

Effect of Aging Heat Treatment on Microstructure and Mechanical Properties of 50 wt.% Ni-42.5 wt.% Ti-7.5 wt.% Cu Shape Memory Alloy

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Abstract

NiTi alloys have good shape memory properties that adding alloying elements such as Cu can help to have smaller temperature hysteresis, better thermoelasticity, smaller superelastic hysteresis, higher damping capacity, and superior fatigue properties. The present work addresses functional mechanical properties of 50 wt.% Ni-42.5 wt.% Ti-7.5 wt.%. We study how aging heat treatment affects the alloy microstructure and mechanical properties. The results exhibit that increasing the aging temperature tend to increase the size of precipitation due to high diffusion in high temperature. The evaluation of mechanical properties reveals that the samples that aged in high temperature has lower fracture strength while these samples shows higher fracture strain. Aging time also has an effect on properties where by increasing the time, the size and amount of precipitations increase due to sufficient time for diffusion. By increasing the time, the fracture strength increase until a critical point and after that the fracture strength reduces.

Keywords

Nitinol, NiTiCu, Shape memory alloys, Heat treatment, Aging, Mechanical properties

Introduction

Shape memory materials (SMMs) are featured with the ability to recover their original shape from a significant and seemingly plastic deformation when a particular stimulus is applied. This is known as the shape memory effect (SME). Superelasticity (in alloys) or visco-elasticity (in polymers) are also commonly observed under certain conditions [1]. Common shape memory alloys are AuCd alloy, NiTi-based, Cu-based (CuAlNi and CuZnAl) and

Fe-based. NiTi should be the first choice since Ni-Ti-based shape memory alloys are attractive engineering materials with superb properties including high mechanical strength [2], superior super-elasticity, Wear behavior [3], excellent corrosion resistance [4], biocompatibility [5], good workability, lower density (is about 26% lower than steel), good pseudoelasticity [6], and self-healing properties [7], good shape memory behavior. So far, the application of NiTi Shape Memory Alloys has spread to aerospace, aviation and medical fields, etc.

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Four important temperature exist which they characterize NiTi shape memory alloys behavior. Austenite with B2 crystal structure is stable at high temperatures. At high temperature austenite is the parent phase. By cooling down, martensite with B19' start to form at a certain temperature that it is the martensite start temperature, M_s . The martensite formation finishes at the martensite finish temperature, M_f . By heating, the austenite start to form at the austenite start temperature, A_s and the transformation finish at as the austenite finish temperature, A_f [8]. In shape-memory alloys, the martensitic phase change is reversible and often termed thermoelastic. Thermoelastic martensitic transformation from high temperature austenite phase (B2) to low temperature martensite phase (B19') tends to gain shape memory and superelasticity behavior [9-18]. There is limited application for specific field where martensitic transformation temperatures around or below the room temperature [19]. By increasing the transformation temperature give more opportunity to employ these alloys in automotive and electro-technique industries [15]. To adjust the transformation temperature and hysteresis, adding an alloying elements and fabricating ternary alloy was suggested. Alloying elements are able to either elevate or lower the transformation temperatures. Accordingly, there are many investigations of NiTi(X) on the effect of ternary alloying elements where X is Cu [15], Fe [20], Al [21], Ag [14], Nb [22], Hf [15,23], and Ta [13]. However, it is not clear how the addition of different alloying elements to NiTi is linked to the elastic properties and, therefore, the transformation temperatures [16]. The big issue with adding alloy elements is that the cost of the alloy increases, unfortunately.

Among the alloying elements, copper is favored as an in substitution for the due to improve several properties while the martensitic transformation temperatures are above the room temperature [15]. NiTiCu have more attention due to its exclusive characteristics: Less composition sensitivity to transformation temperatures, smaller temperature hysteresis, enhancing the thermoelasticity, quicker actuation response, smaller superelastic hysteresis, higher damping capacity, superior fatigue and prevention of the precipitation of precipitates. Limited experimental results, mainly in Cu-based and also in NiTi-based shape memory alloys suggest that there is a critical value of elastic constants at

which transformation takes place which is not sensitive to alloy compositions [24] and is only slightly dependant on temperature [25]. To increase the mechanical properties of NiTiCu, aging treatment was suggested [26]. Ti_3Ni_4 , Ti_2Ni_3 and $TiNi_3$ are three dominate phases in the microstructure depending upon the aging temperature and time. Ti_3Ni_4 , $TiNi_3$, Ti_3Ni_4 are formed at lower aging temperatures (< 600 °C) and shorter aging time, at higher aging temperature and longer aging time and at intermediate temperature and time, respectively [19]. There is unfavorable phase that is Ti_2Ni , presents in the as-cast materials produced by VIM.

Most common methods to fabricating shape memory alloys is arc melting, induction melting, melt-spinning, plasma melting, injection molding, thermal explosion combustion, explosive shock-wave compression, severe plastic deformation, spark plasma sintering and extrusion [27]. Vacuum induction melting (VIM) with graphitic crucible is used extensively [28]. The alloy fabricated by VIM need to homogenize for 1 or 2 h of homogenization heat treatment at 850-1100 °C after producing NiTi shape memory [11,12,29-31].

One of the ways to increase the critical resolved shear stress (CRSS) of a material is Precipitation hardening. Within a metal, precipitates and atoms dissolved in the matrix affect the motion of dislocations which is termed as precipitation hardening and solution hardening, respectively. In precipitation-hardened materials, the CRSS can be defined as the stress required overcoming the interaction between dislocations and particles [32].

For NiTiCu alloy with lower 8 wt.% Cu, it also improves the workability of the alloy [15]. The aim of this study is to investigate the effect of aging heat treatment on microstructure, morphology and mechanical properties of 50 wt.% Ni-42.5 wt.% Ti-7.5 wt.% Cu shape memory alloy at temperatures within the crystallization range.

