



The Effect of Long-Term Heat Treatment on the Thermomechanical Behavior of NiTi Shape Memory Alloys in Defense and Aerospace Applications

Savaş DİLİBAL¹

Abstract

There has been a rapidly growing interest in the use of shape memory alloys (SMAs) in defense and aerospace applications. Shape memory alloys that are well known to exhibit a highly non-linear thermoelastic response involving such unique phenomena as superelasticity and shape memory effect. These fascinating phenomena are highly dependent on the chemical composition and heat treatment applied to the material. Among shape memory alloys, NiTi SMAs are potential candidate for many defense and aerospace applications. NiTi SMA based actuators offer many functional advantages over their conventional counterparts providing higher strokes, reduced weight, smaller size, and simple mechanism. In addition to the actuation, NiTi SMA based composite structures provide self-healing, sensing, and damping for the future composite systems. Besides the chemical composition, the power-to-weight ratio and the operation cost, the operational temperature range should also be carefully considered. This work is focused on investigating the effect of the long-term heat treatment on the thermomechanical response in NiTi SMA for the purpose of shifting its martensitic transformation temperature to the desired operational temperature range. The fundamental thermomechanical behavior of SMAs explained. Defense and aerospace applications of the SMAs are presented. The state of the art technology for the SMAs for the future potential defense and aerospace applications are also stated in the conclusion.

Keywords: *Shape memory alloys, shape memory effect, superelasticity, thermomechanical behavior.*

¹ Yazışma adresi: Yrd.Doç.Dr., İstanbul Gedik Üniversitesi, Mühendislik Fakültesi, Fen Bilimleri Enstitüsü, Savunma Teknolojileri Programı
savas.dilibal@gedik.edu.tr

Savunma ve Havacılık Uygulamalarında Uzun Süreli Isıl İşlemin NiTi Şekil Bellekli Alaşımların Termomekanik Davranışına Etkisi

Öz

Şekil bellekli alaşımların savunma ve havacılık uygulamalarında kullanılması konusunda artan bir ilgi bulunmaktadır. Süperelastik ve şekil bellek özelliklerine sahip olan şekil bellekli alaşımlar doğrusal olmayan termoelastik davranış gösterirler. Alaşımların bu etkileyici özellikleri yüksek oranda malzemeye uygulanan ısı işlem ve kompozisyona bağlılık gösterir. Şekil bellekli alaşımlar içerisinde, NiTi alaşımlar savunma ve havacılık alanlarında kullanım için potansiyel adaydır. NiTi şekil bellekli alaşım esaslı eyleyiciler geleneksel eyleyicilere kıyasla fonksiyonel olmaları yanında yüksek strok, düşük ağırlık, küçük hacim ve basit çalışma mekanizmasına sahiptirler. Eyleyici hareketine ek olarak NiTi şekil bellekli alaşım esaslı kompozit sistemler geleceğin kompozit sistemleri için kendi kendini yenileme, algılama ve sönümlenme gibi özellikler sağlarlar. Kimyasal kompozisyon, güç-ağırlık oranı ve çalışma maliyeti yanında çalışma sıcaklık aralığının da çok dikkatli bir şekilde değerlendirilmesi gerekmektedir. Kullanılan şekil bellekli alaşımın istenilen martenzitik/östenitik dönüşüm sıcaklık aralığına kullanılabilmesi için birçok farklı yöntem denenmiştir. Bu yöntemlerden bir tanesi de uygulanacak ısı işlemler vasıtasıyla NiTi şekil bellekli alaşımların servis sıcaklık aralığına en uygun faz dönüşüm sıcaklık aralığına alaşımın çekilmesidir. Bu çalışmada, NiTi şekil bellekli alaşımların östenit/martenzitik dönüşüm sıcaklıklarının istenilen uygulama sıcaklık aralığına kaydırılması amacıyla, uzun süreli ısı işlemlerin NiTi şekil bellekli alaşımların termomekanik davranışına etkisi araştırılmıştır. Şekil bellekli alaşımların temel termomekanik özellikleri açıklanarak savunma ve havacılık alanlarında kullanılan uygulamaları sunulmuştur. Gelecekte şekil bellekli alaşımlarla ilgili potansiyel teknolojik gelişmeler açıklanmıştır.

Anahtar Kelimeler: Şekil bellekli alaşımlar, Şekil bellek etkisi, süperelastisite, termomekanik davranış.

Introduction

Smart materials, such as piezoelectrics, electrostrictives, magnetostrictives, electroactive polymers, shape memory alloys (SMAs) and shape memory polymers (SMPs) have attracted considerable attention in defense and aerospace industries in recent years (Chopra et al., 2013). Their sophisticated properties enable sensing, actuation, and vibration. Among smart materials, shape memory alloys (SMAs) are a unique class of intermetallic material which can recover as large as 8% strain under

different mechanical and thermal loadings. The effect of solid-to-solid phase transformations on the microstructure creates two fascinating characteristics which are called superelasticity and shape memory effect. Depending on the formation of the martensitic crystallographic structure from the high temperature phase (austenite), stress-induced or thermal-induced martensitic transformations (TIMT) occur. While the stress-induced martensitic transformation (SIMT) forms through loading, the thermal-induced martensitic transformation occurs through cooling. The reverse transformation takes place via unloading or heating. Figure 1 shows the classification of the martensitic formation. The related stress-strain and strain-temperature responses are also illustrated in Figure 1.

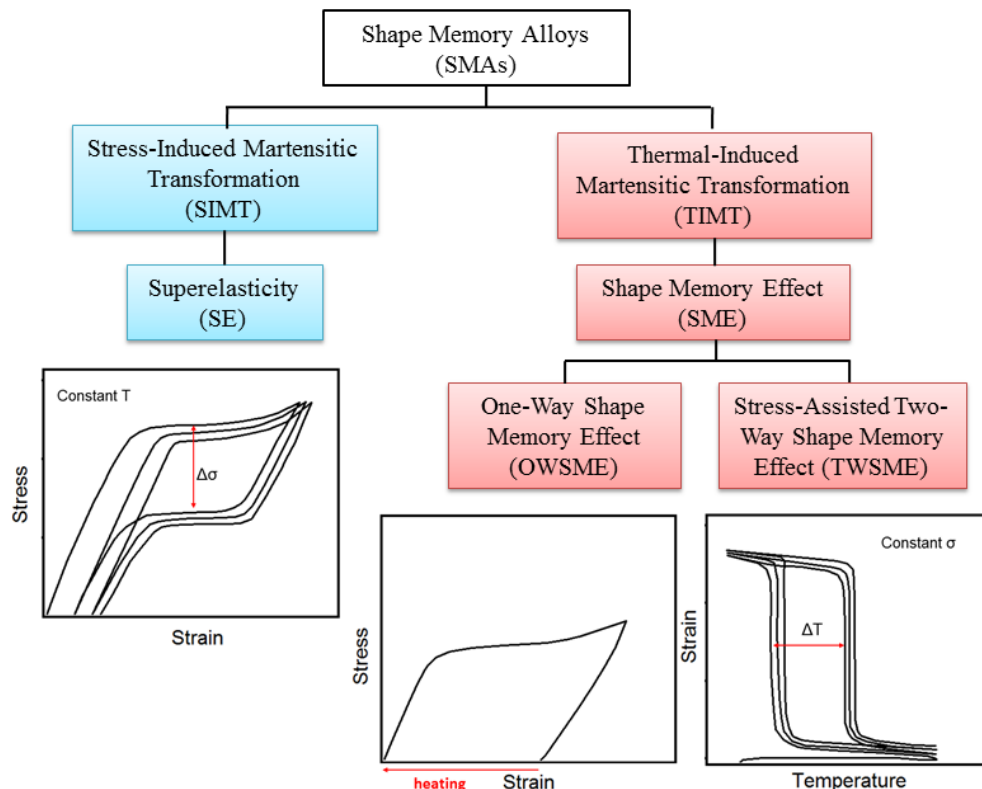


Figure 1. SIMT and TIMT responses in shape memory alloys (Dilibal et al., 2016)

