

Chapter 3

Introduction to Shape Memory Alloy Technology

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3.1 Basics of the Shape Memory Effect

Alloys with the ability to revert to their programmed shape through thermal activation are known as shape memory alloys (SMA). The diffusion-less phase transformation of the low-temperature phase (martensite) to the high-temperature phase (austenite) is the basic characteristic of shape memory alloys. During this martensitic transformation, neither the chemical composition of the alloy nor the collocation of atoms changes. The conversion from the austenite phase to the martensitic phase starts below martensite start temperature (M_s). By way of further sub-cooling, more martensite crystals form in the structure. From an automation perspective, the phase transformation effect is activated by channeling thermal energy into the system. The phase transformation is reversible and not time-dependent, as it only distorts the atomic lattice without causing long-range migration of atoms [1]. Functionally speaking, the phase transformation is an application of stress to both the material and the temperature, leaving the conversion rate unaffected by the duration of the two acting factors. The stress needed to induce phase transformation is dependent on the M_s temperatures and on the ambient temperature. Therefore, the M_s temperature increases in equal measure with the applied mechanical stress. In contrast to hardening processes in steels, only view-irreversible defects may be generated in SMA so that the reversibility of the conversion for the maximum number of cycles is maintained [2]. Figure 3.1 illustrates the phase

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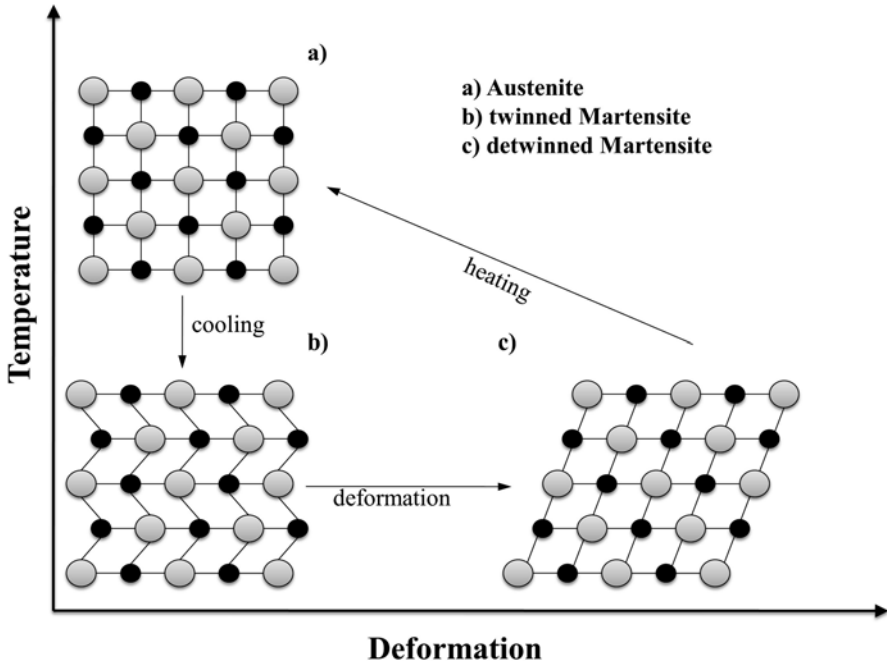


Fig. 3.1 The martensitic transformation in shape memory alloys

transformation schematically. By cooling from austenite (a) to martensite, the twinned martensite is formed (b), and, through displacement of the interfaces, undergoes deformation to (c).

The twinned condition occurs when within the martensite grid several martensite groups arise with different orientations. Due to the material deformation, these martensite groups align in the preferred direction. This so-called detwinning procedure assumes highly mobile martensite interfaces, set by the acting mechanical stress that stretches the material. The atomic grid transforms to the austenitic structure when heated. The phase transformation does not occur at a specific temperature but within a temperature interval. Figure 3.2 shows the phase transformation degree (ξ_A) as a material temperature function.

The transformation process can be described by four characteristic temperatures (also called phase transition temperatures or PTT), which can be determined from diagrams using a tangent method. With a constant mechanical load, the martensitic transformation begins at the so-called austenite start temperature (A_S). Monoclinic austenite is converted to body-centered cubic austenite. The transformation is complete when it reaches the austenite finish temperature (A_F) and the material is fully austenitic. The reverse transformation starts at the martensite start temperature, which is significantly lower in binary nickel-titanium shape memory alloys than the A_F temperature, and ends at the martensite finish temperature, which is

