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The Effect of Different Parameters on Shape Memory Alloys

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Abstract

Shape memory alloys' characteristics are different from ordinary materials because they can memorize their pre-determined shape, thus they are excellent candidates for different applications. In this review article, the most interesting parameters that researchers are using in their investigation have been highlighted. Also, the popular techniques used for the characterization of shape memory alloys have been described. The diagrams and sketches can show a clear view of metallurgies and related research areas.

Keywords: Shape memory alloys, diagrams, sketches, characterization process

1. INTRODUCTION

Shape memory alloys (SMAs) have been found in the 20th century and they merged with technology whenever NiTi (nitinol) was discovered in Naval Ordnance Laboratory [1]. Nowadays, various SMA's families are known and for different applications, their properties have been changed. Several parameters are involved to change SMA's characteristics, such as compositional rate, type of constituents, and microstructure. The effect of different parameters on mechanical, thermal, and other physical properties of SMA can be determined using some specific measurements.

Different techniques have been developed for fabricating and characterizing different properties of SMAs. Figure 1showed the most important techniques utilized for producing NiTi SMAs. The different methods, including VAR, VIM, EBM, CS, SHS, HIP, SPS, MIM, SLS, SLM, and LENS, which are Vacuum Arc Remelting [2], Vacuum Induction Melting, Electron Beam Melting, Conventional Sintering, Self-propagating High-Temperature Synthesis (combustion) Synthesis, Hot Isostatic Pressing [3], Spark Plasma Sintering [4], Metal Injection Molding, Selective Laser Sintering [5], Selective Laser Melting [6], and Laser Engineered Net Shaping [7], respectively.

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There are many review articles that have detailed about various applications and important results. Jani et al. [9] described a general view of SMA, some applications, and opening research gates for investigation. Nespoli et al. [10] overviewed the potential of SMAs to develop miniature mechanical devices. Cisse et al. reviewed the modeling methods for modeling complex bearing in SMAs [11]. Follador and colleagues discussed the general technique for fabricating SMA-based actuator springs [12]. However, there are few studied that mentioned different influenced parameters on SMAs and the main ideas about characterization.

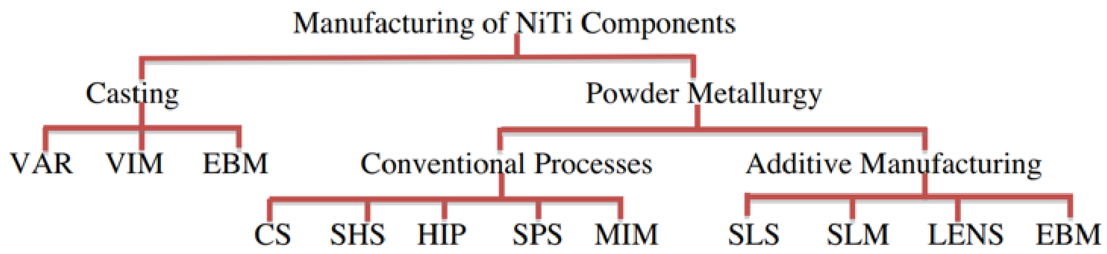


Figure 1 Importance methods used for fabrication NiTi SMA [8]

In this review, SMA has been defined and the superior characteristic has been specified. Some related information about SMA has been described. Also, some application has been given to show the importance of SMAs for technology. In addition, the influence of different studied parameters has been highlighted with some literature review. Finally, the measurement techniques and some important instruments have been clarified in terms of clear sketches with important basics about their operation.

2. SHAPE MEMORY ALLOYS

Some fundamental information needs to understand the shape memory alloys (SMAs), i.e. the alloy should be well defined and also the prefix (shape memory) should be explained through some diagrams.

2.1. Metals

Metals are a group of materials that have some unique characteristics compared with the other materials. One of the significant parameters in the electrical nature of the metals. Since the metal elements have an ocean of free electrons, so this group of elements can transfer electricity via the electrical carrier extremely more than the other groups.

2.2. Metal Alloys

Alloy is a combination of two or more chemical elements to share their physical characteristics. Mostly, the materials are alloyed with other elements to change the properties of the materials for different applications. When a material is alloyed with another element, the dopant elements distribute all over the materials in either substitute, interstitial, and/or cluster form. It should be kept in mind that the two different constituents do not chemically bond but they only share their physical properties, i.e. melting temperature, heat capacity, electrical and thermal conductivity.

2.3. Shape Memory Alloys

Sometimes by doping the material with a particular amount of dopant element, the alloy may achieve a new property, which existed previously in neither host nor dopant material. A characteristic that a shape memory material is based on their new formation of a superlattice crystal structure. Like other metals, a shape memory alloy (SMA) has a parent solid phase, is called austenite phase, which is a high similarity, and normally it has a cubic crystal structure. Furthermore, the SMA has another crystal structure, which is a non-cubic crystal structure and is stable at lower temperatures. The lower temperature is so-called martensite phase [13, 14]. Martensite has twinned crystals that can transform into detwinned martensite whenever an external load is applied. Thus the induced strain can be saved in the detwinned martensite variants and can be recovered through heating to the austenite temperature region. Therefore, the aforementioned characteristic can give a shape memory property to the SMA, which is known as a shape memory effect (SME). In addition, superelasticity (pseudoelasticity) is another shape recovery feature that can occur in the austenite phase without the heating process [15].

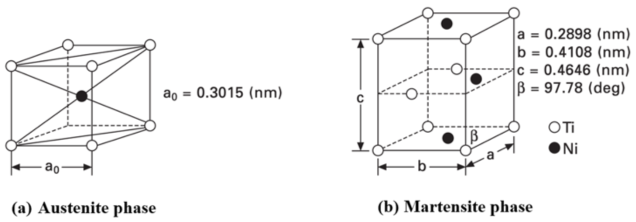


Figure 2 (a) Austenite and (b) martensite phase of NiTi shape memory alloy [16]

Figure 2 shows the austenite and martensite crystal structure of a particular SMA. They have various site arrangements and different lattice parameters. The phase transformation in point of view of the crystal structure is shown in the schematic diagram (Figure 3). The phase transformation temperatures are austenite start, austenite finish, martensite start, martensite finish and martensite transformation temperature by deformation (i.e. maximum temperature at which martensite transformation occurs) Temperatures that are symbolized as A_s, A_f, M_s, M_f, and M_d, respectively [17-29].

2.4. Phase diagrams

In thermodynamics, the state of materials is defined with several parameters. Likewise, there are some parameters in metallurgy that should be controlled to monitor the physical characteristics of an alloy. Based on these parameters, three different phase diagrams are introduced in this review.

2.4.1. Temperature-Composition diagram

In this kind of diagram, the temperature and composition of the constituents are play the rule. A binary phase diagram is more popular, however in some cases the binary alloy doped by a third element with a constant ratio. There is a boundary line between liquid and solid-state of the alloy which can be different by changing the composition. The liquid-solid boundary has less important in the metallurgy of shape memory alloys.