A stress hysteresis ($\Delta\sigma$) can be obtained from the stress-strain diagram of the superelastic response. A temperature hysteresis (ΔT) occurs when the alloy is subjected to the stress-assisted thermal loading. Considering the crystallographic microstructure, while the austenitic phase is a crystallographically symmetric phase, the martensitic phase contains variants due to the low symmetry. The martensite phase exists either as a self-accommodated twinned microstructure or a stress-induced detwinned microstructure. Upon cooling in the absence of the applied load from the austenite phase, austenite to self-accommodated twinned martensite occurs

for the first case. In the second case, the tension or compression loading of the alloy in the austenite temperature causes the formation of the stress-induced detwinned martensite (Saedi et al., 2016; Dilibal, 2013).

NiTi SMAs are preferred in most of the practical applications since it is commercially available. In addition, they have superior ductility, higher corrosion resistance, higher tensile strength. They exhibit superelastic behavior with a large recoverable inelastic strain during an isothermal mechanical load/unload cycle above transformation temperature (Sehitoglu et al., 2001). It is well known that the superelasticity in NiTi SMAs shows strong temperature and nickel concentration dependence. Increasing the nickel concentration in NiTi SMA (more than 50.5% atomic concentration) lowers the martensitic transformation temperature and provides to obtain superelasticity at the room temperature (Gall and Maier, 2002). NiTi SMA requires a critical stress level which is associated with the evolution of the stress plateau during the loading in superelastic case. The superelastic response can be obtained over a wide temperature range up to M_d temperature. M_d is the temperature above which martensite cannot be stress induced. Upon unloading, the reverse transformation occurs with a lower stress plateau. The obtained stress hysteresis ($\Delta\sigma$) represents the dissipated energy during loading/unloading (Figure 1).

Shape memory effect is the other important characteristic of shape memory alloys. Once the austenitic NiTi alloy is cooled in a stress-free environment, the monoclinic NiTi self-accommodated martensite variants form. Many self-accommodating combinations of martensite variants initially form while conserving the alloy's macroscopic shape. During deformation in the martensitic phase, preferentially oriented martensitic variants undergo detwinning and mechanical twinning subsequently. Detwinning is the transition from multiple variants to the formation of a single variant. Upon heating, the shape memory effect occurs by reverse transformation as shown in Figure 1.

The transformation temperature of NiTi SMAs is lower than 100°C. Hence, it is difficult to utilize NiTi SMAs in the high temperature actuator systems which operate higher than 100°C. Many efforts have been made to shift the transformation temperatures of the NiTi based SMAs above the 100°C without compromising its functionality. Additional ternary elements such as Hf, Zr, Pd and Pt in an amount of up to 30% (at.) increase the transformation temperature of the NiTiX SMAs (Bigelow et al., 2011; Wua et al., 2013, Saleeb et al., 2015).

The transformation temperatures of a NiTi SMA can be notably changed by thermal/mechanical treatments, such as thermal cycling, ageing/annealing, cold-working or hot-working. During thermal cycling under constant stress, NiTi SMA provides a large recoverable strain (transformation strain) associated with the shape memory effect behavior in

the range of the operation temperatures (Padula II et al., 2012; Atlı et al., 2013). This process is also called “thermo-mechanical training” due to the thermal cyclic characteristic (Belyaev et al., 2012; Wada et al., 2008). Early studies mostly concentrated on obtaining maximum transformation strain and work output during thermo-mechanical training in the form of thermal cycling under constant stress (Padula II et al., 2008; Scherngell et al., 1998). However, a drawback of this specific case is the dimensional instability, causing a detrimental limitation for the future potential applications of this SMA material. The occurrence of the irrecoverable strain at martensite site (ϵ_M) and austenite site (ϵ_A) creates an evolutionary response under a large number of isobaric thermal cycles. This entire set of repeated thermal cyclic process produces a degradation of total actuation (transformation) strain (ϵ_{ACT}) and the dimensional instability in NiTi SMA.

SMA should be subjected to the thermomechanical cyclic “training” in order to obtain a dimensionally stable response during the desired operation. Many efforts have been made to obtain stabilized response and enhance the cyclic degradation resistance of NiTi SMA. The initial long-term thermomechanical training, which consists of pre-cycling (more than 100 cycles) under the isobaric bias-stress condition, provides nearly stabilized structure. This time-consuming technique can be used before putting the NiTi SMAs into use for any solid-state actuator application.

Considering short-term cases, thermomechanical processing techniques, which affect directly the shape memory property of NiTi SMAs, are extremely used to improve macroscopic dimensional stability characteristic. The aim of this classical methodology is to increase the critical shear stress to make dislocation formation difficult due to the formation of the modified and refined microstructure. However, the high critical stress for plastic deformation influences the martensite reorientation and detwinning which play pivotal roles on shape memory effect behavior of the NiTi SMA material. Therefore, the strain hardening needs to be accomplished without increasing the stress for deformation twinning. The NiTi SMA material should possess adequate ductility to exhibit inherent shape memory effect behavior with its regular amount of strain recovery. Conventional cold-working (Miyazaki et al., 1986), cold working followed by low-temperature annealing (Miller and Lagoudas, 2001) and severe plastic deformation methods, such as high pressure torsion (HPT) (Waitz et al., 2004; Sergueeva et al., 2003) and equal-channel angular extrusion (ECAE) (Kockar et al., 2007; Kockar et al. 2008), have been used aiming at the grain refinement and texture at the micro-structural level. Among these methods, ECAE has shown the most promising results with desired texture formation and grain refinement without changing the sample's cross-section when compared to the other thermo-mechanical counterparts (Kockar et al. 2008). Besides these methods, precipitation hardening through appropriate

heat treatment is also used to control the volume fraction and distribution of the precipitates within the matrix. This method is more effective for the Ni-rich composition to obtain coherent Ti_3Ni_4 nano-precipitates which increase the critical shear stress by impeding dislocation movements (Gall and Maier, 2002). However, Ti-rich (more than 50 % at.) or equiatomic NiTi SMAs are more convenient for the actuator applications due to their higher transformation temperature in comparison with their Ni-rich counterpart.

In addition, laser processing technique is also utilized to modify the thermomechanical characteristics of the NiTi SMA material in order to obtain thermal cyclic stabilization. In this technique, the recrystallization and further columnar grain growth cause strain hardening during cyclic actuation tests (Pequegnat et al., 2012). Further studies are required to clarify the effect of laser processing on the dimensional stability.

Alternatively, addition of ternary alloy (such as the substitution of 20 at.% Hf for Ti) followed by annealing is also tested for dimensional stability. The positive effect of adding Hf for the cyclic stability is consistent and has been already reported in the previous studies (Bigelow et al., 2011; Yang et al. 2013) In addition, its transformation temperatures are higher than its binary NiTi counterpart and low enough to avoid rapid actuator degradation from creep and annealing of dislocations (Yang et al. 2013). However, a series of isobaric thermal cyclic response of NiTiHf SMA revealed that the transformation temperatures increase dramatically with increasing bias-stress (Yang et al. 2013). Furthermore, it is reported that the extended operation at high temperature causes the formation of $(\text{TiHf})_2\text{Ni}$ particulates which embrittles the material and leads to degradation and failure in service (Denowh and Miller, 2012).

