

Effect of Heat Treatment on Mechanical Behavior and Stabilization of Super-Elasticity of Nitinol Alloys Used in Smart Actuators

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Abstract

In this work, the effects of heat treatment on the mechanical behavior and superelasticity stability of nitinol alloys used in smart actuators were studied. Heat treatment was carried out at different temperatures via solution treatment, aging at 300°C, 400°C, and 500°C. Aging at 400°C showed the optimum results to achieve a balance between strength and elasticity as a clear plateau appeared to express the behavior of superelasticity while the austenitic phase transformation temperature reached 16°C. This ensured the existence of the austenitic phase at room temperature, which is the fundamental criterion for medical and industrial applications. Also, the results showed reasonable enhancement in the fatigue life for the samples aged at 400°C and 500°C as the enhancement was three times the as-prepared sample. This confirms the significance of the fine control of heat treatment parameters to produce nitinol alloys for the targeted applications.

Keywords: Nitinol alloys; Mechanical behavior; Super-elasticity; Smart actuators

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1. Introduction

Smart materials represent a developed category of engineering materials with controlled response to external effects such as mechanical stress, electric field, or thermal fluctuations. This response enables these materials to perform simultaneous physical and operational functions with no need for sophisticated external control systems [1-3]. These materials have gained an increasing interest in modern engineering applications due to their pivotal role in system miniaturization as well as enhancing performance efficiencies in important sectors like avionics where smart materials are employed to modify the dynamic morphing wings [4,5]. As well, these materials are employed in biomedical engineering to design the self-expanding stents and orthodontic wires that provide consistent orthodontic forces. Smart materials are also employed in automobile manufacturing to reduce vibrations and noise via adaptive damping systems [6,7].

Smart materials are classified into three categories; piezoelectric materials, magnetostrictive materials, and shape memory alloys. Piezoelectric materials can generate electric potential when subjected to mechanical stress and show mechanical deformation when subjected to electric potential [8-10]. Accordingly, this category of smart materials are ideal as micro-actuators and super-sensitive probes. A magnetostrictive materials can change its dimensions in response to an external magnetic field in addition to its ability to generate large forces and fast response in the applications of effective control of vibrations [11,12]. Shape memory alloys, such as nitinol, have the ability to restore their original shape before apparent deformation when heated up beyond the phase transformation temperature. Also, they show super-elasticity to hold irreversible large deformations that make them an optimum selection in smart actuators and bio-implanted devices, which require high biocompatibility and reliable mechanical performance [13-15].

Nitinol is a binary alloy composed of nickel and titanium with approximately equal atomic content (50:50). This alloy show unique physical and chemical properties when compared to the conventional metallic alloys used in the micro-engineering applications [16,17]. Amongst these properties, nitinol alloy shows high capability to resist corrosion due to the formation of a negative and surface-stabilized titanium dioxide (TiO₂) layer that induces excellent biocompatibility and makes nitinol alloy the first choice in bio-implants and vascular stents with no need for additional protective layers [18]. Furthermore, this alloy show featured fatigue resistance enabling it to hold millions of loading cycles in applications of actuators and micro-pumps without loss of performance [19]. Nitinol alloy also shows relatively low

elastic modulus when compared to conventional medical steel as the stress shielding is reduced in orthopedic applications and accelerates osseointegration [20-22].

Chemically, the high sensitivity of nitinol alloy to the variations in chemical composition and existence of dopants makes the fine control of fractional contents of nickel and titanium a crucial parameter in determining the phase transformation temperatures that make the fundamental of the most featured technological applications of these alloys [23,24] and these applications are based on two main effects. First, the shape memory effect means that the nitinol alloy can be deformed at low temperatures to keep its temporal shape and then completely returns its original shape when heated up beyond the austenite transformation temperature. This feature is invested in thermal and smart actuators operating without traditional electrical machines. Second, the super-elasticity appears when the alloy is used within a thermal range higher than the austenite transformation temperature to hold deformations of 8-10%, i.e., 10 times higher than the conventional metals, then automatically returned to its original shape soon after removing the applied stress [25-27]. Here, a stress plateau appears on the stress-strain curve and it makes nitinol alloy an ideal material for the self-expanding stents and orthodontic wires that provide consistent orthodontic forces, as mentioned before, in addition to the clot retrievers and smart prosthetic limbs that mimic the dynamic performance of living tissue [28].