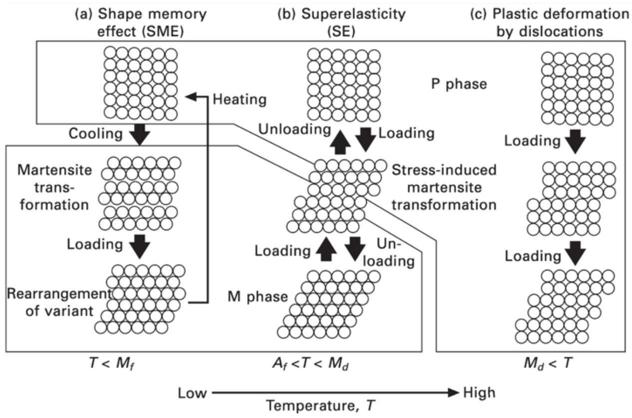


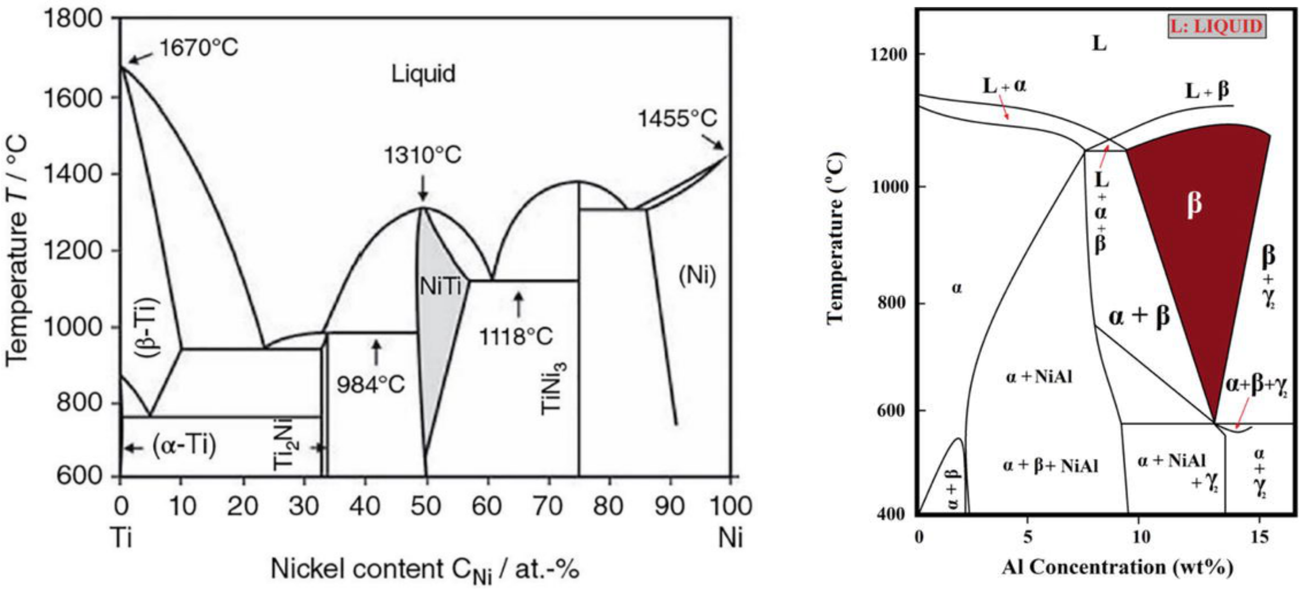
Figure 3 shape memory effect, (b) superelasticity, and (c) plastic deformation by dislocations [16]

Figure 4 shows two important SMA families. The binary NiTi SMA was found by William J. Buehler in the Naval Ordnance Laboratory [30]. In the NiTi phase diagram, it can be seen that there are many different regions with different solid phases. However, only the areas that occurred around eutectoids are important in shape memory studying point of view. For example, in Figure 4a there are just two regions, one in the high composition of titanium (Ti) and the second can be found in the nearly equiatomic composition of nickel and titanium. Although Ti-rich NiTi SMAs and nearly equiatomic NiTi SMAs have different characteristics, the Ti-rich NiTi SMAs are used comparably less than equiatomic NiTi SMAs because titanium is an expensive element in the markets.

CuAlNi shape memory alloys are also an important family that can be alloyed with other elements and/or can be treated to be used in different application areas. The composition change and treatments can play with the existing phases shown in Figure 4. The austenite phase of CuAlNi SMA is called β-phase and martensite phase can be γ' phase with coarse martensite plates and/or β' phase with narrow (needle-shape) martensite plates. Figure 5 illustrates a Cu-Al-Ni SMA that showed the martensite phase. Although the microstructure of the martensitic phases is different, the crystal structure can be either twin or detwinned. It is also known that some precipitation phases can be obtained in the austenite and martensite phase which produced through a diffusion process and make a compound. The type, amount and shape of these precipitations can directly effect on the physical properties of the shape memory alloys.

2.4.2. TTT diagram

Isothermal transformation diagram or time-temperature-transformation (TTT) diagram shows how the cooling time can influence on the microstructure of the alloy. The diagram is essential for designing and making a decision about the production process. The temperature is kept constant (isothermal) during the measuring process. Figure 6 is a simple TTT diagram for $Ti_{48.7}$-$Ni_{51.3}$ (at.%) SMA which is found for experimental results and theoretical calculation [31]. The TTT diagram for some alloys, e.g. Fe-C, is more complicated.



(a) (b)

Figure 4 (a) Binary Ni-Ti alloy and (b) Ternary Cu-Al-3.0 wt. % Ni [32, 33]

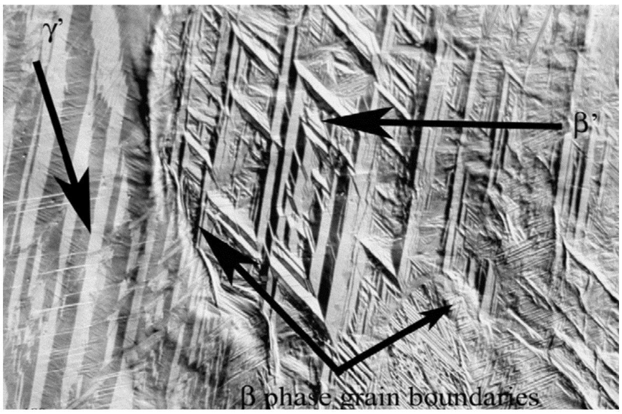


Figure 5 A combination of two different martensite phase in a Cu-Al-Ni shape memory alloy [34]

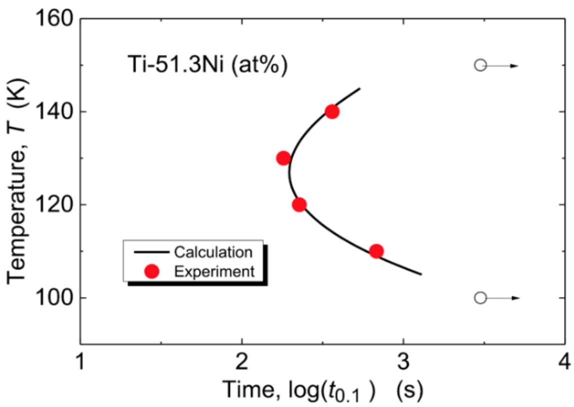


Figure 6 TTT diagram of $Ti_{48.7}$-$Ni_{51.3}$ (at.%) alloy [31]

For a duration of time at a specific temperature, the microstructures undergo changes because of a diffusion process of the constituents. Thus, the microstructure of an alloy, such as steel, can be monitored to obtain a desired feature for a specific application. However, the operation of an SMA, which is a phase transformation between austenite, and martensite, is supposed to be a diffusionless process. The properties of an ideal SMA should be constant, especially the phase transformation temperatures. Therefore, the microstructure should not be changed during phase transformation. Nevertheless, a real SMA is not thermally stable, because the process of heating and cooling take time, which provides the opportunity for the diffusion process and change the grain size of the SMA. The differential scanning calorimetry (DSC) results showed that there is no thermal stability during the cycling process [35-38].

2.4.3. Stress-strain diagram

Metal alloys are classified into ferrous and non-ferrous alloys [39]. In all cases, the metals showing a different stress-strain property. Figure 7 shows the stress-strain test for a brass specimen, whereby applying stress on the brass, it starts to deform. The deformation started with an elastic deformation which has a linear relationship between applying stress and the induced strain. In this stage, materials can recover their induced strain when the applying load is removed. Metals can be further deformed by increasing the applying load, however, when the deformation passed a critical point (yield point) it cannot recover the all induced strain. Thus the predetermined shape cannot be recovered through external treatments, such as hot or cold working. Also, some residual stress can release through heat treatments [40, 41]. Shape memory materials, on the other hand, can recover their original shape spontaneously [42-44].