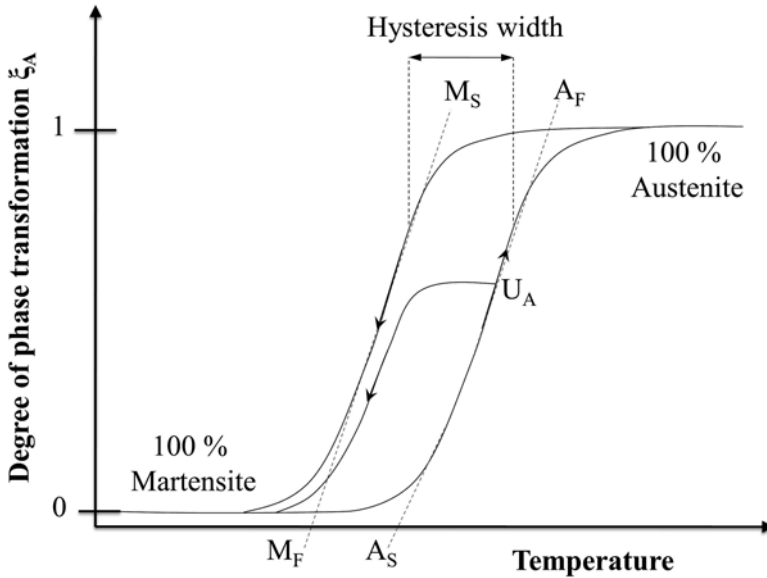


Fig. 3.2 The martensitic transformation in shape memory alloys over temperature

also significantly lower than the A_S temperature. Thus, the phase transformation behavior shows a thermal hysteresis. The hysteresis is a result of internal losses, such as friction and spontaneous relaxation at the atomic level during the structural transformation [3]. Hysteresis properties, such as shape and width, depend on multiple factors, for example alloy composition, thermo-mechanical pre-treatments and mechanical load. During cooling, the structure transforms gradually from individual areas, and returns to the formation of differently oriented martensite groups (twinned structure). In cases where the phase transformation is not complete, the reverse transformation begins in a mixed phase, consisting of martensitic and austenitic phases. The reverse transformation then unfolds as more of a curved branch, starting at the termination point (U_A) instead.

3.2 Shape Memory Effects

An overview of utilizable SMA effects is shown in Fig. 3.3, including examples of application. While pseudo-elastic SMA are well-known in medical applications like stents and highly flexible consumer products like clothing, thermal SMA are increasingly being used in thermal clamps and electrical actuator applications as well. A more detailed explanation of individual effects is presented throughout the following sections.

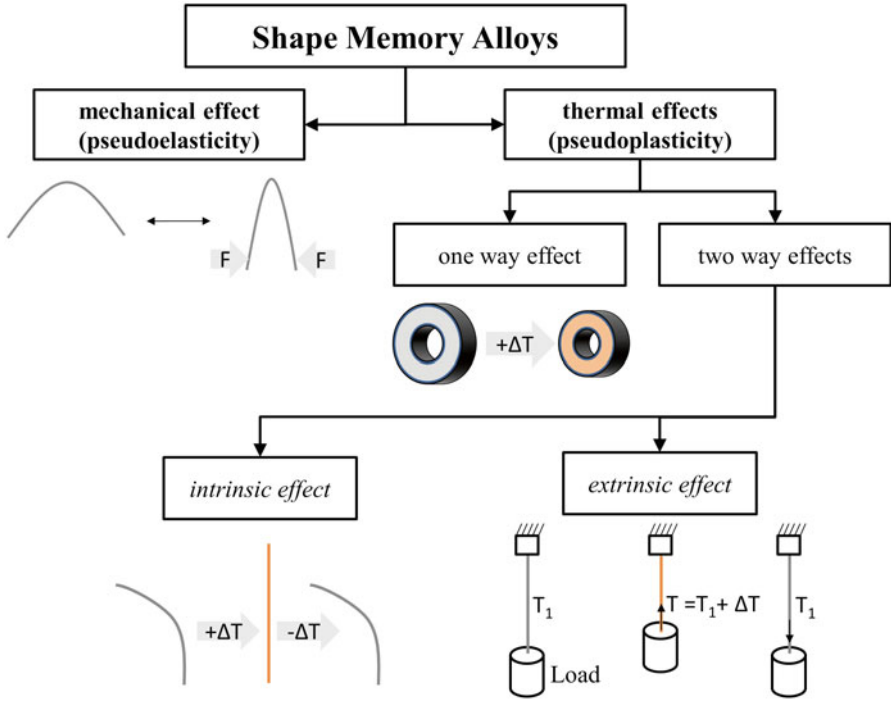


Fig. 3.3 Overview of shape memory alloy effects

3.2.1 The One-Way Effect

SMA elements using the one-way effect can be deformed. After subsequent heating and without applying any extrinsic mechanical load, the element transforms back into its previous shape. Considering the deformation as a function of mechanical stress and temperature, as shown in Fig. 3.4, helps to understand the effect better. The application of stress results in a pseudo-plastic deformation. This deformation appears to be plastic, while the SMA element is relieved. This is called “pseudo” plastic, because it can be reset by shape memory transformation, which is caused by thermal activation. When heated, the austenite start temperature (A_s) is reached. Upon further heating, up to the austenite finish temperature A_f , the material transforms into the austenitic state completely. All atoms adopt the structure of the body-centered cubic grid, which undoes their deformation. Now, cooling occurs down to the initial temperature, at which point the structure transforms back into twinned martensite. During the cooling process, however, the macroscopic structure of the element is not deformed again. This effect causes the material to “remember” exclusively its high temperature form. The one-way effect is suited for applications that generate high forces, or for on-off actuating movements in the lifecycle of

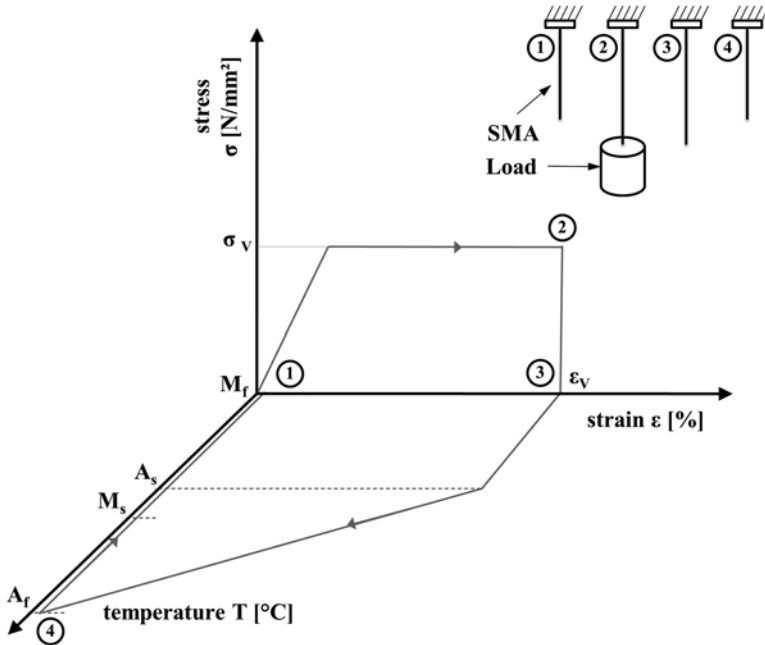


Fig. 3.4 Characteristic of the SMA one-way effect

technical systems. Industrial applications mainly stretch across the field of connection technology (e.g. shrink sleeves) and in space (e.g. one-time opening of flaps or solar sails in satellites).