In this work, the effects of heat treatment on mechanical behavior and super-elasticity of nitinol alloys are introduced and analyzed to determine the transformation temperature required for optimum performance of shape memory alloys and smart actuators.

2. Experimental Part

The experimental work includes three parts; sample preparation, heat treatment, and tests and characterization. Samples were prepared by cold work from nitinol alloy with atomic fractions of 55.8% Ni and 44.2% Ti (Fig. 1a). These samples were classified into four main groups. The first group contains the as-prepared samples without any heat treatment. The second group contains the samples thermally annealed at different temperatures (400°C, 500°C, and 600°C) for one hour then cooled in air. The third group contains samples of solution treatment including heating up to 850°C for 30 minutes then rapidly cooled (quenched) in water to form the martensite phase. The fourth group contains samples subjected to solution treatment, aged at different temperatures (300°C, 400°C, and 500°C) for three hours, and then cooled in air.

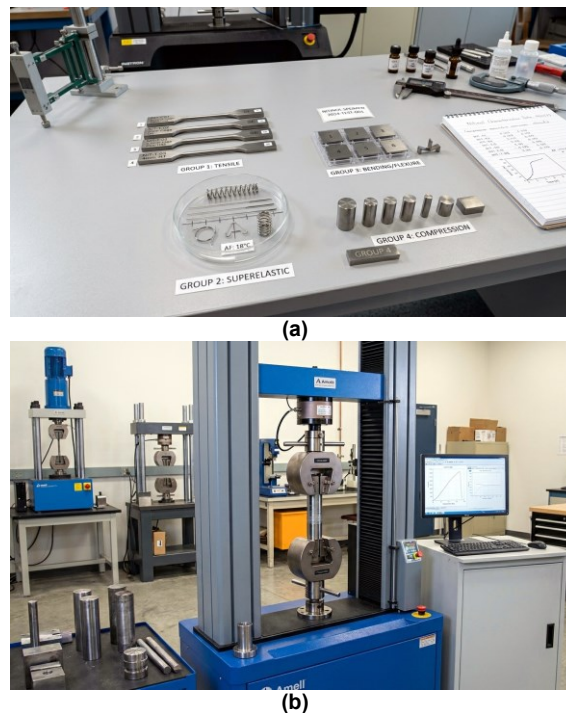


Fig. (1) (a) A photograph of the Nitinol sample groups prepared for mechanical tests and characterization, and (b) the universal testing machine used in this work

The four groups of samples were tested and characterized to introduce the effects of heat treatments on their mechanical behavior and microstructures. These tests include determination of transformation temperatures, scanning electron microscopy (SEM), tensile test, elongation, stress-strain curve, and fatigue test at cyclic loading using a universal testing machine shown in Fig. (1b).