Figure 8 shows the schematic representation of traditional material and two superior behavior of an SMA. In all cases, there is an elastic behavior, where the materials can recover almost all the induced strain. However, when the applying stress is increased, the material deformed and finally is broken in a maximum applying force. After a material deformed it cannot recover the induced strain, while in SMA the material can recover its predetermined shape through shape memory SME or pseudoelasticity (superelasticity) [16, 45].

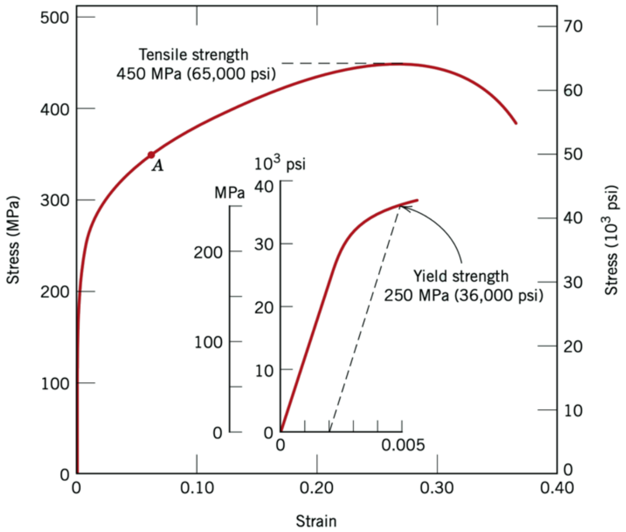


Figure 7 The stress-strain property of brass [39]

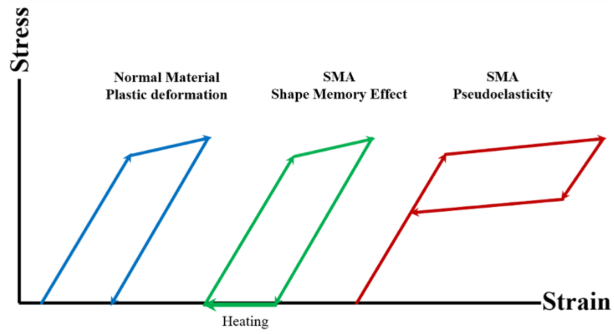


Figure 8 Schematic diagram of stress-strain behavior of a normal material and a SMA that can show SME and pseudoelasticity

2.5. Characteristics

SMAs similar to the other types of materials have physical properties, such as electrical conductivity, heat capacity, and thermal expansion. Also, there are some chemical properties such as corrosivity. Additionally, they have some superior properties that distinguish them from other counterparts. Shape memory effect and pseudoelasticity are two important characteristic that only belongs to SMAs.

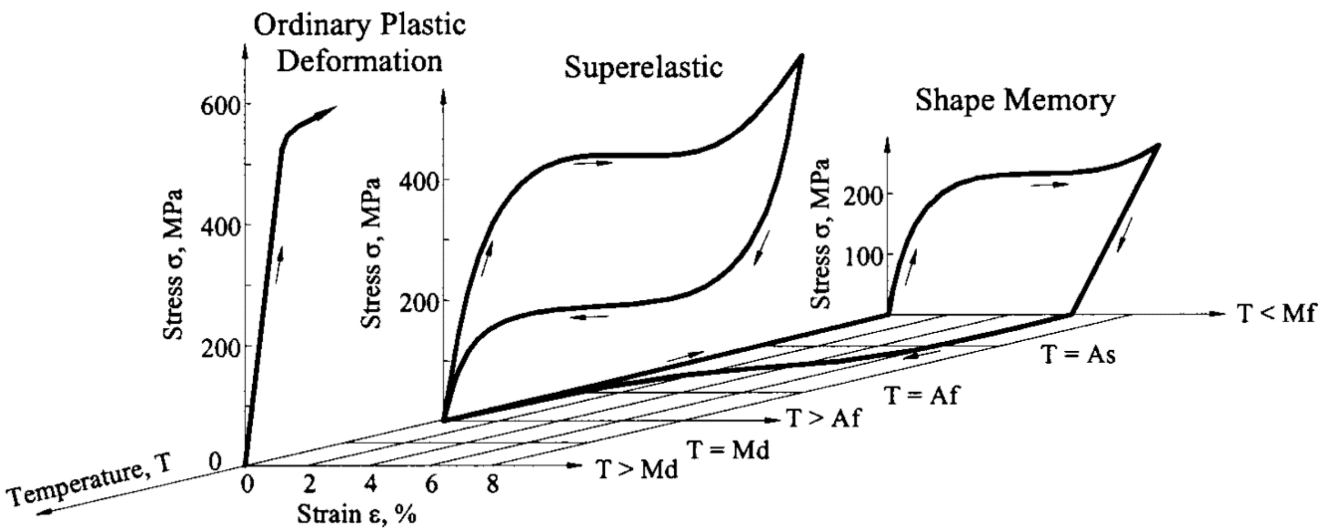


Figure 9 The diagram of an ordinary deformation above M_d temperature, superelasticity in the temperature range ($A_f - M_d$), and SME by heating the NiTi alloy to temperature up to A_f [46]

2.5.1. Shape memory effect

The shape memory effect (SME) is a shape memory recovering process. A deformed SMA in the martensite phase can be heated up to austenite phase to recover the original form, however, the shape cannot precisely be returned to its original. An equiatomic NiTi alloy can recover about 8%

of the induced strain [43, 9]. Figure 9 shows the process of shape recovery for an approximately equiatomic NiTi SMA. The SMA directly responded to the external stress and the deformation can stretch the atomic bonds, thus the energy can release whenever the external force was removed. When the magnitude of the external load exceeded, the SMA deformed like other ordinary materials, however, the slip did not occur for the applying load. The twinned martensite has only been detwinned, so by heating the SMA, the crystal structure of the alloy started to transform from martensite (twinned or detwinned martensite) to the austenite phase. This transformation started from A_s and it finished at A_f. Lastly, the shape-recovered SMA was cooled to the martensite phase in an extremely short time. In reality, some residual phases can remain, and austenite ↔ martensite transformation cannot completely be dissolved through the transformation process. Also, creating precipitation made of a compound can noticeably be detected using XRD measurements [47-49].

2.5.2. Pseudoelasticity

Pseudoelasticity or superelasticity (SE) is another potential shape memory ability of SMAs that the alloy can restore the strain through an isothermal process and without an additional stimulus process. It can be seen in Figure 9 that the characteristic occurred between A_f and M_d temperatures. The Psuelasticity is such that in the prior loading the SMA showed elasticity, like other ordinary materials, and by increasing the load the strain can increase with constant stress. In this stage, the austenite phase transformed into a detwinned meta-stable martensite phase, which absorbed a huge amount of loading. If the loading is not exceeded its limit, the SMA can return back to its original shape. Indeed some energy consumed during internal energy through the phase transformation process, which appeared as a different path of *austenite ↔metastable-martensite* and *metastable- martensite ↔ austenite* transformation.

2.6. Some Applications

Nowadays, several applications based on SMAs. Mostly, SMA is used as an actuator form. Fast response to stimulus and no need for external control are advantages of SMAs. Some applications in three main categories are shortly mentioned.

2.6.1. Medical applications

The first popular utilization is in the Orthodontic field. NiTi SMA, artificial known as NiTiNOL, is the best candidate which is better than the ordinary materials. Table 1 represents some selected mechanical properties of three different materials. It is obviously can be seen that NiTi alloy has bigger ultimate tensile stress and smaller ultimate tensile strain compared with stainless steel (316L), non-magnetic Cobalt-Chromium-Nickel-Molybdenum alloy (ELGILOY). In addition, the NiTi alloy has a comparably lower elastic modulus. These properties made the alloy to be chosen as the best candidate for orthodontic purposes.