3.2.2 The Extrinsic Two-Way Effect

The extrinsic two-way effect describes shape memory elements that are continuously under mechanical preload. Figure 2.3 shows the extrinsic two-way principle exemplified by a shape memory wire. When the wire is heated, it contracts and lifts the attached weight. Figure 3.5 additionally illustrates the schematic stress–strain–temperature diagram of the effect with marked reference points that connect back to the main diagram. The unoriented martensite (1) gets deformed by external stress until it reaches an apparent yield point (2). At this point, the martensite is detwinned. This pseudo-plastic deformation, which remains intact even after the SMA element is released, is called pseudo-plastic elongation. When the SMA element is heated, the phase transformation starts at A_s temperature and the martensite structure is transformed into austenite (3). The nature of this transformation can be attributed to the orientation relationship between the austenite and martensite grid. Because of the continuously applied mechanical stress, for example by an external load, the material is stretched directly into the detwinned martensitic state during the cooling process

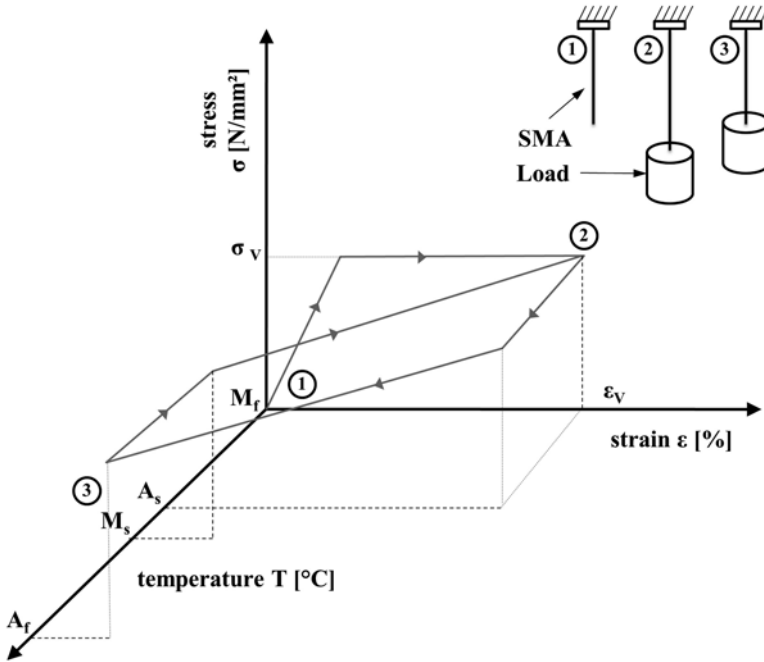


Fig. 3.5 Characteristic of the SMA extrinsic two-way effect

(2). This effect is suitable for cyclic operations in valve drives. The phase transformation from martensitic to austenitic state can be triggered via Joule heat generation in order to contract the SMA wire by electrical current, as presented in the main diagram. The shape memory alloy exerts a mechanical reaction to the applied stress level. The product is a resulting force, or a resulting stroke, which can be used for mechanical work. In the actuator application, SMA can normally be switched between states (2) and (3). It is also possible to target intermediate positions along the hysteresis curve and maintain those by means of control strategies. This allows for the use of SMA actuators as proportional drives.

3.2.3 The Intrinsic Two-Way Effect

The intrinsic two-way effect describes shape memory alloys that show reversible transformation behavior both during heating and cooling. This behavior is introduced into the material by thermo-mechanical treatment (training). The effect stabilizes during the training cycles in the preferred orientations of the martensite. This results in residual stress levels in the SMA element, which apply the required minimum stress to reset the SMA element by itself. During the training process, the intrinsic two-way SMA element is deformed to its real yield strength in both the

austenitic and the martensitic state. Due to the plastic deformation of the material, dislocations and existing grid defects in the material are formed and stabilized. This, in turn, ensures residual stresses, which do not get degraded, not even in later phase transitions. After about 20–30 training cycles, a stable intrinsic two-way effect can be observed [2]. Due to lack of stability and a much smaller general effect compared to the extrinsic two-way effect, the intrinsic two-way effect is not used in actuator applications.