3. Results and Discussion

Figure (2) shows the stress-strain characteristics for the as-prepared and annealed nitinol samples to introduce the effect of thermal annealing on mechanical behavior of these samples. All samples show similar behaviors with a direct proportionality until the failure point is reached. Thermal annealing leads to enhance the ductility with reasonable reduction in both yield strength and ultimate tensile strength. For the as-prepared sample, the curve confirms a relatively brittle behavior with a fracture strain of about 7% and maximum stress up to 680 MPa. This behavior is attributed to the accumulation of dislocations and residual internal pressures resulted from the previous forming processes without consequent thermal annealing. With thermal annealing at 400°C, a reasonable modification in ductility was observed as the strain at fracture increased to about 9% with slight decrease in the maximum stress to 670 MPa. This enhancement is ascribed to the initiation of stress relief as well as partial re-arrangement without complete recrystallization. This lead to maintain high hardness with an enhancement in the deformability. For the sample annealed at 600°C, the curve shows high increase in the strain at fracture of about 12.5% with an increase of about 79% compared to the as-prepared sample, whereas the yield strength has decreased to about 310 MPa at strain of 3%, which means a decrease to 28% compared to the as-prepared sample. This high ductility is mainly attributed to the complete recrystallization and formation of new grains free of dislocation and internal pressures, which reduces the dislocation slip resistance and stress required for the stress-induced martensite [29]. This is the fundamental phenomenon that interprets the reduction of stress curve at the initial deformation in shape memory alloys. When comparing the ultimate stress for the sample annealed at 600°C with that of as-prepared and 400°C-annealed samples, the reasonable decrease reveals that annealing at elevated temperatures – despite the large enhancement in ductility – causes large elasticity in the microstructure, which may be undesired in the applications requiring high stress values. This necessitates the selection of optimum annealing temperature according to the practical requirements to satisfy the required elongation, keeping sufficient mechanical strength for smart actuators and implantable medical devices.

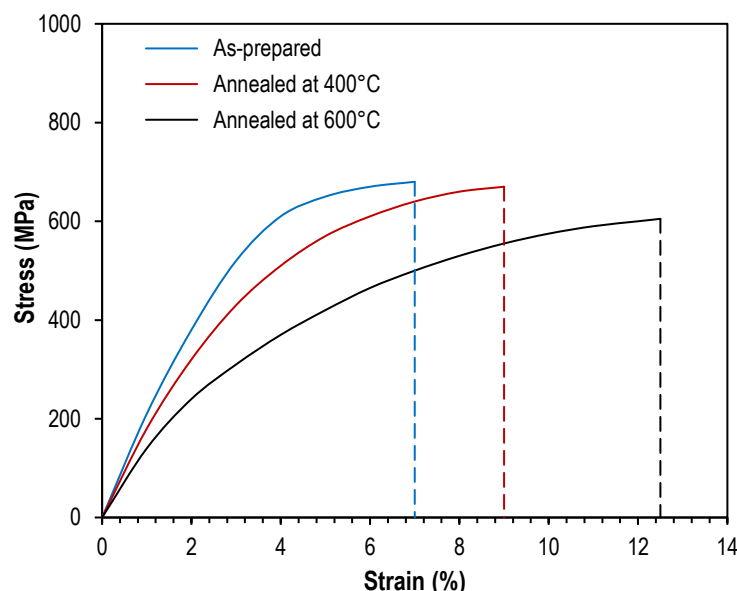


Fig. (2) Effect of annealing on the stress-strain characteristics of the nitinol samples

Figure (3) explains the apparent effect of aging parameters on the mechanical behavior and superelasticity effect of nitinol alloy with four different heat treatments. The sample annealed at 400°C exhibited the optimum mechanical performance and superelasticity with the highest ultimate stress of about 620 MPa at strain of 12% and elongation at break up to 14%. An apparent extended stress plateau

has appeared in the range of strain between 4% and 10%, which is a distinct feature for the superelasticity allowing the alloy to hold large reversible deformations without permanent damage. This distinct performance is attributed to the uniform precipitation of Ti_3Ni_4 nanoparticles during aging at $400^\circ C$ as these precipitants act as effective boundaries against the propagation of dislocations. Moreover, they provide homogeneous nucleation sites for stress-induced martensitic transformation that enhances the cyclic stability and reduces the hysteresis. In contrast, the solution treated sample showed lower efficiency behavior as elongation at break reached 10% with ultimate stress of 590 MPa. This is attributed to the absence of precipitants enhancing the microstructure and hence larger freedom for dislocation propagation and lower ability to reverse the deformation. The samples aged at $300^\circ C$ and $500^\circ C$ showed moderate performance as the elongation at break reached 12% and 10%, respectively, with ultimate stress of 600 MPa and 590 MPa, respectively. This is because aging at $300^\circ C$ is not enough to form the optimum density from Ti_3Ni_4 precipitants, while aging at $500^\circ C$ lead to particle coarsening that reduces their effectiveness in maintaining the microstructure and hindering the uniform phase transformation [30]. Consequently, the mechanical performance and superelasticity are reduced when compared to the aging at $400^\circ C$. These results confirms that the fine control of aging temperature is crucial to achieve the optimum balance between strength and ductility in medical and industrial applications of nitinol alloys based on their superelasticity.