Table 1
Some mechanical properties of stainless steel (316L), non-magnetic Cobalt-Chromium-Nickel-Molybdenum alloy (ELGILOY), and nitinol (NiTi) alloys. UTS and UTE are ultimate tensile stress and strain, respectively [50]

Alloys	Elastic Modulus (GPa)	Yield stress (MPa)	Yield strain (%)	UTS (MPa)	UTE (%)
316L	193	340	0.17	670	48
ELGILOY	221	450	0.20	950	45
NiTi	E_A =53.5, E_M =29.2	σ^{AS} =400	ε_y^s =9.0	1355	14.3

Figure 10a shows a schematic comparison for un-deformed stainless steel and nitinol shape memory alloy. The arc wire should be chosen such that it has sufficient stiffness and it should exert optimal stress on the displaced teeth. Thus, the induced stress, by the arc-wire, is classified to subthreshold (has no effect), suboptimal (has a low effect), optimal (the sufficient effect), and excessive (the undesired effect). It can be seen that the effective strain of nitinol is comparably more than stainless steel. Figure 10b showed the stress-strain characteristics of NiTi and stainless steel, where the stainless steel permanently deformed while the NiTi SMA instantly deformed and the induced strain is stored in terms of phase transformation from austenite to detwinned-metastable-martensite phase. The overall effective strain of NiTi SMA is several times bigger than stainless steel due to the pseudoelasticity effect. Therefore the SMAs are a selective candidate for orthodontic purposes [51].

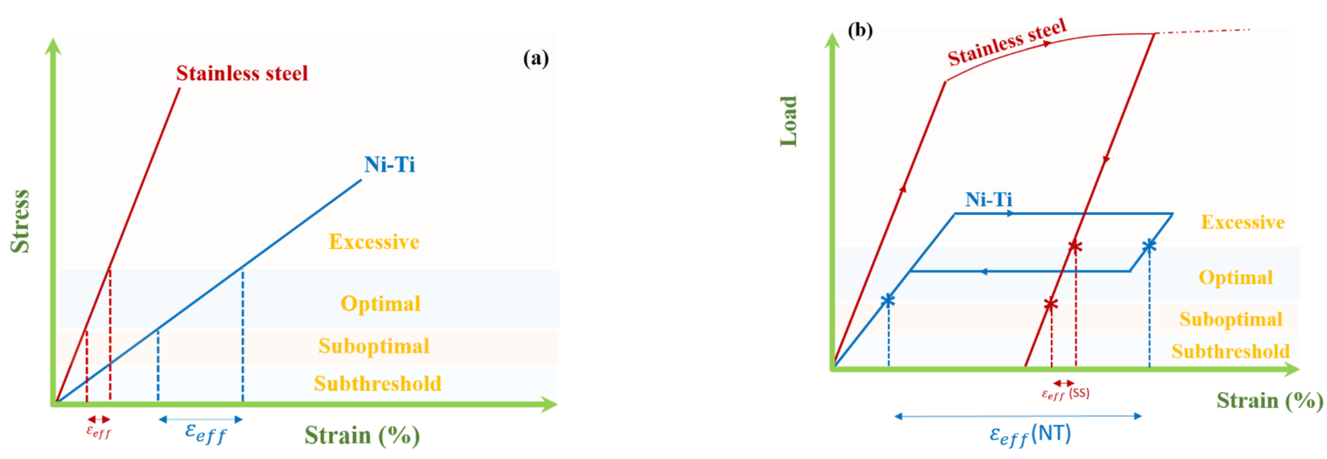


Figure 10 (a) A comparison of elasticity between stainless steel and NiTi SMA (as an ordinary material); (b) compare the strain recovery after deforming stainless steel and NiTi alloy [13]

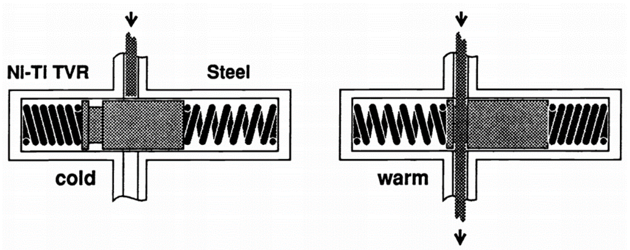


Figure 11 A steel-SMA system [52]

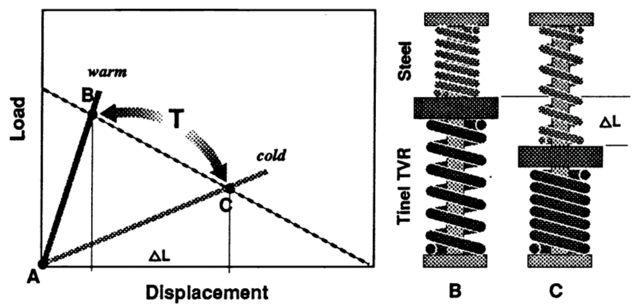


Figure 12 Ni-Ti spring properties for biasing forces

2.6.2. Engines and Aerospace

There is numerous application in this area, such as Smart Aircraft and Marine Project System Demonstration SAMPSON [53, 54], Rotor blade system [55], reconfigurable rotor blade [56], and nozzle configuration [56]. The most SMA-applications is used in term of the actuator. For example for making a cooling system based on temperature, a combination of steel and a SMA, such as NiTi alloy, can make a smart independent valve system (Figure 11). In low temperature, the steel-spring has more stiffness compared to the NiTi alloy, so the valve is closed and coolant does not flow into the engine. When the temperature increases the NiTi transforms from martensite to austenite phase, and therefore its stiffness becomes bigger than the steel. In such condition, the NiTi-based spring pushes the steel-based spring and consequently the valve is opened for passing the coolant into the engine. Figure 12 displays the austenite and martensite response of a NiTi alloy to a biasing load. The austenite phase has bigger stiffness compared to the martensite phase.

2.1. Heat treatments

Some physical properties of a SMAs, such as phase transformation hardness, stiffness, electrical resistivity and magnetization, can be influenced through heating (or cooling) process. Since, the type and size of microstructure is one of important factors to give different characteristic to a SMAs, so by controlling these parameters their physical properties can be monitored in a specific range. There are also isothermal process, which depend one time and temperature called aging. On the other hand the process of cooling is one of key point to determine the microstructure and hence to change the SMAs' characteristics.

3. DIFFERENT STUDIED PARAMETERS

SMAs response to different physical stimuli and phenomena, such as heating, electrical and magnetic fields, oxidation, and mechanical stress. In following sections some parameters are explained with a recent literature review for each.

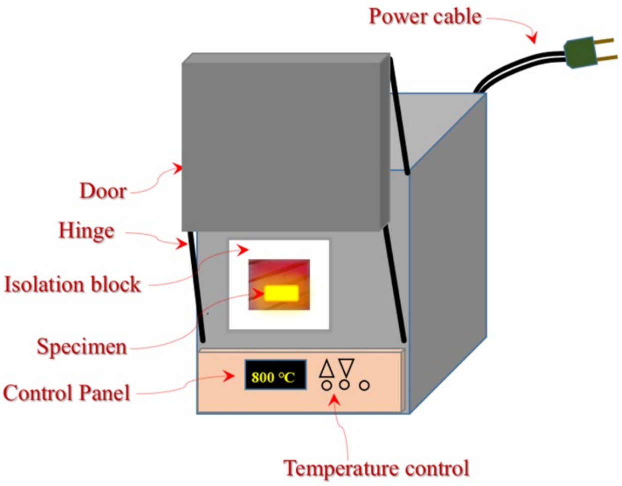


Figure 13 A sketch of furnace for aging SMA

3.1.1. Aging

Aging is a thermal process, where a specimen is taken in a particular temperature for a determined time [28]. A convenient furnace need for heating up a SMA. The furnace should be designed with some particular features. The temperature and the sample atmosphere should be under control. Generally, lab furnace uses an electrical heater to heat up and they have a panel to set the temperature and time of operation. Figure 13 illustrates a sketch of a furnace used for aging SMAs. It has an isolation block that is passive and can tolerate high temperatures. The atmosphere should be controlled to enhance the efficiency of the aging process. In addition, vacuolization avoids the specimen from oxidation in high temperatures. However, the capsulation of the sample is another way for those furnaces that have no atmosphere control facilities.