3.2.4 The Pseudo-Elastic Effect

Pseudo-elastic (or super-elastic) shape memory alloys make up the largest part of the shape memory technology market. In contrast to the one-way and two-way effect, the pseudo-elastic effect can be achieved mechanically at a constant temperature. This effect occurs at operating temperatures above the martensitic start temperature (M_s). Currently, obtainable pseudo-elastic shape memory alloys are usually completely austenitic at room temperature. Due to external mechanical stresses, the austenitic atomic grid gets deformed and a directly stress-induced detwinned martensite grid arises. Since this detwinned martensite is not thermodynamically stable, the grid retransforms into an austenite state upon mechanical relief. Figure 3.6 shows a

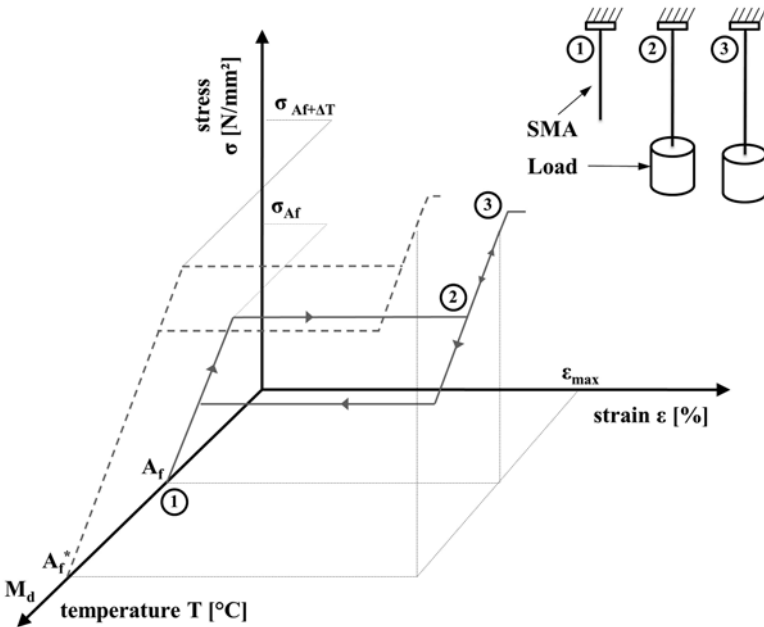


Fig. 3.6 Outline of the SMA pseudo-elastic effect

pseudo-elastic shape memory wire as main figure as well as a functional diagram. If loads are applied to pseudo-elastic elements, the material becomes elongated. Upon alloy relief, the pseudo-elastic wire reassumes its original shape. Figure 3.6 also shows a stress–strain diagram of a pseudo-elastic alloy with reference points to the main diagram.

If the material is mechanically loaded, it reaches a state in which the material deformation increases at nearly constant mechanical stress, namely after an approximately linear stress–strain deformation has occurred. This so-called stress plateau region of the curve allows for highly flexible deformations. When the stress-induced transformation is complete, the stress–strain curve rises again in a linear fashion. The exact amount of plateau stress depends on material temperature. This can be altered by thermo-mechanical behavior. With rising material temperatures, more mechanical energy must be introduced in order to initiate a phase transformation. Overall, the elongation of pseudo-elastic shape memory alloys can reach up to 8 % of their initial length. In comparison, the reversible spring-steel elastic yield strength lies approximately at 0.5 %.

3.3 Shape Memory Alloy Types

All alloy systems with a thermo-elastic martensitic transformation are conceivable, in principle, for the use as a shape memory alloys. Due to adverse mechanical properties, however, many of these alloys are of minor technical importance. Comparatively, the best shape memory alloys for cyclic, medical and actuator applications are NiTi alloys followed by Cu-based or Fe-based alloys, which are suitable only for industrial applications. Table 3.1 compares the various alloys with regards

Table 3.1 Overview of functional properties of different shape memory alloys [4]

Properties	NiTi	CuZnAl	CuAlNi	FeNiCoTi	FeMnSi
Transformation temperatures [°C]	–50...100	–100...100	80...200	–150...300	50...250
Max. one-way effect [%]	8	5	5	1,5	2,0
Max. two-way effect [%]	6	1	1	0,5	0,3
Max. pseudo-elasticity [%]	8	2	2	1,5	1,5
Handicaps	Poor machinability, high costs	Segregation, coarse grain formation	Poor cold formability	Stability and effect of low	Stability and effect of low
Advantages	Max. effects, highest stability, corrosion-resistant	Low cost, good formability	Low cost	Low cost, good formability	Low cost, good formability

to size of their utilizable shape memory effects, which are, in descending order, NiTi-based, Cu-based, and Fe-based alloys.

The material demands for industrial applications of SMA (such as valves) are as follows:

- High pseudo-plastic strain.
- Low deformation resistance of the martensite.
- High strength of the austenite.
- Good machinability of the material.

Additionally, the requirements for controlled elements in valve applications can be satisfied by the following demands:

- Good long-term stability of the shape memory effect.
- High conversion rate of electrical energy into mechanical work.
- High transition temperatures.
- Low thermal hysteresis for thermal-proportional control valves.
- Wide thermal hysteresis for thermal safety valves.
- Significant change of electrical resistance for electrical-proportional control valves.

To meet these requirements, and to use an actuator as efficiently as possible, only the use of NiTi alloys is recommended. This is nowadays encouraged by the general availability of NiTi materials. Besides the greatest shape memory effects, they also offer long-term stability and a long lifetime. With NiTi alloys, work cycles up to a million can be realized, however, to achieve such a high lifetime performance, a specific and detailed design of the shape memory components is essential [5].

3.3.1 Binary Nickel-Titanium Alloys

The most commonly used shape memory alloys are binary nickel-titanium alloys. The alloy system exhibits a high dependence on phase transition temperatures of its chemical composition. Thus, Fig. 3.7 represents the change in the martensite start temperature (M_s) when nickel content is added to the function (listed in atomic percent). When the chemical composition gets modified, for example, by adding 0.1 at.% of nickel content, the M_s temperature shifts by about 10 Kelvin. What is striking is the characteristic inflection point at about 50.5 at.% nickel. A reduction of the nickel content, and thus a substitution of the more expensive titanium, below this value affects only small increases in the phase transformation temperatures. This leads to high demands for the equipment used in the melting process. Sometimes the material needs to be re-melted several times to ensure a homogeneous composition of the ingot.