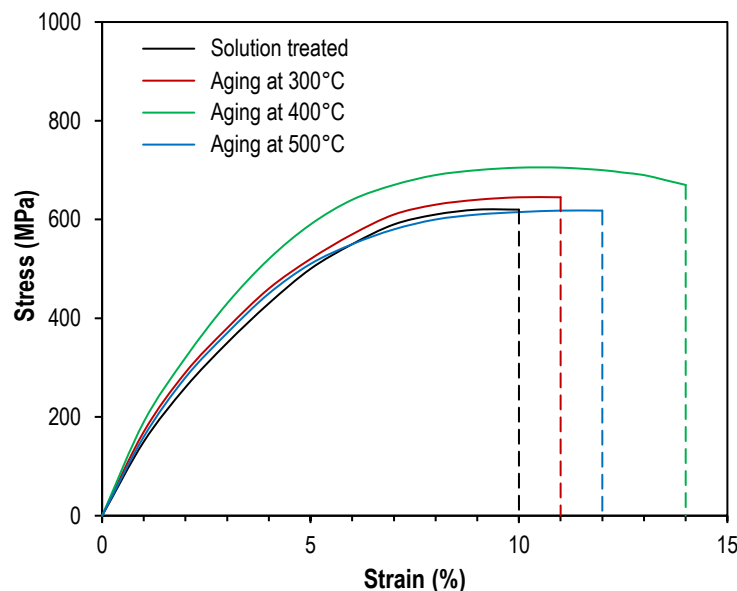


Fig. (3) Effect of aging on the stress-strain characteristics of the nitinol samples

Figure (4) shows the effect of aging temperature on the phase transformation temperatures of nitinol alloy with four different heat treatments. Aging at $300^\circ C$ lead to reasonable increase in all transformation temperatures as the final austenitic transformation (A_f) temperature was about $66^\circ C$, which means that the alloy stays in martensitic phase at room temperature. This is known as the martensite stabilization, which makes this alloy suitable for the applications of shape memory effect that depends on heating beyond room temperature to restore the original shape. In contrast, the solution treated sample showed moderate transformation temperatures of about $56^\circ C$, which makes the alloy in a transition region between the martensitic and austenitic phases at room temperature. Therefore, the alloy would not show apparent distinct functional properties.

The most important result was exhibited by the sample aged at $400^\circ C$ as a sharp decrease in all phase transformation temperatures was observed ($A_f \sim 16^\circ C$). This ensures completely austenitic phase of the alloy at room temperature ($25^\circ C$), which is an optimum condition for superelasticity effect required for applications of smart actuators and medical stents as the alloy transforms from austenite to martensite under the effect of stress only then automatically returns to austenite when removing the stress with no need for external heating. This behavior is ascribed to the uniform precipitation of Ti_3Ni_4 nanoparticles at $400^\circ C$, so, the transformation temperature is reduced and the stability of elastic response is enhanced. Finally, the sample aged at $500^\circ C$ showed a slight increase in the transformation

temperature when compared to the sample aged at 400°C as the A_f was 40°C. This is attributed to the coarsening of Ti_3Ni_4 particles that limits their effectiveness to reduce the phase transformation temperatures and hence retract the alloy performance when compared to the optimum sample aged at 400°C. These results reveal that the fine control of aging temperature is crucial in guiding the behavior of nitinol alloy towards the required application as the aging at 400°C represents an optimum point to ensure the austenitic phase at room temperature with an ability to transform into martensitic phase under stress, which is the fundamental condition to achieve the superelasticity effect in the applications of smart actuators.

