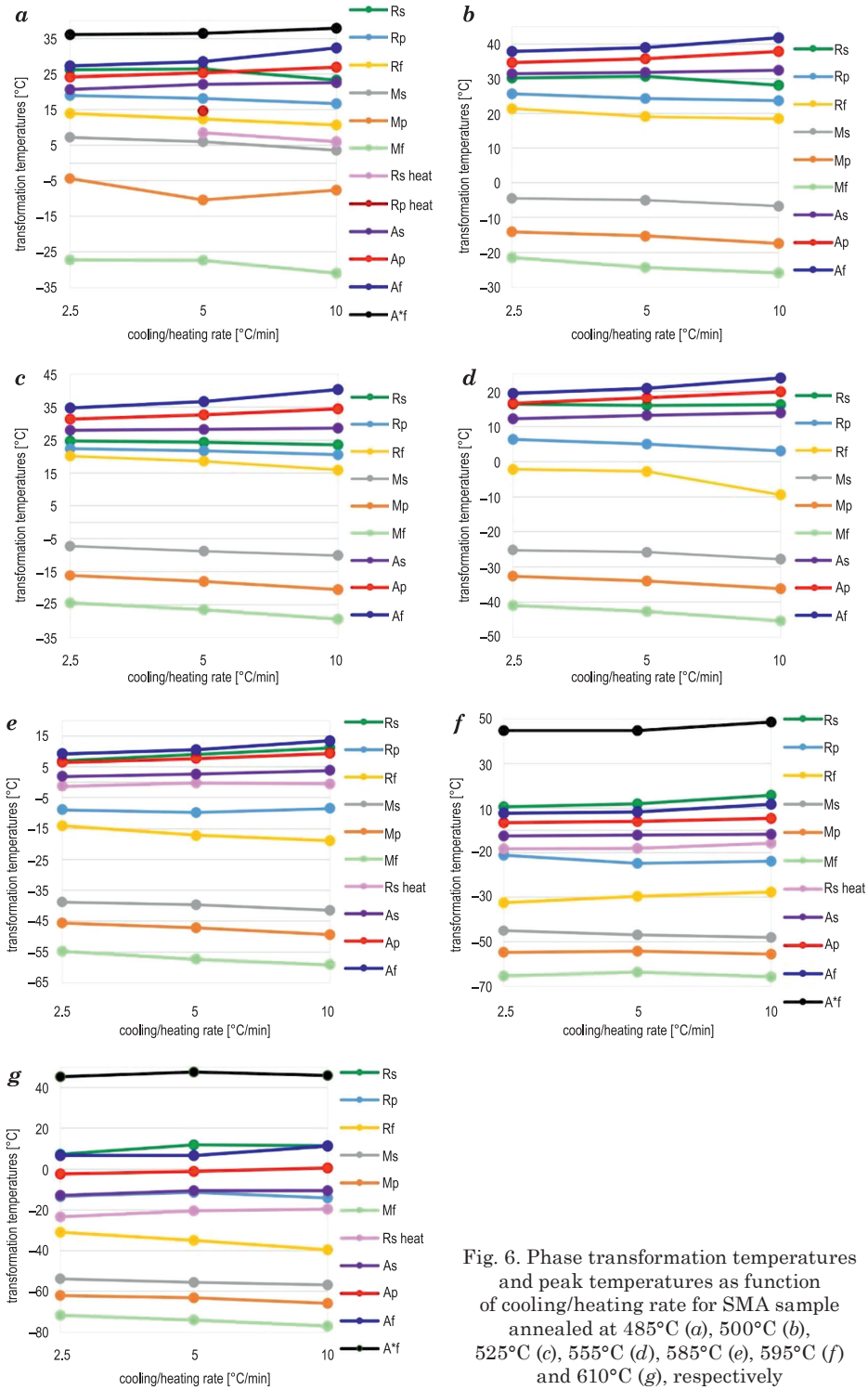

Fig. 6. Phase transformation temperatures and peak temperatures as function of cooling/heating rate for SMA sample annealed at 485°C (*a*), 500°C (*b*), 525°C (*c*), 555°C (*d*), 585°C (*e*), 595°C (*f*) and 610°C (*g*), respectively

rate. Annealing at 610°C caused the temperatures of M_p, M_f and R_f decreased slightly, while A_s and A_p increased as the cooling/heating rate increased.

According to the above mentioned experiments it can be seen that the temperature-induced transformation from one phase to another, with respect to SMA, can be monitored through the DSC method. Basing on the thermograms, it is possible to detect (among other parameters), the critical temperatures of the phase transformations, which are among the most important characteristics in terms of the target applications of SMA. In general, it was shown that, there is an influence of the cooling/heating rate performed in the DSC on the evaluated thermal characteristics of the tested material. The applied cooling/heating rates varied from 2.5 to 10°C/min. This influence is perhaps not so noticeable if one takes the states of the material after annealing conducted at different temperatures.

With regard to the individual anneals, some of the characteristic transformation temperatures were sensitive to changes in cooling/heating rates, while in case of others this effect was not observed. In general, as the cooling rate increased the temperatures associated with the start and end of the formation of the martensitic phase tended to be lower. As the heating rate increased, the temperatures associated with the beginning and end of the formation of the austenitic phase also increased. Basing on the analysis of all the transformation parameters, the R_s and R_p temperatures appeared to be the most stable while changing the scanning rate. Analysing the DSC curves obtained after different annealing of the material, only some of the phase transformation parameters showed specific changes with a change in cooling/heating rate. If trends were consistent, mostly dynamic changed.