Although there are many competing methods to minimize thermal cyclic instability, an ideal combination has not been found to mitigate the dimensional instability. Despite the extensive research on NiTi SMA in the material science community, the cyclic stability is still a highly debated topic.

Many heat treatment studies are concentrated on Ni-rich NiTi SMAs due to the effect of precipitations; particularly TiNi_3 precipitates (Paula et al., 2004). The applied cooling rate also affects the formation of the TiNi_3 intermetallic precipitates. The stress fields around these precipitates have a great impact on the martensitic phase transformation temperatures (Motemani et al., 2009).

SMA devices are being used in a wide range of defense and aerospace applications using thermal- or stress-induced characteristics in different temperature and loading regimes (Hartl et al., 2007; Benafan et al., 2013). SMA actuators have many advantages over other conventional actuators, such as their large force-output/weight ratio, large stroke, flexibility in design and compactness. They also work without any dust or

noise during operation. SMA based morphing can be obtained using different configuration, such as wire, beam, tube, spring or ball. The design and geometrical configuration of the SMA affect the strength and efficiency of the actuator. SMA tube couplings are the first successful application for the hydraulic system of the F-14 aircraft in defense and aerospace industries. In this specific application, SMA coupling is deformed in its martensitic state to larger diameters for easy sliding along the pipes (Figure 2.a). SMA coupling is heated above the A_f temperature to hold the ends of pipes firmly with appropriate compressive forces as shown in Figure 2.b. This process can substitute socket welds or compression fittings in defense and aerospace industries by generating a highly reliable metal seal.

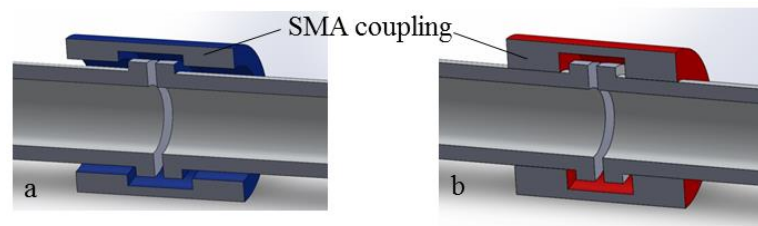


Figure 2. The cross-section of the SMA coupling a) in its martensitic state
b) heated above the A_f temperature

The NiTi SMA actuator beams are placed on the nozzle of a jet engine to bend the nozzle composite. The inward bending of the variable geometry chevrons (VGC) occurs through the activation of the surface heaters placed on the NiTi SMA beams. The bending mechanism provides to mix the jet engine hot gases and rear end cold air with less turbulence. It also provides an incremental reduction of the noise generated during take-off and landing. The NiTi SMA beams actuated variable geometry chevron flight test project was executed by Boeing between 2002 and 2006 (Calkins et al., 2010).

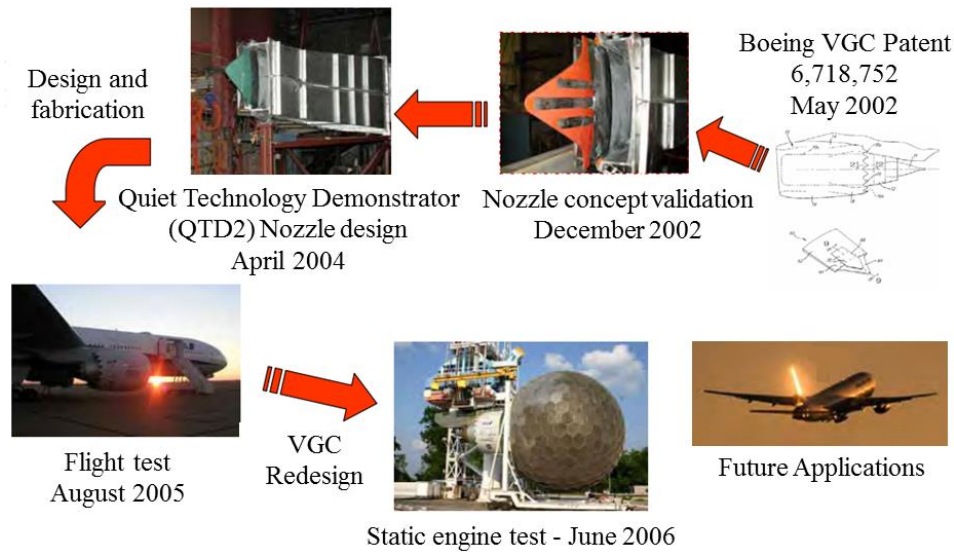


Figure 3. The applied Boeing flight testing process for the NiTi SMA beams as a variable geometry chevron (Calkins and Mabe, 2010)

In addition to the bending beam, the tubular SMA actuators have found many applications in the aerospace industry. They are used for the reconfigurable rotor blade and airplane wing morphing systems (Owusu-Danquah et al., 2015). The thickness and length of the SMA tube determines the magnitude of force or torque required to produce the desired amount of stroke in the SMA actuator. Helicopter blades are designed to operate between the ideal hover/cruise conditions. The required blade twist can potentially be achieved using different conventional actuator system, such as hydraulic, pneumatic, electromechanical or electro-hydraulic systems. Hydraulic and pneumatic actuators cannot be used without developing and installing new fluid slip-rings, violating the existing hub requirement. Electro-hydraulic actuators and electric motors are too heavy to place inside of the rotor blade. However, NiTi-based SMA torque tube actuators are designed based on the active helicopter rotor blade configuration. In this specific configuration, thermally activated SMA torque tubes twist the helicopter blade for different flight conditions. Its high power/weight ratio and low volume enable the actuator to function inside of the spinning blade. The basic concept of the reconfigurable rotor blade (RRB) is illustrated in Figure 4. The SMA torque tube actuator located inside the spar near the root of the blade (Arbogast et al., 2008). A passive torque tube is used to transmit torque from the SMA actuator to the tip of the blade.

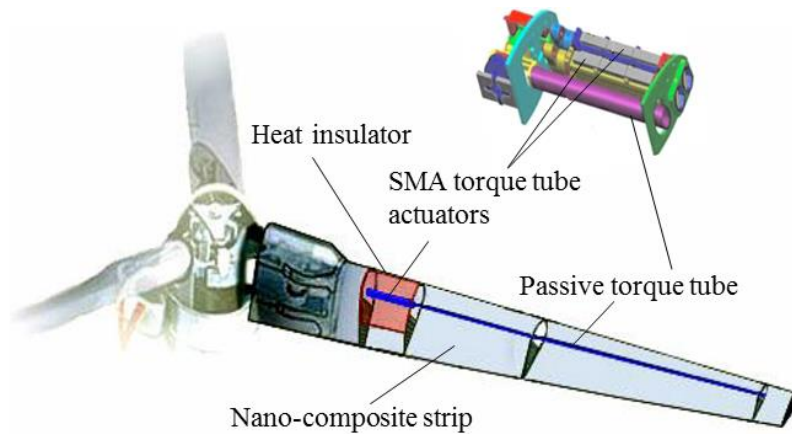


Figure 4. Active rotor blade using NiTi SMA torque tube (Arbogast et al., 2008)

The NiTi SMAs are used in various multi-fingered anthropomorphic robotics applications with different forms, such as wire, spring, plate and tube. Their light weight and silent properties provide crucial advantages over the hydraulic, pneumatic and electrical motor systems. The most famous SMA actuated hand is the Hitachi Ltd. Hand. It works using NiTi SMA wire and pulley system (Nakano et al., 1984). NiTi SMA actuated ITU robot hand system is the other three finger robotics hand configurations (Figure 5). The ITU robot hand consists of gripping, releasing and control subsystems. It is used for mine clearance task in dangerous environment. The gripping force is obtained via Joule heating of the NiTi SMA plates. The low bandwidth problem of the NiTi SMAs is solved using the cooling fluid in the ITU robot hand. The releasing subsystem activated using the cooling fluid that is pumped from the fluid pump to the surface of the NiTi plates through the plastic pipes. It is triggered by the pressure sensor which is placed onto the thumb (Dilibal et al., 2004).