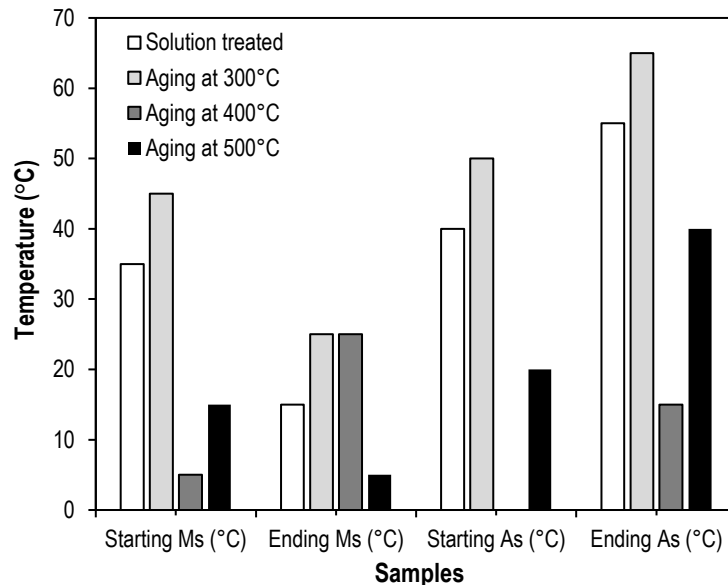


Fig. (3) Effect of heat treatment on transformation temperature of the nitinol samples

Figure (5) shows the effects of heat treatment on the fatigue life of nitinol alloy under cyclic stress. The results showed that all heat treatments lead to enhance the fatigue life when compared to the as-prepared sample, which showed 26000 cycles. The fatigue life was increased to 34000 cycles after solution treatment, to 42000 cycles after aging at 300°C, to 44000 cycles after aging at 400°C, and to 49000 cycles after aging at 500°C. Therefore, the sample aged at 500°C was better than that aged at 400°C, in contrast to results in Fig. (3) and this result can be indicated as the aging at elevated temperatures leads to enhance the precipitation of Ti_3Ni_4 nanoparticles and hence support the microstructure by hindering the dislocation propagation and the localized stress concentration. This in turn leads to crack initiation and propagation and hence reasonably enhances fatigue life. In some engineering applications, the small differences between samples may not have large statistical indicator as the overall mechanical performance and superelasticity are the crucial parameters in selecting optimum aging temperature beside the fatigue life [31,32]. Accordingly, aging at 400°C can be considered as the optimum to satisfy multiple practical requirements.

Figure (6) shows the effects of heat treatment on the fatigue resistance by measuring the relative fatigue life for the treated samples and compared to as-prepared sample as a reference with unity value. The solution treatment has led to a relative enhancement of 1.4 due to the reduced role of residual internal pressures resulted from previous forming processes to slightly increase the resistance to crack initiation. For aging at 300°C, a reasonable enhancement was observed as the relative fatigue life was increased to 2.0 due to the formation of precipitants from Ti_3Ni_4 nanoparticles that hinder the dislocation propagation and limit the localized stress concentration leading to fatigue failure. Aging at 400°C and 500°C has resulted the highest value of the relative fatigue life (3.0), which is three times the value of the as-prepared sample. Consequently, aging at moderate to elevated temperatures leads to major enhancement in fatigue resistance throughout an intense and uniform precipitation of Ti_3Ni_4 nanoparticles that hinder dislocation propagation and delay crack propagation. Also, the homogeneous dispersion of the precipitants within the microstructure reduces the localized stress concentration and distribute stresses under cyclic loading and this noticeably causes longer functional life of the alloy. As

no difference was observed in the relative fatigue life of samples aged at 400°C and 500°C, both treatments achieve comparable fatigue resistance values despite the differences in volume and precipitants distribution. Therefore, the enhanced fatigue resistance is achieved over a relatively wide range of aging temperatures, while the overall mechanical behavior and superelasticity effect still the crucial parameter when selecting the optimum temperature.