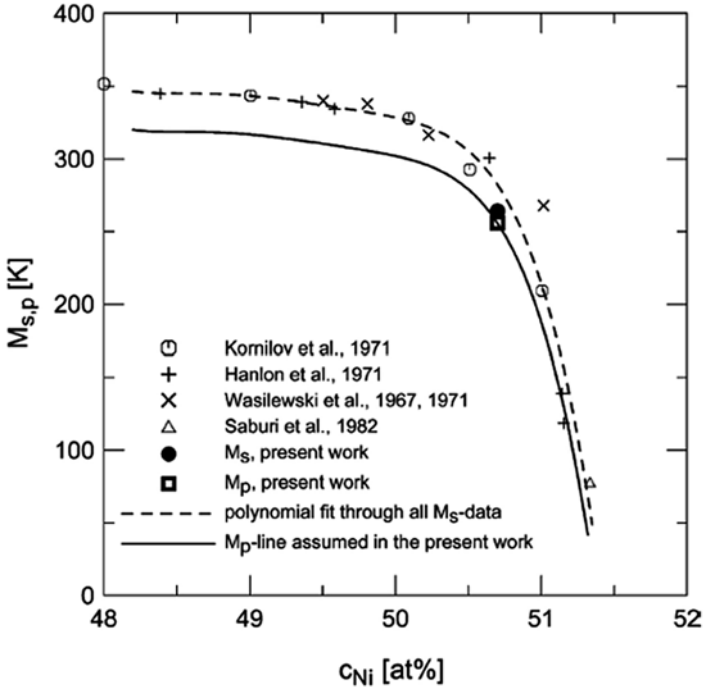


Fig. 3.7 Martensite start temperature as a function of the nickel component [6]

Table 3.2 The effect of ternary and quaternary elements in NiTi-based SMA [4]

Properties	C	O	N	H	Cu	Cr	Co	Fe	V	Nb
Transformation temperatures	↓	↓	↓	↓	→	↓	↓	↓	↓	→
Max. applied stress	↑	↑	↑	↑	↓	↑	↑	↑	↑	↑
Brittleness	↓	↓	↓	↓	→	↓	↓	↑	↓	→

3.3.2 Ternary and Quaternary Nickel-Titanium Alloys

With the addition of other elements to nickel-titanium alloys, the shape memory effect properties—such as phase transition temperature (PTT), hysteresis, and mechanical properties like strength and impact brittleness—can be modified. Table 3.2 depicts the qualitative influence of alloying elements on NiTi-alloys.

In addition, there are different ternary alloys, which have been researched and market-tested, containing elements such as hafnium, copper, niobium, palladium, platinum, or zirconium. For actuators, the NiTiCu alloy is most relevant. Available NiTiCu alloys include about 10 at.% of copper [2] and are used as substitution for a correspondingly high atomic percent share of nickel. NiTiCu alloys, however, exhibit considerable differences compared to binary NiTi alloys. On the one hand,

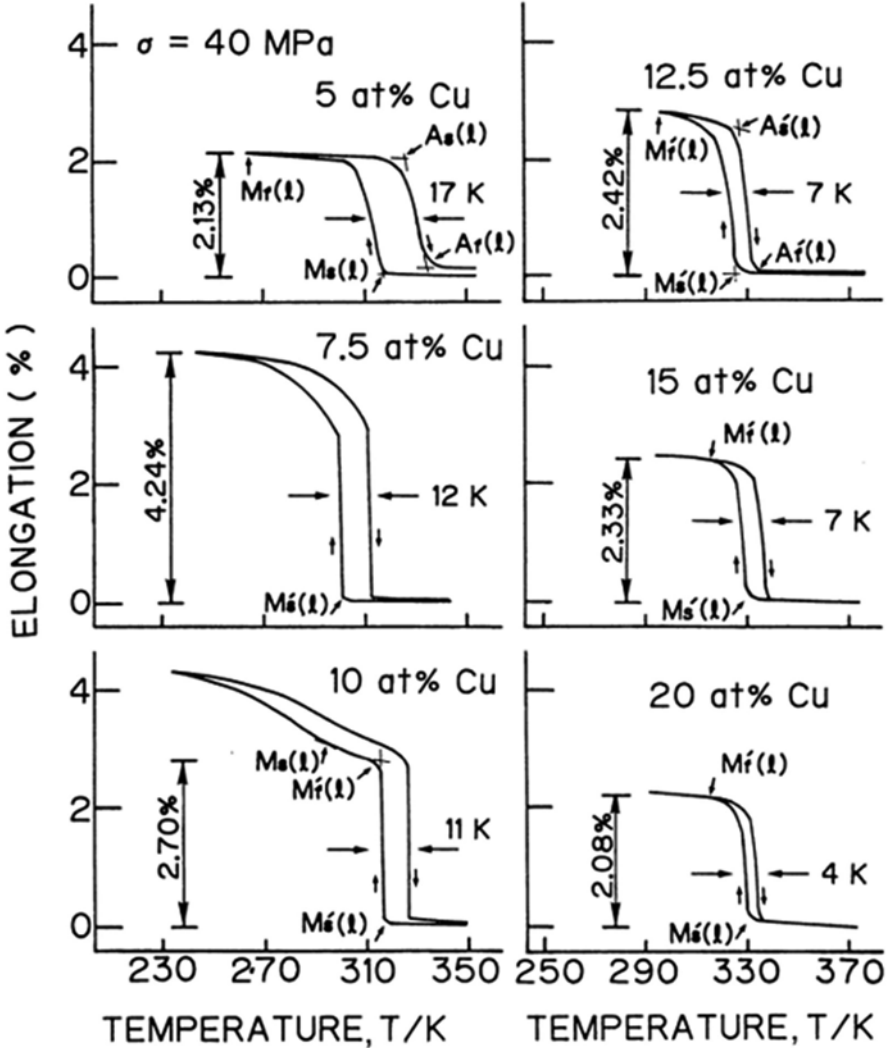


Fig. 3.8 Transformation behavior of NiTiCu depending on the amount of copper [7]