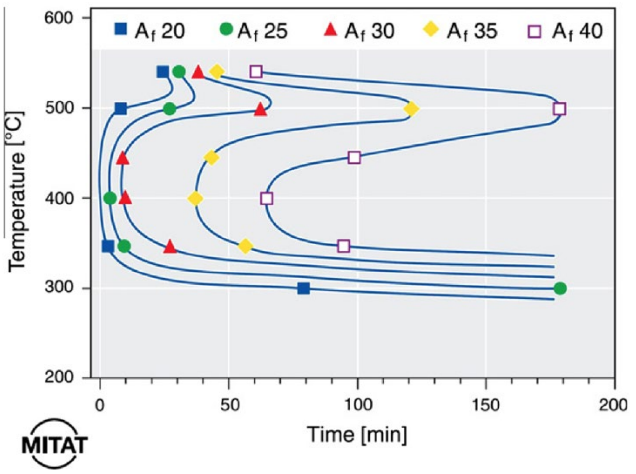


Figure 14 Aging and cooling process as a function of time for austenite finish temperature (A_f) [57]

The temperature of aging can be changed to investigate its influence on some physical properties of SMAs. There is a temperature-composition-phase diagram for most binary SMAs, where the melting temperature, eutectoid temperature as a function of composition is determined. The metallurgists can choose the effective temperature of aging, however, for some SMAs with more than two constituents, the researchers should investigate different temperatures using trial and error. Figure 2 shows the effect of aging and cooling process on austenite finish temperature. The TTT diagram has obtained for $Ni_{49.2}Ti_{50.8}$ (at.%). The alloy has been aged at 300 and 500 °C for 2-180 min with initial A_f =11°C. The best-obtained aging temperature is nearly 400 °C because the most precipitation occurs in this temperature and there is a balance was achieved between the driving force and diffusion rate needed for phase transformation. It is found that to obtain a full martensite phase in the lower temperature, the alloy should have a high austenite finish temperature. However, for the temperature <500°C, a higher diffusion rate happens and consequently the time needed for transformation to be ended diminished [8].

Sari et al. [58] studied the effect of aging on a Cu-Al-Ni-Mn SMA in three different temperatures (300, 400 and 500 °C). They realized that some precipitation, including bainite, α, and γ_2 phases, were obtained. The phase transformation temperatures were decreased with increasing time and temperature of aging, while the hardness values were increased. On the other hand, the time of aging can impact on a SMAs characteristic. Shamimi et al. [59] reported that increasing the time of aging in different temperatures increased the forward and reverse phase transformation temperatures.

3.1.2. Quenching

One of the aforementioned phase diagrams is the TTT diagram (temperature-time-transformation) which showed how the time of cooling can affect the obtained phase in the lower temperature. Generally, the time of cooling is taken in a fraction of second, in order to achieve a martensitic microstructure in SMAs. Moreover, the phase transformation from austenite to the martensite phase is a diffusionless phase transformation process, thus the time of cooling should be as short as possible. Fast cooling, or quenching, depends on the medium where a high-temperature SMA in the austenite phase immediately loses a huge amount of heat energy. Two of the important parameters, which should be kept in mind, are the temperature difference and heat capacity of the medium. In this process, the temperature of cooling-medium is supposed to be constant, therefore its amount should be comparably more than the SMA.

Dagdelen et al. [60] studied some physical properties of Cu–13Al–4.5Ni–1.5Ti (wt. %) SMA, where subjected to heat treatment at 930 °C for 30 min. They used liquid nitrogen, alcohol, and iced-brine mediums that had –196, 0, and 6 °C, respectively. They reported that alcohol and iced brine increased phase transformation temperatures about 100 K. Also, grain boundaries were more identifiable for the alloy in which quenched into the alcohol and iced brine. Likewise, Saud et al. [61]found that the quenched Cu-Al-Ni-Fe SMA into the oil had the best grain refinement because it has a higher cooling rate compared to ice-brined water.

3.2. Changing Compositions

Materials with pure elements have particular properties, which may not be used for all applications, thus alloying with different amounts of other elements can give novel features to the materials. SMAs are also can obtain new characteristics by alloying with different elements [62, 22]. Khalil-Allaf et al. [63] studied the different composition of NiTi alloy. They found that the enthalpy and entropy changes of martensitic transformation, elastic energy, and chemical forces were diminished with increasing Ni content in the SMA. On the other hand, Buytoz et al. [21] reported that enthalpy and entropy changes of NiTi-Hf alloy decreased with increasing Hf content, while PTTs the alloy increased. Also, they found that the alloy with different composition showed different oxidation behavior. Mehrabi et al. [64] added tungsten (W) into nitinol (Ni-Ti) SMA to investigate some physical behavior, such as hardness, through changing the composition. They reported that the hardness of the alloy significantly increased by increasing tungsten content. Zheng et al. [65] alloyed NiTi with Ag for medical investigation. They found that the strength and bacteria adhesion was improved by adding Ag into the NiTi SMA, whereas, the corrosion resistance and cyto-biocompatibility had not been significantly affected.

3.3. Mechanical treatments

The mechanical treatment does not affect the microstructural types in a SMA, but it can grain refinement and, thus, these treatments can influence the SMA characteristics. There are two popular methods of rolling, cold and hot rolling. Sometimes, the rolling process makes some micro-cracks which is one of the disadvantages of the method. It may be followed by annealing at a particular higher temperature. Sharifi et al. [66] studied the effect of cold rolling on an equiatomic-NiTi SMA. Some samples with different thicknesses were produced at room temperature, where the stiffness was increased by reducing the thickness. They realized that the dislocation of the alloys was increased by applying a rolling process. The obtained amorphous alloys were completely crystallized by annealing at 400°C for 1h. The plateau region in the stress-strain test was obtained only for those alloys that annealed after cold rolling.

3.4. Coating

The coating is one of the attractive processes to improve some properties of a SMA, e.g. corrosion resistance and biocompatibility. Hu and colleagues [67] produced an ultrafine grain layer on NiTi SMA consisting of nanocrystallites. They improved the hardness and wear resistance of the alloy by grain refinement of the surface. Furthermore, the coated alloy had lower friction coefficients compared with uncoated coarse grain NiTi alloy. Cheng et al. [68] coated NiTi with biocompatible-Ti. The corrosion resistance obtained by the electrochemical method showed that the alloy achieved an excellent corrosion resistance property due to obtaining a thin oxide film on the NiTi alloy, which is a passivated material. Maleki-Ghaleh et al. [69] electrophoretically deposited hydroxyapatite on a NiTi SMA with various electrical potential differences. The in-vitro study results showed that the samples coated at 60 V had comparably better protection against Ni (toxic element) release into simulated body fluid (SBF).

4. CHARACTERIZATIONS

All aforementioned effective parameters are characterized through some advance types of equipment. The common and important devices includes Differential scanning calorimetry (DSC), thermogravimetry / differential thermal analysis (TG/DTA), x-ray diffraction (XRD), scanning electron microscope (SEM)- energy dispersive x-ray spectroscopy (EDS), metallurgical microscope or optical microscope (OM), Vickers microhardness, and the stress-strain measurements; also, there are different in vitro (or in vivo) studies to investigate the biocompatibility of materials.