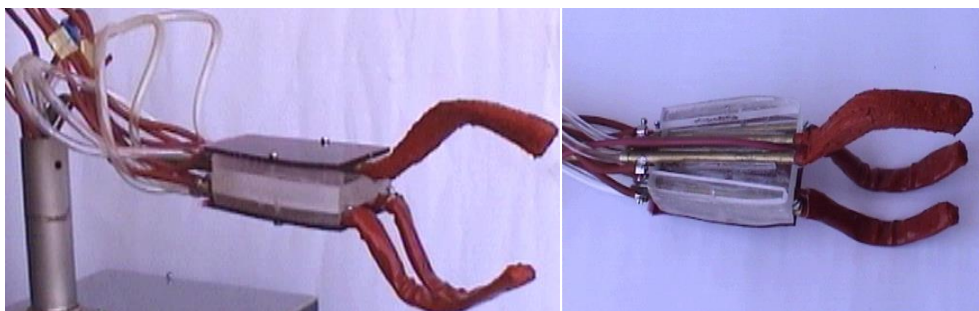


Figure 5. NiTi SMA actuated ITU Robot Hand (Dilibal et al., 2004)

The antagonistic response of two different SMA wires (Figure 6.a), plates (Figure 6.b), tubes or springs affects the response time of the applied mechatronics system. Both shape memory and superelastic properties can be used in an antagonistic SMA actuator system. The antagonistic SMA actuator can potentially be built in various configurations for any space related simple mechanism. Two SMA components are assembled below M_f temperature. They apply antagonistic force on each other upon systematically heating above A_f temperature. The cyclic heating and cooling processes create a reversible actuation stroke to use in different defense and aerospace actuation system.

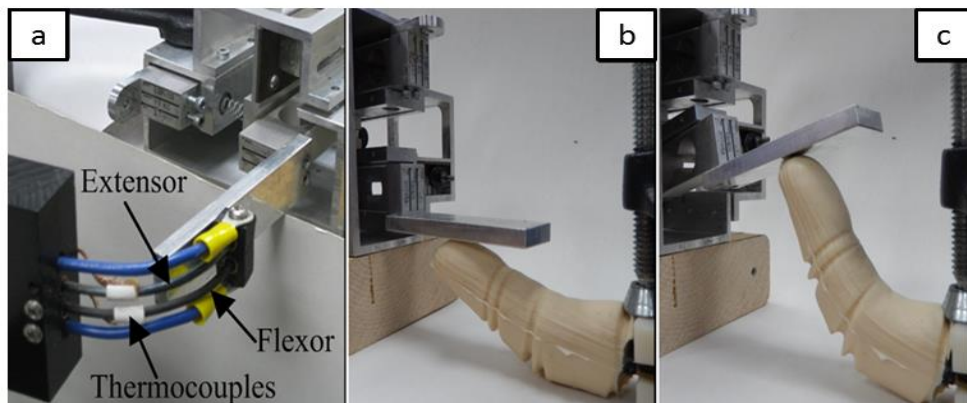


Figure 6. Robotics fingers using antagonistically actuated NiTi SMA wires (a) and plates (b, c) (Dilibal et al. 2015; Engeberg et al., 2015)

Many defense and aerospace applications use explosive tools to achieve a release mechanism. Cylindrical SMA actuated bolt breaking release system which is called Frangibolt, works as an alternative hold down and release mechanism to the explosive pyrotechnic process (Busch and Bokaie, 1994). The actuator unit consists of a SMA cylinder with surrounding heater and insulation (Figure 7). In this system, the notched bolt and the bearing washers are assembled along with the SMA actuator which is in the compressed state. The assembly is preloaded by tightening the assembly to the prescribed torque. Upon heating, the compressed SMA actuator applies tension on the bolt to break and separate the unit. The fracture occurs at the notch point. The SMA based hold down and release mechanism system can be adapted on different aerospace deployment systems, such as satellite solar panels deployment unit (Nava et al., 2015).

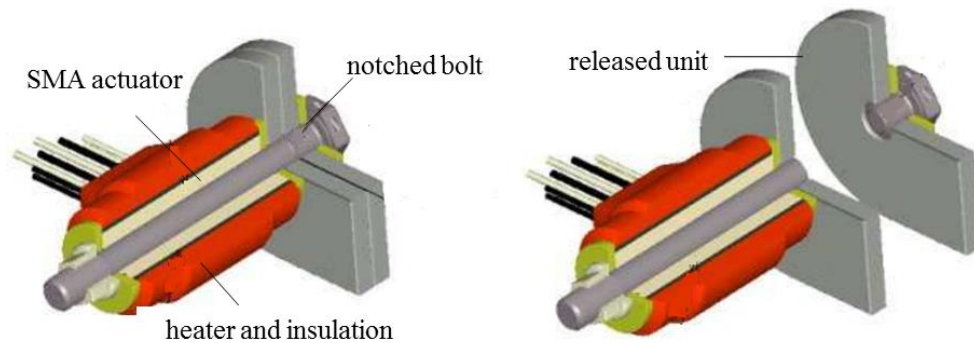


Figure 7. Schematic illustration of SMA actuated Frangibolt device (Busch and Bokaie, 1994)

The SMA actuated deployable antenna system is another important device in the aerospace satellite projects (Lana et al., 2007). It can be packed in the limited amount of space in the launch vehicle. The superelastic SMA based advanced antenna system is crushed in a box before the deployment. The crimping force is applied on the system during the launch process. When the antenna system reaches at the desired orbit system, it is activated through the release of crimping force or through the Joule heating above the A_f temperature. The antenna system unpacks to its full length once in orbit.

The integration of SMAs into composite structure provides many benefits; such as self-healing, sensing and damping. Moreover, a composite system which contains SMA fibers can be stiffened or controlled through heating (Lester et al., 2015). Most of the SMA based adaptive composite systems are produced using embedded SMAs into polymer matrix composites.

The performance of the above-mentioned applications is significantly dependent on the thermomechanical response of the SMAs. Care must be taken in the thermal operation range in addition to the design parameters. Hence, the effect of the heat treatment on the thermomechanical response should be understood well. Many researchers worked on heat treatment of the NiTi SMAs using different aging time, aging temperature and cooling rate. The volume of Ni_4Ti_3 , Ni_3Ti_2 , and Ni_3Ti precipitates affect the austenite yield strength of the nickel-rich NiTi SMAs (Gall and Maier, 2002; Besseghini et al., 1999). The change of nickel concentration in the NiTi matrix through the nickel depletion during precipitate growth significantly affect the thermomechanical response (Sehitoglu et al., 2001; Saedi et al., 2016). In the present work, the martensitic NiTi SMA is obtained at the room temperature through the long-term heat treatment.