the transformation hysteresis is considerably narrower than with binary alloys, but only at the cost of reducing the mechanical output potential. Thus, NiTiCu alloys can only permanently generate about half as high mechanical tensile stresses in comparison to NiTi alloys. Figure 3.8 shows the variation of material hysteresis NiTiCu of alloys as a function over copper content. In this figure, the elongation is reset by thermal heating. The gradient of the hysteresis can be significantly higher, and can consist of several gradient sections as displayed in Fig. 3.8 (for example at 10 % for Cu). A striking advantage of this alloy type is better fatigue performance

and the linear change of electrical characteristics during electrical actuation, compared to NiTi. Unfortunately, the application field of NiTiCu alloys is limited to the lower ambient temperature field considering electrical applications. Since the M_F temperature can only reach around 50 °C, the system will not retransform completely when the ambient temperatures exceed this limit. Hence, NiTiCu alloys are not suitable for automotives, aircrafts, and many other industrial applications with ambient thermal fields higher than 50 °C.

Interestingly, ternary NiTi (X) alloys, and in particular NiTi quaternary (X, Y) alloys, are known as high temperature alloys. In these alloys, the martensite finish temperature (correspondingly also the martensite start temperature) reaches over 80 °C. Industrial users and potential industrial users of shape memory alloys require these types. Common application fields of actuators, for example in the automotive and aerospace industry, require full system functionality at 85 °C. This equals an electric system that retransforms completely into the martensitic state at this critical temperature. Hence, the M_F temperature must be higher than 85 °C.

A similarly high system dynamic is required due to the cooling rate, which demands the highest possible transformation temperature.

The alloying additives, which increase the phase transformation temperatures, are usually elements like platinum, palladium or hafnium, and in some cases also tantalum–aluminum combinations. Material-scientific research worldwide focuses on the development of such alloys [8]. The predominant objectives are to increase the martensite temperatures above 100 °C and to reduce the temperature hysteresis, with the intention to ensure that at a room temperature of 85 °C, the PTT can be increased to about 200 °C, will say to a high potential between the actuator temperature and the ambient temperature, in order to increase the retransformation speed as well. During cooling, an extensive thermal-potential difference leads to a much faster retransformation as with NiTi alloys, whose M_s temperature surpasses 80 °C only slightly. Figure 3.9 shows the martensite start temperature of a ternary NiTiPd alloy as a function over palladium content. The dependence of the M_s temperature significantly expresses itself linearly from a share of 15 %. In Fig. 3.10, a dynamic test result of a NiTiPd is shown over time at an ambient temperature of 85 °C (SMA wire compared to binary NiTi alloys). At equivalent mechanical stress, the brittle NiTiPd material can be elongated only about 1.5 %. NiTi could be deformed to up to 4.4 % in this experiment.

Both samples (diameter: 0.2 mm, length: 120 mm) are activated by a heating current of 1 A for a heating time of 2 s. Comparing the two experiments shows that the NiTiPd alloy transforms more slowly. This is due to the longer necessary heating time to reach the A_F temperature, which is higher compared to the conventional NiTi at about 100 °C. Taking cooling into consideration, the figure also shows that a NiTiPd alloy retransforms significantly faster than a binary NiTi sample.

However, the mechanical properties of this material allow only for small distortions, and therefore further technological breakthroughs are needed to achieve proper usability. Ultimately, the use of this material in the automotive and automation industry is questionable from an economic perspective. Since the price of palladium equals 1,500 times the price of nickel and titanium, cost-per-kg for high temperature

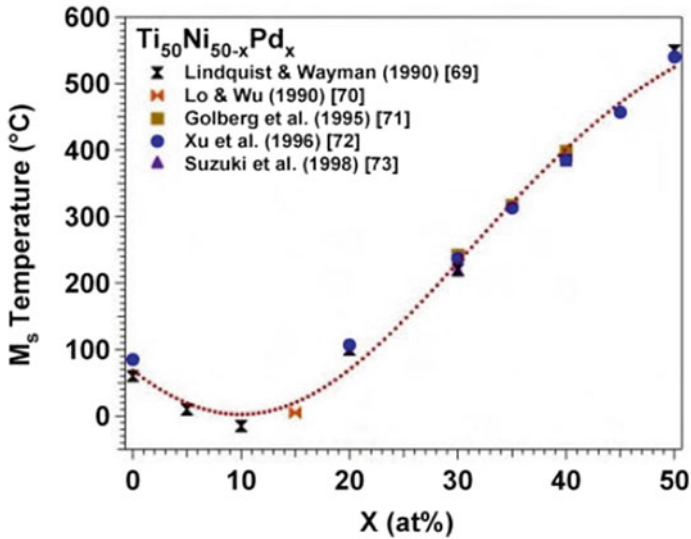


Fig. 3.9 PTT dependence of palladium content in NiTiPd alloy [8]