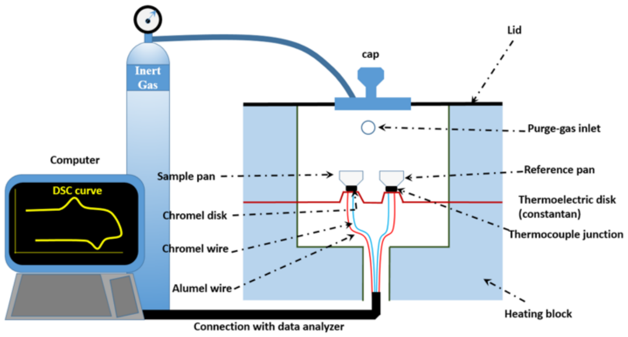


Figure 15 Sketch of a Differential Scanning Calorimetry (DSC) connected with a data analyzer

4.1. Thermal Characteristics

4.1.1. DSC

Differential scanning calorimetry (DSC) is one of the fundamental tests for measuring phase transformation and obtaining some thermodynamics properties of a SMA. Figure 15 shows a sketch of a DSC measuring system with a DSC, inter gas, and a data analyzer (computer). The inert gas flowed into the DSC chamber to minimize the oxidation. There are two unique crucibles put on the sensitive thermocouple. The reference pan is left empty and the specimen is taken inside the Sample pan. The heat flow recorded as a function of temperature, thus and change in heat flow gives information about a physical (or chemical) reaction, which the phase transformation between austenite and martensite is what a researcher is looking for in a SMA. The computer utilizes a special software program to analyze the DSC measurement data. The phase transformation temperatures and enthalpy changes of the transformation are the main DSC results of a SMA.

4.1.1. TG/DTA

Thermogravimetric/differential thermal analysis (TG/DTA) is another tool that can be used for obtaining some physical properties, such as phase transformation of heating, oxidation, obtaining an oxide layer in an isothermal process. Similar to DSC, TG/DTA device includes two unique crucibles, one for the sample and the other is the reference. Mass gain/loss as a function of temperature and temperature change as a function of time are two important results that can be achieved with this device. The atmosphere can be controlled by injecting inert gas or maybe left naturally. Normally, TG/DTA is used for phase transformation in high temperatures [70].

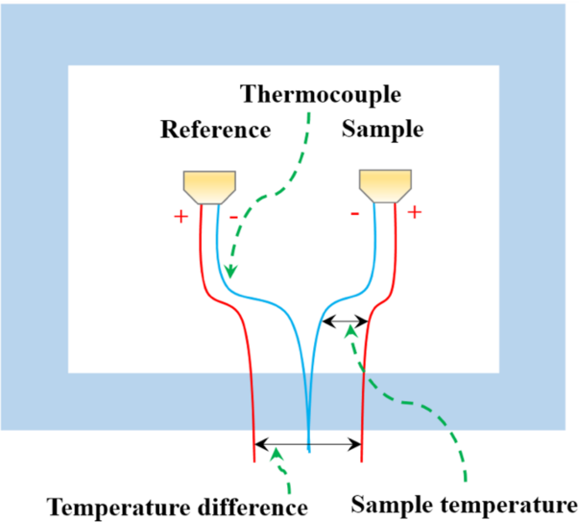


Figure 16 A sketch of a DTA

4.2. Crystal and Microstructural Analysis

4.2.1. XRD

X-ray diffraction is one of the effective techniques that is used by many material Scientifics. Basically, an x-ray source is needed which is generally obtained from the first electron transition of the Cu element, known as k_{α} (with a wavelength of 1.5406 Å [21]). The incident x-ray reflects from different atoms. The atoms work as a plan, where the interplanar distance can be found in the XRD result (Figure 17). The wavelength of reflecting x-ray is not changed and the collision between x-ray and atoms is an elastic collision. The diffracted x-rays detected by a special detector and finally, the pattern is analyzed by some calculations so by using some database the obtained peaks can be indexed. Since metals have a crystalline structure, so the pattern consists of peaks, where the angle of detection, wideness, and intensity of the peaks give valuable information about the crystal structure of the material. The XRD is not for a single atom crystal structure, but it can be used for complicated molecules and to find different compounds [71]. Some phases after phase transformation will not completely transformed, such as austenite ↔ martensite phase, and the DSC results cannot give any information about the residual phases and other precipitations that can influence the physical properties of a SMA.

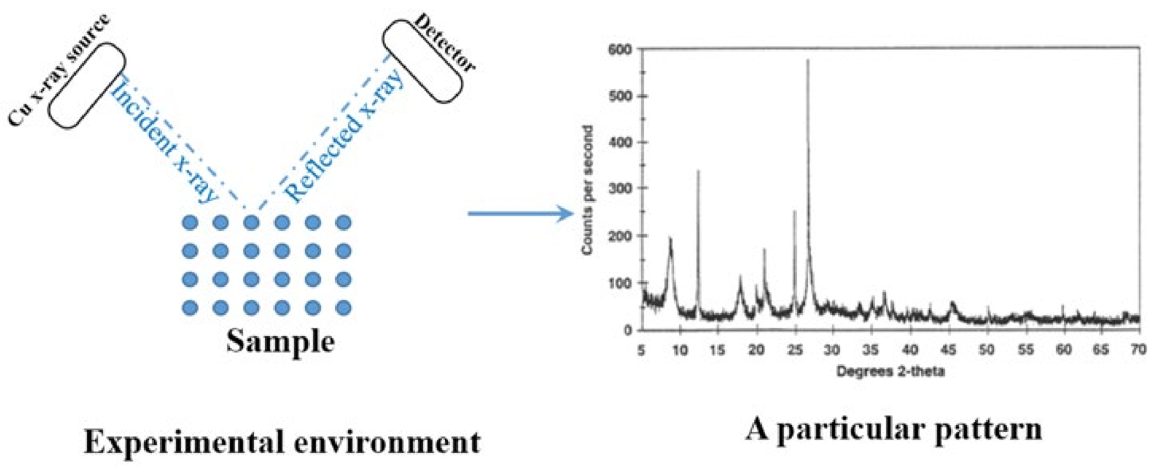


Figure 17 A schematic representation of an XRD device and an output x-ray pattern

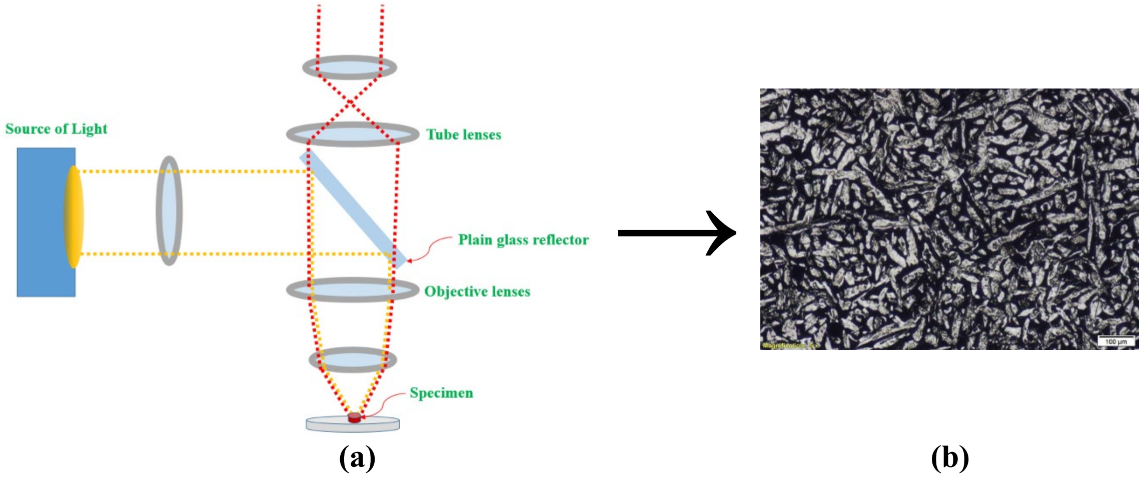


Figure 18 (a) Schematic representation of an optical microscope, (b) a particular CuAlNi-based SMA [72]

4.2.2. Optical Microscope

An optical microscope (OM) is one of the pre-examining microstructures of SMAs. An OM consist of a source of visible light that illuminated on the specimen's surface. The scattered light is collected with objective lenses and partially transmitted through plain glass reflector. A clear image can be achieved with magnification about 1000x.