Experimental Procedure

$\text{Ni}_{50.1}\text{Ti}_{49.9}$ (at.) single crystal SMA oriented in the [001] direction is used in this work. The samples are solutionized (SL) at 1000°C for 2 hours in an inert gas atmosphere. The stress-free thermal-induced martensitic transformation temperatures of the NiTi samples were measured using a differential scanning calorimeter (DSC) which unveil starting and finishing temperature of martensite and austenite phases. Perkin-Elmer Instruments Pyris-1 DSC is utilized to determine the reverse and forward martensitic phase transformation temperatures for the SL and SL + heat treated NiTi SMA samples. The DSC analysis starts with heating to high temperature, which ensures the matrix is fully austenitic. Afterwards, the sample is cooled to the low temperature to determine the related peak temperature. The heating and cooling rates are 15°C per minute. The result of the DSC analysis gives the stress-free characteristic temperatures; M_s : martensite start temperature, M_f : martensite finish temperature, A_s : austenite start temperature, A_f : austenite final temperature.

Small dog-bone shaped samples were used for the tensile test with the gauge length of 2.73 mm. The incremental tensile tests were performed at the room temperature (18°C) using a servo-hydraulic load frame. The length and thickness of the samples were 26,63 mm and 1.21 mm respectively. The heat treatment process was conducted at 450°C for 100 hours in a furnace for the long term case. The process was ended by water quench. The incremental tensile tests were conducted on the heat treated NiTi samples.

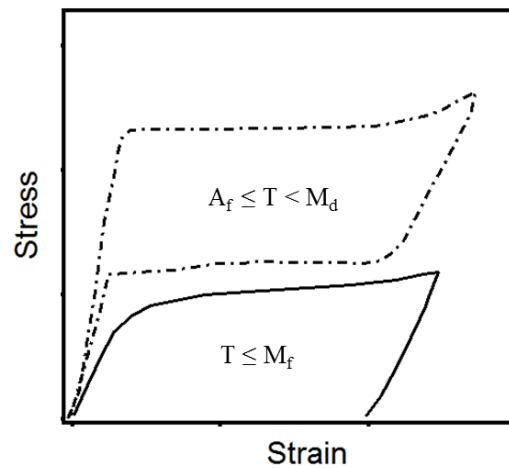


Figure 8. Schematic for the loading NiTi SMA in austenitic and martensitic phases

The tensile stress-strain response of the NiTi SMAs is schematized for the austenitic and martensitic NiTi cases in Figure 8. The complete superelastic behavior takes place when the alloy is loaded between the austenite final (A_f) and M_d temperatures. M_d is the temperature above which

martensite cannot be stress induced. The recovery of deformation occurs upon unloading. The detwinning of the martensitic NiTi SMA occurs when the alloy is loaded below the M_f temperature.

Results and Discussion

The DSC results for the solutionized and long-term heat treated NiTi SMA samples are shown in Figure 9. The thermal hysteresis is defined from the highest peak to peak range for the exo- and endothermic thermo-gram results. The measured DSC results for the solutionized and heat treated NiTi are shown in dotted and solid lines respectively in Figure 9. The heat treatment which is applied at 450°C for 100 hours shifted the hysteresis temperatures to the room temperature operation range. Furthermore, the thermal hysteresis obtained from heat treated case ($\Delta T_{HT} = 44^\circ\text{C}$) is larger than that for the solutionized case ($\Delta T_{SL} = 12^\circ\text{C}$).

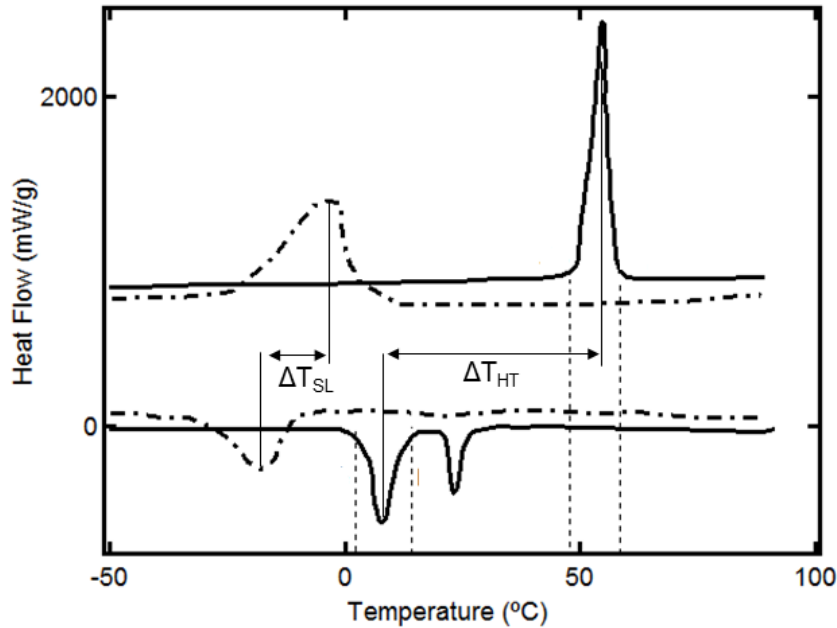


Figure 9. DSC results for the solutionized and heat treated Ni_{50.1}Ti_{49.9} SMA

The tensile stress-strain response of the NiTi single crystal is shown in Figure 10. The tensile test is conducted after applying the long-term heat treatment. Ni_{50.1}Ti_{49.9} (at.) single crystal SMA oriented in the [001] direction subjected to 2% strain incremental tensile loading at room temperature in martensitic phase. The applied load is unloaded at the end of each 2% strain increment. The stress-strain diagram of the incremental tensile test results is shown in Figure 10.

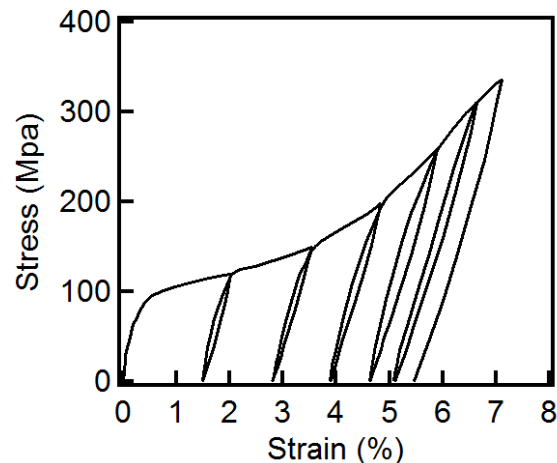


Figure 10. The incremental tensile loading response of martensitic NiTi

Long term heat treatment can be applied on the other SMAs to shift the transformation temperature. Recently, the long-term heat treatment has been applied on CoNiAl SMA to obtain the superelastic response at the room temperature. The alloy is heat treated at 1200°C for 36 hours before conducting the compressive test. A lower austenite finish temperature (40°C) is obtained as a result of the heat treatment (Dilibal et al., 2011).

Additive manufacturing methods are the state-of-art technologies for the fabrication of the highly complex components. The long-term heat treatment technique can be used as an option for these components to obtain the desired operational temperature range.

Conclusions

The present study is focused on the investigation of the effect of long-term heat treatment on the thermomechanical response of the NiTi SMAs. The results show that transformation temperatures increase dramatically with the long-term heat treatment. The applied heat treatment (450°C for 100 hours) increased the transformation temperature with a wide temperature hysteresis. In addition, the operation range of the SMA is shifted to the room temperature service condition. The obtained stress-strain loading plots proved that the heat treated samples are in the martensitic state at the room temperature.