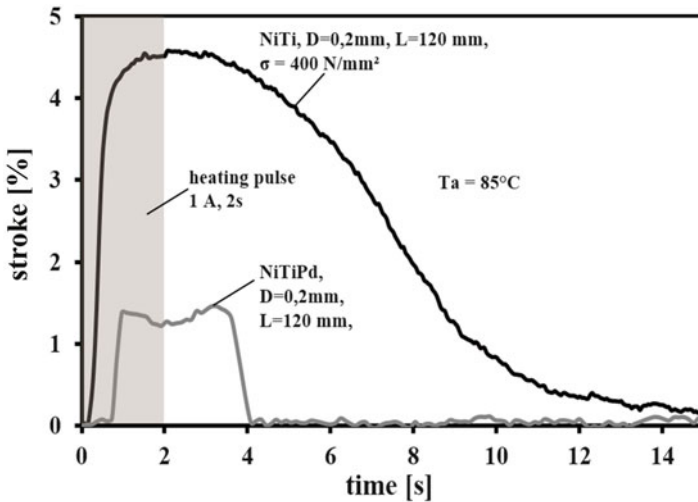


Fig. 3.10 Comparison of dynamic properties of NiTi and NiTiPd alloys in comparable conditions

shape memory alloys with a 30 % share of palladium would economically not be feasible for many industrial applications. Hope is placed on NiTaAl alloys [9], because this alloy combination is more economical, and thus production has significant advantages over NiTiPd, NiTiHf and NiTiPt systems at high-phase transition temperatures (approx. 130 ° C).

3.3.3 R-Phase NiTi Alloys

R-phase transformation (R) precedes the transformation from austenite (B2 resp. A-phase) to martensite (B19 resp. M-phase) in thermo-mechanically treated near-equiatomic NiTi alloys, in Ni-rich NiTi alloys aged at an appropriate temperature between 300 °C and 550 °C, and also in ternary NiTiFe or NiTiAl alloys [10]. The appearance of R-phase transformation can be explained through lattice defects in microstructures such as dislocations or precipitations [11]. In contrast to transformation from martensite to austenite (up to 8 %), transformation from austenite to R-phase yields a small shape change (up to 1 %) and generates less power. The typical temperature hysteresis during transformation from austenite to the R-phase is very small (2–5 K) compared to the transformation from austenite to martensite (20–70 K) [12]. Figure 3.11 compares the presented transformations using a wire actuator as example.

The development of shape memory materials and the handling of actuators based on the R-phase transformation are very complicated. Factors influencing the R-phase transformation negatively are: false material development (e.g. thermo-mechanical pre-treatment), incorrect actuator design (e.g. false shear strain) or misuse during the production (e.g. overstress). Figure 3.11 shows important influences on selected properties of R-phase actuators for Ni-rich NiTi alloys. Rising values of some influences cause different and sometimes opposite effects. For example, the temperature transformation width and the transformation temperature R_S decrease with increasing annealing temperature. In contrast to this effect, the transformation temperature R_S increases with increasing annealing time.

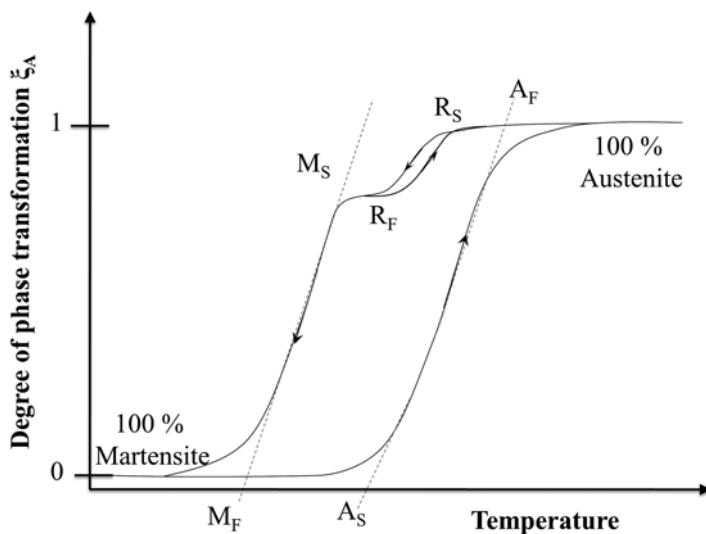


Fig. 3.11 The characteristics of a pre-stressed SMA with R-phase transformation

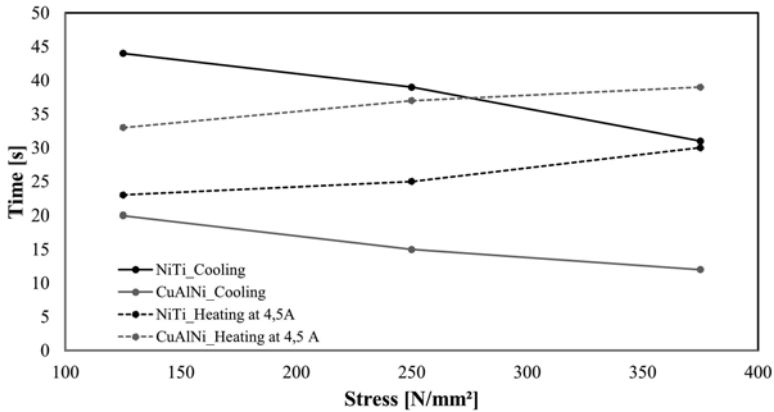


Fig. 3.12 Comparison of dynamic properties of CuAlNi alloy to binary NiTi alloy in comparable testing conditions

3.3.4 Copper-Based Alloys

The best-known copper-based alloys are CuZnAl and CuAlNi. They cannot be used as implant materials due to their chemical composition and are therefore suitable only for industrial purposes. While CuZnAl enables only low-phase transformation temperatures (for industrial applications), CuAlNi reaches in certain chemical compositions even the high temperature needs of the automotive industry. Alloys containing a weight percentage of about 12 % aluminum and 5 % nickel have an M_F temperature of about 120 °C. Mechanical characterizations further show reversible and high mechanical stresses [13]. The maximal deformation (also contraction) can reach 8 % as well as high mechanical stresses of about 350 N/mm². A major disadvantage is the processing ability and the brittle behavior of these materials. The wire drawing process is sometimes not suitable for these materials, which is why alternative design methods are used. Regarding actuator activation, more problems are noticeable. Due to the low electrical resistance of copper, high temperature copper-based SMA need much higher electrical activation currents than comparable NiTi elements (Fig. 3.12).