Figure 18 represents a schematic diagram of an OM and a specific image obtained for a CuAlNi-based SMA. The microstructure can be investigated for the selective range of magnification. However, most professional researchers used to use a scanning electron microscope, which can give more crystal images, moreover using the EDS facility the compositional rate also can be obtained.

4.2.3. SEM-EDS

A scanning electron microscope (SEM) is a type of electron microscope that frequently is used by many researchers. Its magnification is thousands as more as an optical microscope. Electrons are produced by a tungsten filament (electron gun), then the electrons are arranged and accelerated with an anode (has a positive charge). The electrons passing through a magnetic lens and then using a scanning coil the concentrated-high speed-electrons are directed to the different position of the specimen's surface. A part of electrons are scattered from the surface, which is known as a backscatter electron, and they detected by a detector to obtain an image (SEM image). Another part of the incident electrons can penetrate into the specimens and since they have high energy, so they can kick and eject a core electron. The electrons in the upper energy levels transmitted into the lower energy level that produce an electromagnetic wave (EM). Then the EM is analyzed to obtain the type of elements in the specimens. The concentration of most metallic elements can be determined by energy dispersive x-ray spectroscopy (EDX or EDS) [73-75].

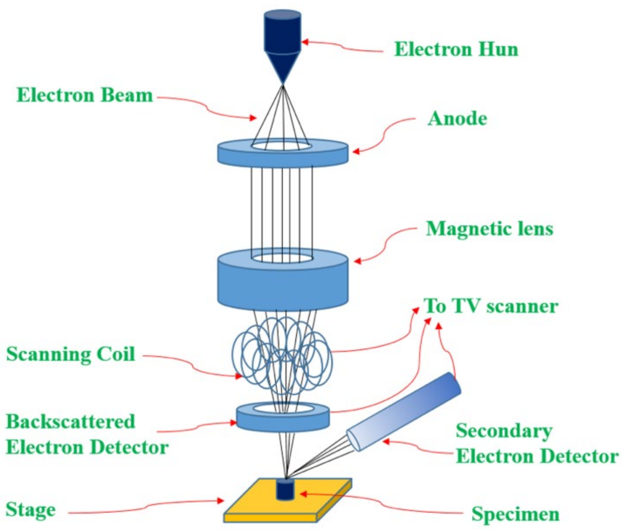


Figure 19 Schematic diagram of a scanning electron microscope

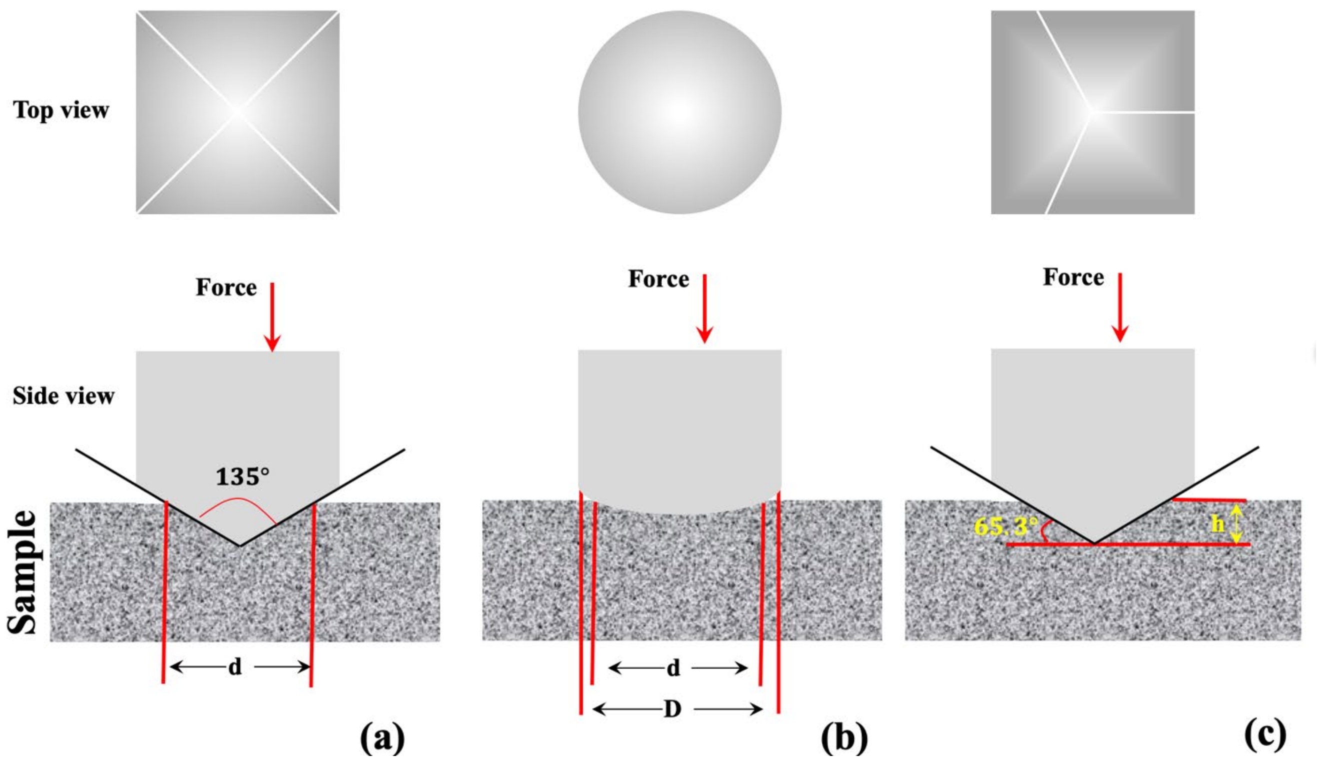


Figure 20 Schematic diagrams of different types of microhardness testing instruments, (a) Vickers microhardness (four-sided pyramid tip), (b) Brinell microhardness (spherical tip) and (c) Nanoindentation (Berkovich tip) [82]

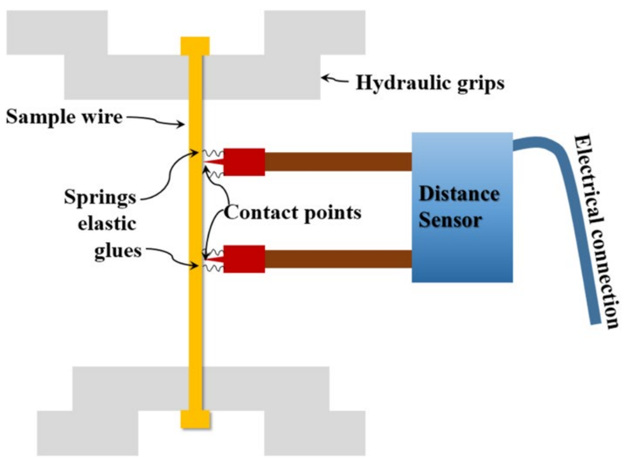


Figure 21 Schematic set-up of tensile testing [83]

4.1. Mechanical testing

4.1.1. Microhardness

There are various types of measurements for testing hardness of a sample, including Vickers hardness (HV), Brinell hardness (HB), Knoop hardness (HK), Janka hardness, Meyer hardness, Rockwell hardness (HR), Shore durometer hardness, Nanoindentation, and Barcol hardness testing. Figure 20 represents Vickers microhardness, Brinell hardness, and Nanoindentation indentation testing. The aforementioned techniques depend on different indentation and different calculations. There are defined standards for each technique [76-78].

ickers microhardness is one of popular testing because its calculation does not depend on the size of the indenter. The units of Vickers microhardness are Vickers Pyramid Number (HV) or Diamond Pyramid Hardness (DPH), and sometimes it can be converted to SI unit (Pascal) [79-81].