The long-term heat treatment can be transferred into different applications in the defense and aerospace industries for the implementation of smart actuation and smart composite systems. The state of art technology can be created with the design of the SMA integrated future aircrafts, lightweight UAVs, reconfigurable spacecraft parts and compact satellite systems. Furthermore, a variety of SMA solutions can be created using the proper heat treatment on the additive manufactured SMA based aerospace components. Selective laser melting (SLM), electron beam melting (EBM),

selected laser sintering (SLS), direct metal laser sintering (DMLS), electron beam free form fabrication (EBFFF), laser engineered net shaping (LENS) and direct metal deposition (DMD) are the most common additive manufacturing techniques. The additive manufacturing process can provide to build up various NiTi SMA based defense and aerospace applications by using 3D CAD design.

Acknowledgements The author acknowledges Prof.H.Sehitoglu for his support.

References

- Arbogast, D.J., Ruggeri, R.T., and Bussom, R.C., (2008). Development of a ¼-Scale NiTiNol Actuator for Reconfigurable Structures. Proceedings of SPIE 6930. Industrial and Commercial Applications of Smart Structures Technologies 69300L.
- Atli, K.C., Franco, B.E., Karaman, I., Gaydos, D., Noebe, R.D. (2013). Influence of Crystallographic Compatibility on Residual Strain of TiNi Based Shape Memory Alloys during Thermo-Mechanical Cycling. *Materials Science Eng. A*, 574, 9-16.
- Belyaev, S., Resnina, N., Sibirev, A. (2012). Peculiarities of residual strain accumulation during thermal cycling of TiNi alloy. *J. Alloy Comp.* 542 37-42.
- Benafan, O., Notardonato, W.U., Meneghelli, B.J., and Vaidyanathan R., (2013). Design and Development of a Shape Memory Alloy Activated Heat Pipe-Based Thermal Switch. *Smart Mater. Struct.*, 22, 105017.
- Besseghini, S., Villa E., and Tuissi, A. (1999). Ni-Ti-Hf Shape Memory Alloy, Effect of Aging and Thermal Cycling. *Mater. Sci. Eng. A*, 273(8), 390–394.
- Bigelow, G.S. Garg, A. Padula II, S.A., Gaydos, D.J. and Noebe, R.D. (2011). Load-Biased Shape-Memory and Superelastic Properties of a Precipitation Strengthened High-Temperature. Ni_{50.3}Ti_{29.7}Hf₂₀ Alloy, *Scripta Mater.*, 64(8), 725–728.
- Busch, JD., Bokaie, MD. (1994). Implementation of heaters on thermally actuated spacecraft mechanisms, 28th Aerospace Mechanism Symposium, LeRC, Cleveland, Ohio.
- Calkins, F.T., Mabe, J.H. (2010). Shape Memory Alloy Based Morphing Aerostructures. *J. Mech. Design* 132, 111012.
- Chopra, I, Sirohi J, (2013). *Smart Structures Theory*, (ss. 223-235). New York: Cambridge University Press.

- Denowh, C.M., Miller, D.A. (2012). Thermomechanical training and characterization of Ni–Ti–Hf and Ni–Ti–Hf–Cu high temperature shape memory alloys. *Smart Mater. Struct.* 21, 065020.
- Dilibal, S., Sahin, H., Dursun, E., Engeberg, E.D. (2016) Nickel–titanium shape memory alloy-actuated thermal overload relay system design, *Electrical Engineering*, Springer DOI: 10.1007/s00202-016-0458-2
- Dilibal, S., Güner, E., Akturk, N. (2002). Three-finger SMA Robot Hand and its Practical Analysis. *Cambridge Univ Press, Robotica Journal*, 20,175-180.
- Dilibal, S., Tabanlı, M., Dikicioğlu, A. (2004). Development of shape memory actuated ITU Robot Hand and its mine clearance compatibility. *Journal of Materials Processing Technology*, 1390-1394.
- Dilibal, S., Engeberg, E.D. (2015). Finger-like manipulator driven by antagonistic NiTi shape memory alloy actuators, *IEEE Int. Conference on Advanced Robotics*, Istanbul.
- Dilibal, S. (2013). Investigation of Nucleation and Growth of Detwinning Mechanism in Martensitic Single Crystal NiTi Using Digital Image Correlation. *Metallography, Microstructure, and Analysis*. 2 242-248.
- Dilibal, S., Sehitoglu H., Hamilton R., Maier H.J., Chumlyakov Y. (2011). On the Volume Change in Co-Ni-Al during Pseudoelasticity. *Materials Science and Engineering A*, 6, 528.
- Engeberg, E.D, Dilibal, S., Vatani M., Choi JW and Lavery J. (2015). Anthropomorphic finger antagonistically actuated by SMA plates. *Bioinspiration & Biomimetics*, 10 (5): 056002.
- Kockar, B., Karaman, I., Kulkarni, A., Chumlyakov, Y., Kireeva, I.V. (2007). Effect of severe ausforming via equal channel angular extrusion on the shape memory response of a NiTi alloy. *Journal of Nuclear Materials* 361, 298-305.
- Kockar, B., Karaman, I., Kim, J.I., Chumlyakov, Y.I., Sharp, J., Yu, C.-J. (2008). Thermomechanical cyclic response of an ultrafine-grained NiTi shape memory alloy. *Acta Materialia* 56, 3630-3646.
- Lana, X., Leng, J., Du, S. (2007). Design of a Deployable Antenna Actuated by Shape Memory Alloy Hinge. *Materials Science Forum Vols. 546-549* 1567-1570.
- Lester, B., Baxevanis, T., Chemisky, Y., Lagoudas, D.C. (2015). Review and perspectives: shape memory alloy composite systems. *Acta Mech.* 226(112) 3907-3960.
- Gall, K., Maier, H.J. (2002). Cyclic deformation mechanisms in precipitated NiTi shape memory alloys. *Acta Materialia* 50, 4643-4657.

- Hartl, D.J. and Lagoudas, D.C. (2007). Aerospace Applications of Shape Memory Alloys, *Proc. Inst. Mech. Eng. Part G*, 221, 535– 552.
- Miller, D.A., Lagoudas, D.C. (2001). Influence of cold work and heat treatment on the shape memory effect and plastic strain development of NiTi. *Materials Science and Eng. A*, 308, 161-175.
- Miyazaki S, Igo Y, Otsuka K. (1986). Effect of thermal cycling on the transformation temperatures of Ti-Ni alloys, *Acta Metall*, 34, 2045.
- Motemani, Y., Nili-Ahmadabadi, M., Tan, M.J., Bornapour, M., Rayagan, Sh. (2009). Effect of cooling rate on the phase transformation behavior and mechanical properties of Ni-rich NiTi shape memory alloy, *Journal of Alloys and Compounds* 469 164–168.
- Nakano, Y., Fujie, M., Hosada, Y. (1984). Hitachi's robot hand. *Robotics age*, 6(7), 18-20.
- Nava, N., Collado M., Palladino, M., Patti, S. (2015). A novel hold-down and release mechanism for non-explosive actuators based on sma technology. 16th European Space Mechanisms and Tribology Symposium, Bilbao, Spain.
- Owusu-Danquah, J.S., Saleeb, A.F., Dhakal, B., Hurley, A.E., Dilibal, S., Padula II, S.A., Noebe, R.D., and Bigelow G. S., (2013). Large-scale Simulation of a Torque-Tube Actuator Using a 3D Multi-mechanism Material Model: A Comparative Study with Ni_{49.9}Ti_{50.1} and Ni_{50.3}Ti_{29.7}Hf₂₀ SMAs, *ASME Conference on Smart Materials, Adaptive Structures and Intelligent Systems*, Snowbird, Utah, USA.
- Padula II, S., Qiu, S., Gaydosh D., Noebe, R., Bigelow, G., Garg, A., R. Vaidyanathan, (2012). Effect of Upper-Cycle Temperature on the Load-Biased, Strain-Temperature Response of NiTi. *Metallurgical and Materials Transactions A*, 43,12, 4610-4621.
- Padula, II S.A., Bigelow G., Noebe R.D., Gaydosh D., and Garg A. (2006). Challenges and Progress in the Development of High-Temperature Shape Memory Alloys Based on NiTiX Compositions for High-Force Actuator Applications. *Int. Conf. on Shape Memory and Superelastic Technologies*, 7-11 May, Pacific Grove, CA.
- Paula, A.S., Canejo, J.P.H.G., Martins, R.M.S., Braz Fernandes (2004). F.M. Effect of thermal cycling on the transformation temperature ranges of a Ni–Ti shape memory alloy. *Materials Science and Engineering A* 378 92–96.
- Pequegnat, A., Daly, M., Wang, J., Zhou, Y., Khan, M.I. (2012). Dynamic actuation of a novel laser-processed NiTi linear actuator. *Smart Mater. Struct.* 21, 094004.