Following annealing at selected temperatures ranging from 485°C to 635°C, changes in the shapes of the calorimetric profiles were obtained. On the basis of obtained curves and the literature review, annealing happens to modify the microstructure of SMA. The DSC profiles revealed the presence of three different phases as a function of temperature (austenite, *R*-phase and martensite). For example, the *R*-phase transformation commonly appears as an intermediate phase in most commercially available cold-worked/annealed Ni-Ti alloys. The occurrence of this phase often complicates DSC thermograms, especially when dealing with multiple overlapping stages of transformation behaviour, like in the case of Ni-Ti alloys with higher nickel content and subjected to certain types of heat treatments. It is necessary to note that not all phase transformation temperatures were obtained from the experiments due to overlapping peaks or their poorly defined signatures. In particular, this is exemplified by the DSC curves presented in Figure 5. Nevertheless, it can be seen that after annealing carried out at the highest temperature, the recorded inflection points on the calorimetric traces shift as the cooling/heating rate changes from 2.5 to 10°C/min.

In fact, DSC studies of SMA using different cooling/heating rates can be found in the published literature. On the other hand, the reasons for one or the other choice are not often discussed there. This is a quite important issue, because, as can be seen, the different DSC cooling/heating rates adopted in the studies can result in more or less differences in the definition of characteristic temperatures in SMA. Particularly, for their target application, a key requirement is to accurately determine the temperatures at which phase transformations begin and end. To be clear, this refers to cases where the transformations occur without any external stress applied. The M_s and A_f transformation temperatures (the latter being the shape recovery temperature) are particularly significant in the design of SMA applications and they are often considered in the literature and engineering practice. As these two temperatures do not usually coincide, they create a temperature hysteresis.

Many other factors can affect the quality of the DSC results (good thermal contact between the sample pan and the material sample, proper sample preparation, but also different sample weights in the same experiment). As pointed out in the study (TURABI et al. 2016), increasing the heating rate increases the temperature gradients in the sample, resulting in the reduction of the quality and resolution of the obtained signal. Higher heating/cooling rates give sharper enthalpy peaks that can be integrated more accurately, but may of them do not give accurate transformation temperatures due to thermal lag (SHAW et al. 2008). As reported in (TANG et al. 2000), if the scanning rate is greater than 10°C/min, the M_s temperature measured on cooling and the A_s on heating tend to be lower and higher than the intrinsic values, respectively. In the case of transformation heat, the slower the scan rate is, the more heat transfer can be detected. Referring again to the authors of the paper (SHAW et al. 2008), lower temperature rates reduce thermal lag, but too slow rates can make enthalpy peaks rather indistinct. Summing up, the slower the cooling/heating rate is set, the more accurate measurements can be obtained. Of course, cooling and heating rates can be individually set by the user. It may be noted that a commonly used rate in SMA testing is 10°C/min. Taking into account the aforementioned considerations, it is important to be aware that this value represents a certain trade-off.

Conclusions

This paper concerns the effect of different DSC scanning rates of the cooling/heating process on changes in the thermal phase transformation parameters of the Ni-Ti SMA following annealing at different temperatures. The DSC cooling/heating rates were 2.5; 5 and 10°C/min, respectively. The main experimental findings of this study may be summarized as following:

1. The SEM with EDS analysis confirmed the presence of nickel and titanium (Ni = 56.92 and Ti = 43.08 in wt.%) as the main elements in the investigated SMA material.

2. The annealing carried out at various selected temperatures ranging from 485 to 635°C on the supplied Ni-Ti SMA changed the shape of the resulting DSC curves, especially the endothermic ones.

3. In general, experiments demonstrated that there is an influence of the cooling/heating rate performed in the DSC on the evaluated thermal characteristics of the SMA tested material. This influence is perhaps not so clear if one takes the states of the material after annealing conducted at different temperatures. In this case, only some of the phase transformation temperatures showed changes with a change in scanning rate. If temperature trends were consistent, mostly dynamic changed. Disregarding the effect of the preceding annealing, some of the characteristic transformation temperatures were sensitive to changes at cooling/heating rates, while for the other temperatures this effect was rather not observed. Thus, as the cooling rate increased from 2.5 to 10°C/min, the temperatures associated with the beginning and end of the formation of the martensitic phase tended to be lower. As the heating rate increased, the temperatures associated with the beginning and end of the austenitic phase formation also increased. Of all the transformation parameters studied, those associated with the transformation to the *R*-phase during cooling (specifically the R_s and R_p temperatures) appeared to be the most stable with scanning rate changing. It should be noted that not all the phase transformation parameters were identified from the experiments due to overlapping peaks or extreme broadening of the thermograms. This problem especially occurred after annealing at the highest temperature.

4. The literature indicates that the slower the cooling/heating rate in the DSC technique is, the more accurate measurements can be obtained. Particularly, in the case of target SMA applications, it is important to accurately determine the temperatures at which phase transformations begin and end. A commonly used rate in SMA testing is 10°C/min, but this represents some kind of a compromise in relation to the experimental data obtained. If necessary, this value can always be changed to a smaller one, if accurate data of the characteristic temperatures of the transformations in the SMA are expected, but not in the shortest time.

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