3.4 Manufacturing of Shape Memory Alloys

In order to understand the influences on SMA material parameters, it is sometimes necessary for product developers to understand the production processes of the material itself. In this chapter, a brief summary of the production process from melt to an SMA round wire is given. For detailed data on production processes of SMA, [3] offers detailed explanations.

At the beginning, an ingot has to be produced out of pure nickel and pure titanium. A common method for melting NiTi shape memory alloys is vacuum induction melting, which is used to produce the SMA ingot. Due to the high sensibility of phase transformation temperatures to the Ni-ratio, as discussed in Fig. 3.6, the quantities of used materials have to be weighed accurately. Figure 3.13a shows primarily the melting process. Through high frequency induction, heated raw material components melt in a vacuum or inert gas. The mold is stirred and solved, which leads to perfect homogeneity of the material. Afterwards, the liquid is molded into an ingot form. After cooling down, the ingot can be extracted from the form and processed in the hot forging process (b).

The critical step in SMA production is the thermal treatment which has significant influence on plateau stress levels and transformation temperatures. Alloys which are used commonly for SMA electric actuation are often programmed by the SMA material suppliers for optimal behavior.

However, a cold-worked SMA material has to be programmed for its optimal behavior. Figure 3.14 shows in the left part the major influence factors on the heat treatment. All heat treatments have a connection to the SMA element's dimensions and its volume/mass which has to be heated. The duration of the heat treatment has also influences on the SMA characteristics as well as the treatment temperature. Also the atmosphere has significant influences, mainly on the surface of the SMA element (relevant for medical applications).

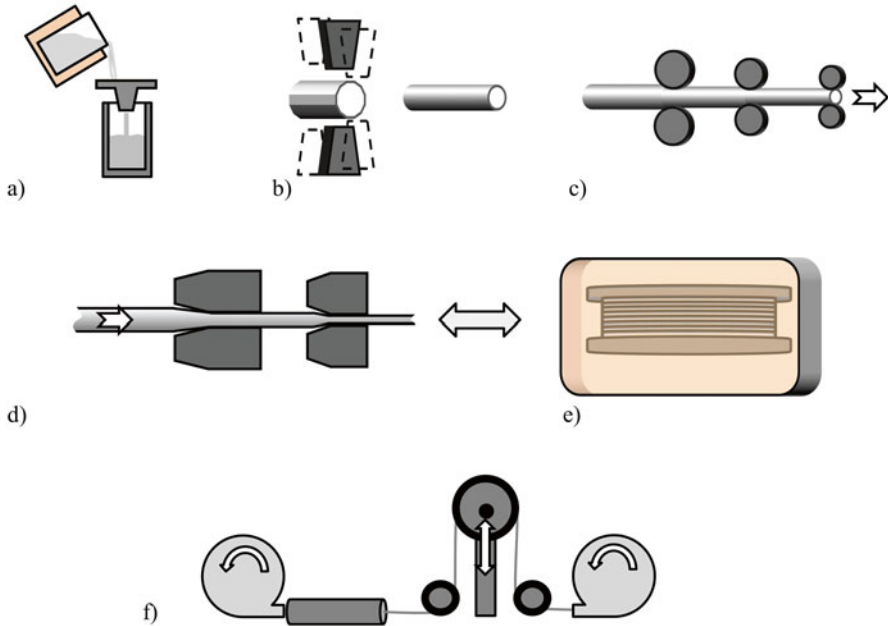


Fig. 3.13 The production process of SMA wires in various steps: **a)** vacuum-induced melting of NiTi, **b)** round forging in hot state, **c)** hot rolling, **d)** wire drawing, **e)** thermal treatment, **f)** thermo-mechanical training

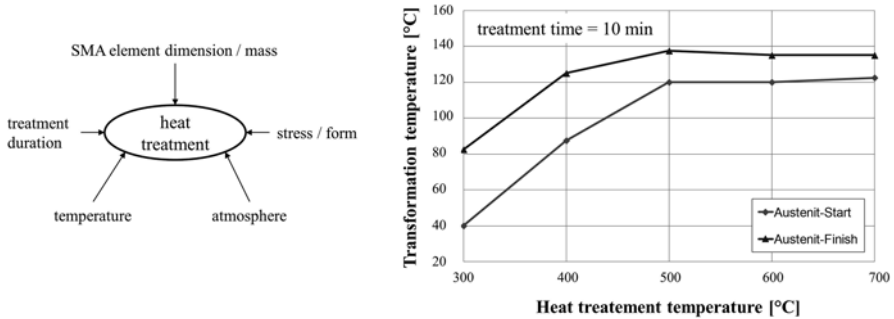


Fig. 3.14 Major influences on SMA heat treatment (left), experimental result of thermal heat treatment (right)

Normally, SMA straight wire elements are treated without stress, but also the imprint of the shape can be used by the utilization of a tool. In such case, the SMA element is heat-treated at a relative small mechanical stress level.

In the right part of Fig. 3.14, a result of heat treatment of a NiTi (54,8 wt. % Ni) SMA wire is presented. The austenitic temperatures increase with heat treatment temperatures up to 138 °C. The experimental validation has been made at stress levels higher than 350 N/mm².

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