- Saedi, S., Turabi, A.S., Andani S.M.T., Haberland, C., Karaca, H., Elahinia M. (2016). The influence of heat treatment on the thermomechanical response of Ni-rich NiTi alloys manufactured by selective laser melting. *Journal of Alloys and Compounds*.
- Saleeb, A.F., Dhakal, B., Dilibal, S., Owusu-Danquah, J.S., and Padula, II S.A. (2015). On the Modeling of the Thermo-mechanical Responses of Four Different Classes of NiTi-Based Shape Memory Materials Using a General Multi-mechanism Framework, *Mechanics of Materials*, 80, 67- 86.
- Sehitoglu, H., Jun, J., Zhang, X.Y., Karaman, I., Chumlyakov, Y., Maier, H.J., Gall, K. (2001). Shape Memory And Pseudoelastic Behavior of 51.5%Ni-Ti Single Crystals In Solutionized and Overaged State. *Acta Mater.* 3609 49.
- Scherngell, H., Kneissl, A.C. (1998). Training and stability of the intrinsic two-way shape memory effect in Ni-Ti alloys. *Scripta Materialia*, 39, 2, 205-212.
- Sergueeva, A.V., Song, C., Valiev, R.Z., Mukherjee, A.K. (2003). Structure and properties of amorphous and nanocrystalline NiTi prepared by severe plastic deformation and annealing, *Materials Science and Engineering A*. 339, 159, 165.
- Yang, F., Coughlin, D.R., Phillips, P.J., Yang, L., Devaraj, A., Kovarik, L., Noebe, R.D., Mills, M.J. (2013) Structure analysis of a precipitate phase in an Ni-rich high-temperature NiTiHf shape memory alloy. *Acta Materialia*, 61 3335–3346.
- Wada, K., Liu, Y. (2008). On the mechanisms of two-way memory effect and stress-assisted two-way memory effect in NiTi shape memory alloy. *J. of Alloys and Compounds* 449 125-128.
- Waitz, T., Kazykhanov, V., Karnthaler, H.P. (2004). Martensitic phase transformations in nanocrystalline NiTi studied by TEM. *Acta Materialia* 52, 137-147.
- Wua, Y., Patriarcaa, L., Sehitoglu H., Chumlyakov, Y. (2016). Ultrahigh tensile transformation strains in new Ni50.5Ti36.2Hf13.3 shape memory alloy. *Scripta Materialia*, 118 51-54.

Extended Summary

Savunma ve Havacılık Uygulamalarında Uzun Süreli Isıl İşlemin NiTi Şekil Bellekli Alaşımların Termomekanik Davranışına Etkisi

Giriş

Son yıllarda, akıllı malzemeler olarak bilinen piezoelektrik, elektrostriktif, manyetostriktif, elektroaktif polimerler, şekil bellekli alaşımlar ve şekil bellekli polimerlerin savunma ve havacılık uygulamalarında kullanılmasına olan ilgi artmıştır. Akıllı malzemeler içerisinde yer alan intermetalik malzemeler olan şekil bellekli alaşımlar, uygulanan gerilmeye veya sıcaklık değişimine bağlı olarak %8`lere varan birim şekil değiştirmenin geri dönüşümünü sağlayabilmektedir. Alaşımda düşük sıcaklık fazı olan martenzit fazın oluşumu gerilme veya sıcaklık değişimi ile sağlanmaktadır. Alaşımda elde edilen martenzitik dönüşüm; gerilmenin etkisiyle elde edilen martenzitik dönüşüm ve sıcaklık etkisi ile elde edilen martenzitik dönüşüm olmak üzere iki gruba ayrılır. Kristalografik mikroyapı olarak östenit faz simetrik bir yapı içerirken martenzit faz düşük simetri içerir.

Şekil bellekli alaşımlar süperelastik özellik ve şekil bellek özelliği olmak üzere iki temel özelliğe sahiptir. Birinci durumda; östenit fazdaki alaşım üzerine bir gerilme uygulandığında, gerilmeden dolayı martenzitik faz dönüşümü oluşmakta ve uygulanan gerilme kaldırıldığında alaşım tekrar östenit faza geçerek ilk şekline geri dönmektedir. Bu özellik alaşımın süperelastik özelliği olarak bilinir. Mikroyapı olarak östenit fazdaki alaşım üzerine gerilme uygulanmasıyla karşıt-ikizlenmiş (detwinned) martenzit yapı oluşur. Uygulanan gerilme kaldırıldığında alaşım tekrar östenit faza geçer. Şekil bellek özelliğinde ise, gerilme ile elde edilen martenzitik yapıdan farklı olarak, alaşım martenzit fazda deformasyona uğratıldığında, uygulanan sıcaklık ile tekrar östenit faza geçerek makroskopik olarak ilk şekline geri döner. Mikroyapı olarak östenit fazdaki alaşım, östenit faz sıcaklığı altına soğutulduğunda, kendiliğinden martenzit varyant ikizleri oluşur. Oluşan varyant ikizleri, martenzit fazda uygulanan deformasyon ile birlikte yönlendirilmiş varyant çiftlerini oluşturur. Martenzit fazda uygulanan deformasyonun artırılması ile birlikte mikroyapıda karşıt-ikizlenme (detwinning) başlar. Alaşıma uygulanan sıcaklık ile birlikte alaşımın mikroyapısı tekrar östenit faza dönüşür.