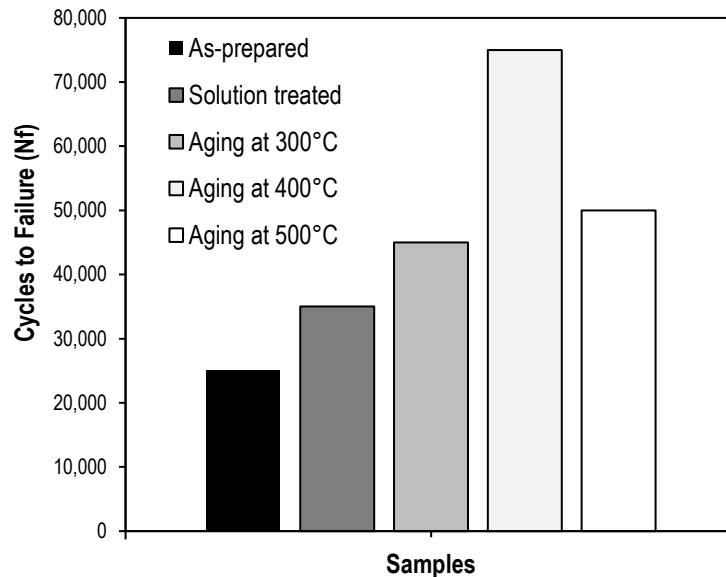


Fig. (5) Effect of heat treatment on the number of cycles before failure of the nitinol samples

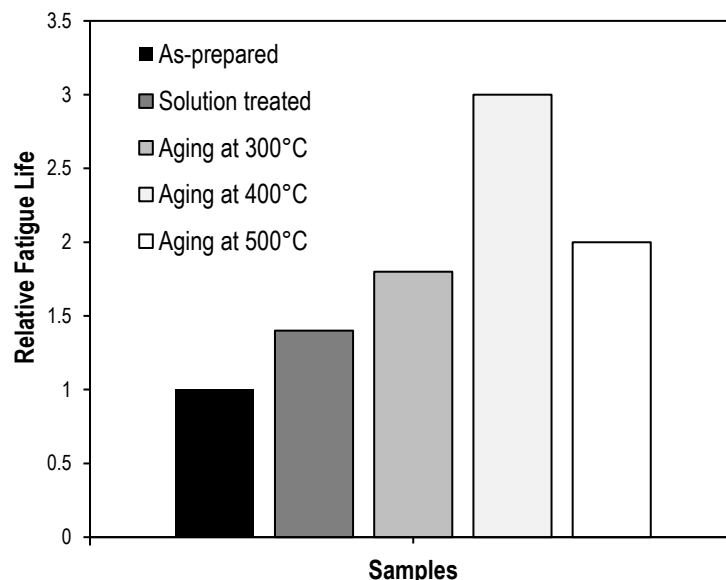


Fig. (6) Effect of heat treatment on the relative fatigue life of the nitinol samples

Figure (6) shows the SEM images of the nitinol alloy samples prepared in this work and differently heat treated. The as-prepared and solution-treated samples show relatively homogeneous morphology with ultrafine and scattered clusters. In case of the solution-treated sample, the dissolution of most elements in the austenite matrix can be observed, which leads to decrease the concentration of large precipitants when compared to the as-prepared sample. In the sample aged at 300°C, ultrafine precipitants appeared with high concentration and uniform distribution over the matrix. At this temperature, the activation energy is sufficient to form fine precipitants nucleation from the Ti_3Ni_4 phase. These precipitants play an important role in increasing the hardness of the material as well as enhancing

the shape memory throughout hindering the dislocation propagation. In the sample aged at 400°C, an observable transformation in the grain size of the precipitants is seen due to the coarsening phenomenon. The precipitants became larger and much more separated when compared to the aging at 300°C. This is ascribed to increasing the atomic diffusion rate with temperature, which allows the larger precipitants to contain the smaller precipitants according to Ostwald ripening phenomenon [33]. In case of aging at 500°C, the over-aging stage is reached as the precipitants are spherical or lenticular in shape and up to several hundred nanometers in size. The excess growth of the Ti_3Ni_4 phase size often leads to decrease the overall concentration of precipitants as well as change the chemical content of the surrounding matrix (loss of nickel), which increase the phase transformation temperature.