4.1.2. Tensile test

In this mechanical test, the samples should be prepared in a standard form (Figure 21). When the sample is pulled from its ends it will firstly be elongated elastically, then it reached yield point. By. On the other hand, the compression test is another popular test for ceramic materials, such as concretes.

Figure 22a and Figure 22b shows the schematic diagram of a stress-strain test for an ordinary metallic material and for a SMA that showed pseudoelasticity. The real experimental data for aluminum and steel (ordinary material) and an approximately equiatomic NiTi SMA is given in Figure 4.8c and Figure 4.8d, respectively. The tensile test gives information about many mechanical properties of materials, such as modulus of elasticity, ductility, stiffness, superelasticity behavior (in SMAs).

4.1. Biocompatible tests

There are several applications of SMAs that need to be biocompatible, thus the SMAs should be checked out some tests, including carcinogenic, genotoxic, mutagenic, cytotoxic, allergic, and corrosion behavior [84]. For example, corrosion tests can give information about the reactivity of a SMA with different environmental conditions.

The specimens, in the implanted SMAs, should be put inside a simulated body fluid (in-vitro study) or, their behavior can be investigated inside a real medium (in-vivo study). Figure 23 shows a schematic comparison between the in-vitro and in-vivo studies. Since the in-vitro study is easier to control parameters, so it is used more frequently in this research area. Although there are many passive elements that have high biocompatibility, some functional materials are beyond this scope. Therefore material scientists should treat them through some techniques, e.g. coating is one of a practical method to reduce the toxic release into the alive tissues [87-90].

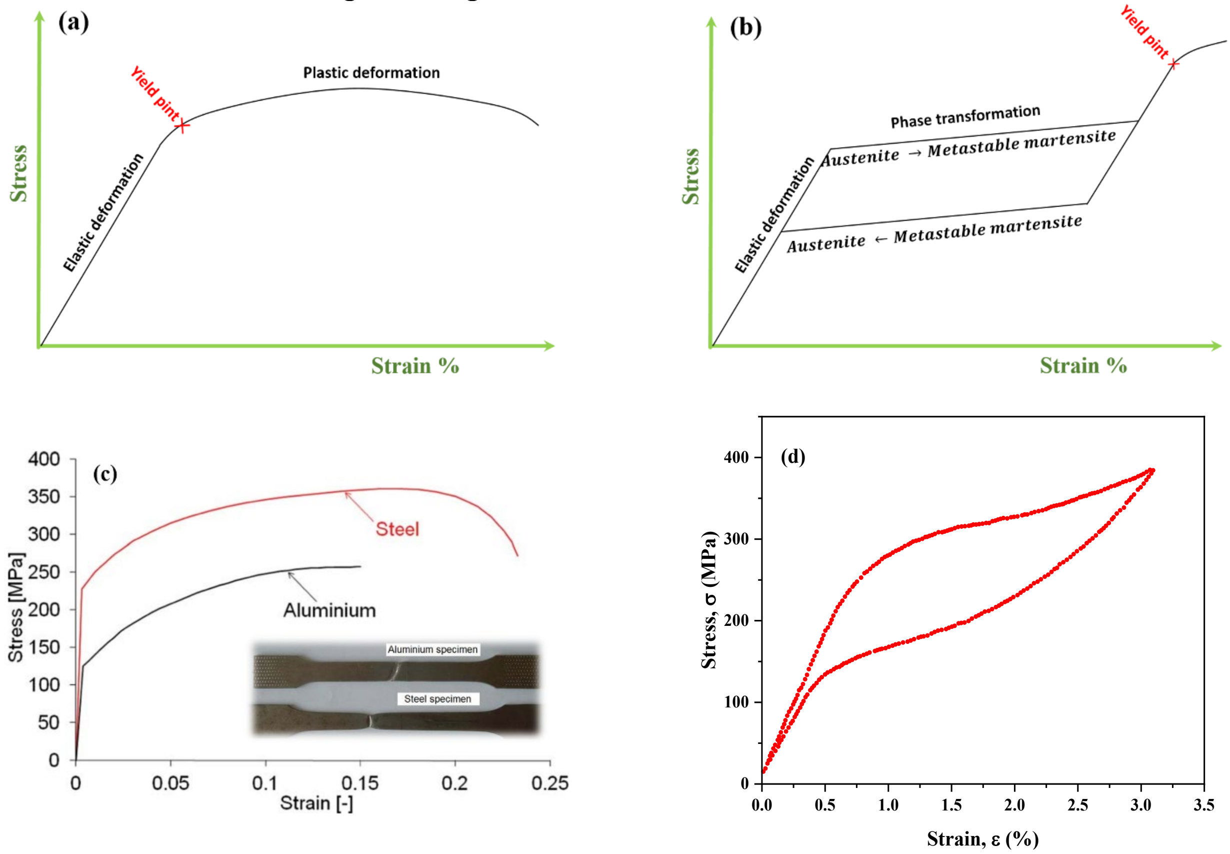


Figure 22 Stress-train test for (a) an ordinary metallic material and (b) for a SMA. (c) Experimental result for a particular aluminum and steel [85], and hysteretic behavior of $Ni_{51}Ti_{49}$ (at.%) SMA obtained at 303K [86]

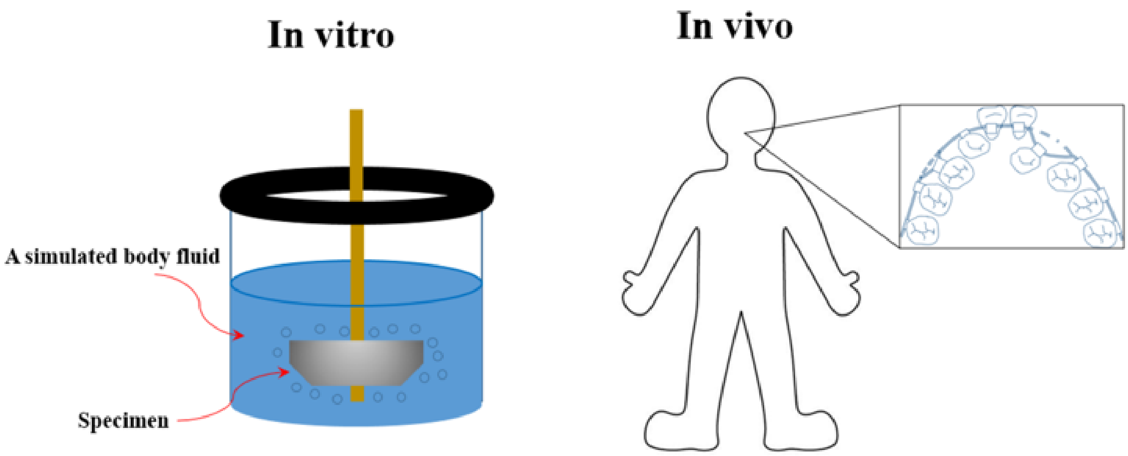


Figure 23 A comparison between in-vitro and in vivo study

5. CONCLUSION

In this review, different shape memory alloys' characteristics were explained. Also, the most extended effective parameters on shape memory alloy were reviewed. In addition, the techniques used for the characterization of SMAs were defined with some related devices. The basics of the device operation were explained through some sketches. This review can open a gate for new researchers, who have not enough information in this field.

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Authors' Contribution

Conception: INQ-SSA, Design: MK-FD,
Supervision: FD-MK, Materials: INQ,
Data Collection and/or Processing: INQ-SSA,
Analysis and/or Interpretation: INQ-MK-FD
Literature Review: SSA,
Writer: INQ, Critical Review: INQ-MK-FD-SSA

The Declaration of Ethics Committee Approval

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The authors of the paper declare that they comply with the scientific, ethical and quotation rules of SAUJS in all processes of the paper and that they do not make any falsification on the data collected. In addition, they declare that Sakarya University Journal of Science and its editorial board have no responsibility for any ethical violations that may be encountered, and that this study has not been evaluated in any academic publication environment other than Sakarya University Journal of Science.

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