Elektromekanik, akışkan basıncı (hidrolik/pnömatik), mekanik, akıllı malzemeler ve hibrid eyleyiciler doğrusal veya döner hareket sağlayan eyleyicilerdir. Bu eyleyici mekanizmaları içerisinde elektromekanik, akışkan basıncı ve mekanik eyleyiciler geleneksel eyleyici sistemleri olarak

bilinir. Geleneksel eyleyici sistemlerinden farklı olarak kullanılan şekil bellekli alaşımlar, savunma ve havacılık alanında başta uçak ve helikopter kanat ve pal hareket sistemlerinden, jet motor nozul hareket sistemlerine, adaptif kompozit sistemlerden kaplin sistemlerine kadar birçok uygulamada geleneksel eyleyici sistemlerinin yerini almaya başlamıştır. Şekil bellekli alaşımlar, mekanizma olarak sistem içerisinde uygulamada kullanılmadan önce boyutsal olarak kararlı davranış göstermesi için sabit sıcaklık veya sabit yük altında termomekanik döngüsel şekil bellek eğitimi olarak gerçekleştirilen işlemlere tabi tutulurlar. NiTi şekil bellekli alaşımların ilk kullanım alanı, F-14 savaş uçaklarında hidrolik boru bağlantılarının NiTi kaplinler ile birleştirilmesi ile olmuştur. İlerleyen yıllarda jet motoru nozulunda V şeklindeki uç kısımların NiTi şekil bellekli alaşımı plakaların içe doğru bükülmesi ile kalkış ve inişte gürültü azaltma maksatlı kullanılmıştır. Başka bir uygulamada boru şeklindeki NiTi alaşımlardan elde edilen torkun kanat hareketlerinde hidrolik eyleyici sistemler yerine kullanılması üzerine araştırmalar yoğunlaşmıştır. Helikopter palleri içerisine yerleştirilen boru şeklinde tork eyleyici sistemleri hafif olması ve yüksek güç/ağırlık oranına sahip olması nedeniyle NiTi şekil bellekli alaşımların potansiyel olarak kullanılmasına olanak vermektedir. Bunların yanında robotik uygulamalarda eyleyici olarak sistemin basit bir şekilde kullanılabilmesi, kullanılan robotik sistemin etkinliğini artırmıştır. Özellikle iki farklı NiTi tel, levha, yay veya borunun zıt yönlü (antagonistik) eyleyici olarak birlikte kullanılması sistemin strok kapasitesinin artırılmasına ve tepki zamanının düşürülmesine olanak vermiştir.

Uzay ve havacılık uygulamalarında, mekatronik fırlatma sistemleri içerisinde işlemi tamamlanmış olan parçanın sistemden uzaklaştırılması için kullanılan patlama sistemleri, yerlerini NiTi esaslı Frangibolt ayırma sistemlerine bırakmıştır. Bu sistemde ön gerilme uygulanarak sisteme yerleştirilen silindirik NiTi şekil bellekli alaşım üzerinden kısa süreli elektrik akımı geçirilir. Elde edilen faz dönüşümü ile uygulanan ön gerilme kaldırılır ve silindirik NiTi içerisinde bulunan vidanın kırılmasına yol açar. Böylece mekatronik sistem içerisinde işlemi tamamlanmış parça sistemden uzaklaştırılmış olur. Şekil bellekli alaşımlar kullanılarak geliştirilen konuşlandırılabilir anten sistemleri, havacılık uydu projelerinde potansiyel olarak kullanılabilir bir başka kullanım alanıdır. Fırlatma araçlarında kısıtlı yer bulunmasından dolayı küçük bir alan içerisine yerleştirilen anten sistemleri belirlenen orbit sistemine ulaştırıldığında sistem açılarak antenin süperelastik özelliği sayesinde geniş bir alana yayılmasına neden olur.

NiTi şekil bellekli alaşımların, özellikle polimer ve metal matrisli kompozit sistemlerde gömülü olarak kullanılması, sönümleme, sensör ve kendi kendini yapısal iyileştirme özelliklerinin kompozit üzerinde elde edilmesini sağlamaktadır. Fonksiyonel bir kompozit yapının elde edilmesi

savunma ve havacılık alanlarında geliştirilecek mekatronik sistemlerde birçok farklı kullanım imkanı sağlayacaktır.

Bu çalışmada, NiTi şekil bellekli alaşımların östenit/martenzitik dönüşüm sıcaklıklarının istenilen uygulama sıcaklık aralığına kaydırılması maksadıyla, uzun süreli ısı işlemlerin NiTi şekil bellekli alaşımların termomekanik davranışına etkisi araştırılmıştır.

Deneysel Çalışmalar

Çalışmada, deneyler için [001] kristalografik doğrultudaki tek kristal Ni_{50.1}Ti_{49.9} (at.) numuneler kullanılmıştır. Öncelikle Ni_{50.1}Ti_{49.9} (at.) numuneler 1000°C'de 2 saat süreyle inert gaz ortamında çözeltiye alma işlemine maruz bırakılmış daha sonra diferansiyel taramalı kalorimetre cihazı (DSC) kullanılarak numunelere ait martenzitik ve östenitik dönüşüm sıcaklıkları tespit edilmiştir. DSC işlemleri Perkin-Elmer Instruments Pyris-1 DSC cihazı kullanılarak yapılmıştır. Çözeltiye alma işlemi sonrası uzun süreli yaşlandırma ısı işlemi olarak uygulanan 450°C'de 100 saat süreli tutma ve suda soğutma yaşlandırma işlemi sonucunda elde edilen mikroyapıya ait martenzit ve östenit faz dönüşüm sıcaklıkları DSC analizi ile tespit edilmiştir. DSC analizi sonucunda tespit edilen sıcaklık dönüşümleri M_f (numunenin tamamen martenzit fazda olduğu sıcaklık), M_s (numunenin östenit fazdan martenzit faza geçiş sıcaklığı), A_s (numunenin östenit faza geçiş sıcaklığı) ve A_f (numunenin tamamen östenit fazda olduğu sıcaklık) olarak bulunur. Kemik (dogbone) şeklindeki numunelerin numune uzunluğu 26,63 mm ve kalınlığı 1.21 mm boyutlarındadır. Numuneler ısı işlem sonrasında toplam %7 birim şekil değiştirme elde edilecek şekilde kademeli olarak çekme testine tabi tutulmuştur.

Sonuçlar ve Gelecekte Şekil Bellekli Alaşımlarla İlgili Savunma ve Havacılık Uygulamaları

Uzun süreli yaşlandırma ısı işleminin NiTi alaşımların termomekanik yapısına etkisinin araştırıldığı bu çalışmada çözeltiye alma işlemi sonrası uzun süreli yaşlandırma ısı işlemi olarak uygulanan 450°C'de 100 saat süreli tutma ve suda soğutma yaşlandırma işlemi sonucunda faz dönüşüm sıcaklıklarının 11°C-55°C aralığına kayarak bu aralıkta yapılacak uygulamalar için kullanılabilir hale geldiği tespit edilmiştir. Faz dönüşüm sıcaklıklarındaki kayma ile birlikte aynı zamanda elde edilen sıcaklık histerisisinin 12°C(ΔT_{SL})'den 44°C(ΔT_{HT})'ye genişlediği görülmüştür. 18°C oda sıcaklığında gerçekleştirilen çekme testi gerilme-birim şekil değiştirme sonuçları numunelerin test yapılan sıcaklıkta martenzitik yapıda olduğunu göstermiştir.

Şekil bellekli alaşımlar kullanılarak geliştirilen ürünlerin katmanlı imalat yöntemi ile üretimi konusunda yoğun çalışmalar bulunmaktadır.

Katmanlı imalat teknolojisi döküm, dövme ve talaşlı imalat yöntemleri ile karşılaştırıldığında kalıp veya fikstür ihtiyacını ortadan kaldırarak üç boyutlu model üzerinden yeni parçalar geliştirilebilmesine olanak sağlayan bir teknolojidir. Katmanlı imalat teknolojisi kullanılarak üretilen parçaların üç boyutlu modeli değiştirilerek tasarım hataları düzeltilebilmekte, montaj kolaylığı sağlanmakta veya mekatronik sistem parçalarının tasarımında kolaylıkla değişikliğe gidilebilmektedir. Gelecekte katmanlı imalat yöntemi kullanılarak üretilen şekil bellekli alaşımların farklı tasarımlar ile savunma ve havacılık alanlarında birçok uygulamada hızla yaygınlaşacağı değerlendirilmektedir.