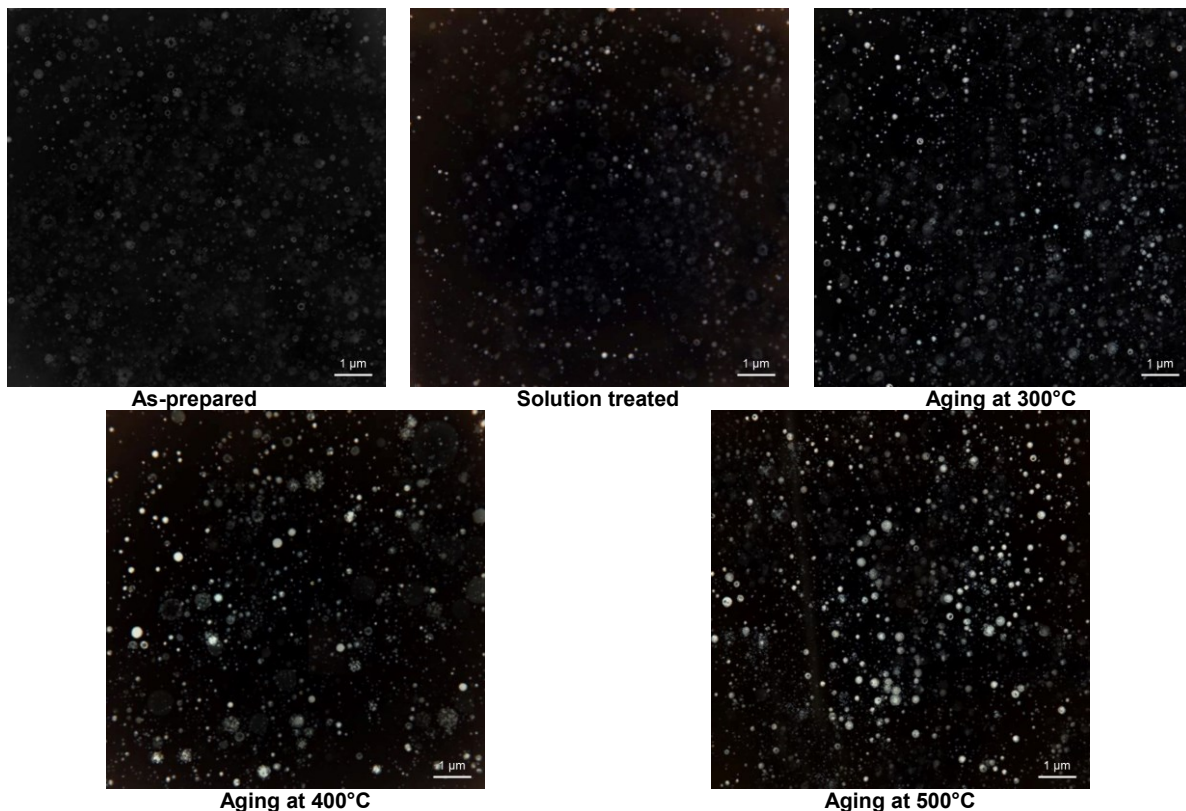


Fig. (7) SEM images of the nitinol alloy samples prepared in this work

4. Conclusion

In concluding remarks, based on the presented results, the fine control of heat treatment parameters of nitinol alloys – mainly aging temperature – plays a crucial role in modifying the behavior of the alloy to satisfy the requirements of specific applications. Aging at 400°C can be considered as the optimum conditions to achieve the superelasticity required for smart actuators and medical stents. At this temperature, the existence of the austenitic phase can be completely guaranteed at room temperature as both strength and elasticity are enhanced. As well, the fatigue resistance can be enhanced over a wide range of aging temperatures while the overall mechanical behavior still the fundamental standard to select the appropriate heat treatment.

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