Experimental Procedures

Vacuum Induction Melting (VIM) with high frequency was employed to prepare an ingot with a nominal composition of 50 wt.% Ni-42.5 wt.% Ti-7.5 wt.% Cu. The purity of metal was more than 99.9%. The chemical compositions of as-cast alloys were analyzed by scanning electron microscope using EDX. The real composition (in wt.%) was is 50 wt.% Ni-42.65 wt.% Ti- 7.35 wt.% Cu for the ternary

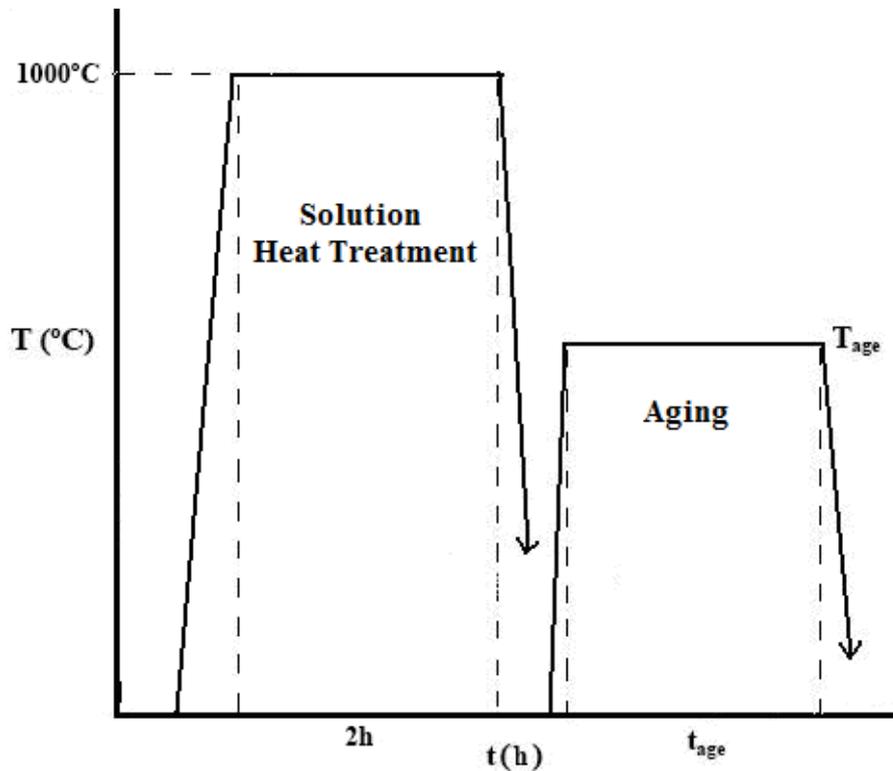


Figure 1: Graph of heat treatment.

Element	Series [wt.-%]	norm. C [at.-%]	Atom. C
Titanium	K series	42.65	47.97
Nickel	K series	49.99	44.45
Copper	K series	7.35	6.58

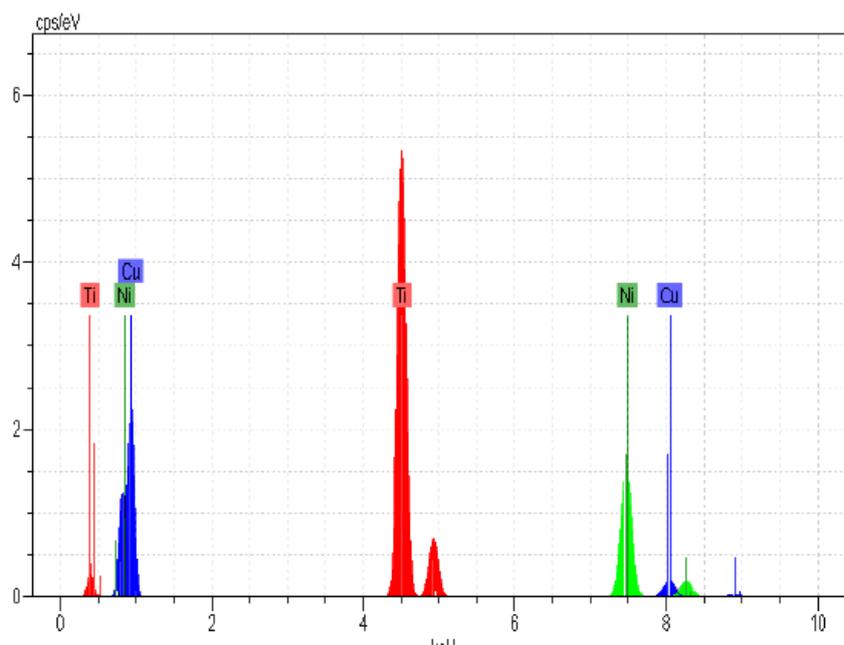


Figure 2: EDX result of as-cast NiTiCu.

NiTiCu alloy that it is very close to expected composition. The as-cast samples homogenized at 1000 °C for 1 hour and then cooling at furnace [11,12] to create a location for nucleation of second phases. To increase the number of dislocations, the 30% hot rolling at 1000 °C was done. The sub-size tensile test samples were extracted from ingot to do aging on samples. Before aging, the solution heat treatment at 1000 °C for 2 hours and then cooling in wa-

ter was done. In following, the selected temperature and time for aging heat treatment are the 400 °C and 700 °C for temperature and 10 minute and 1, 8 and 16 hour for time of aging were selected as it is shown in the graph of heat treatment in Figure 1. The microstructure of specimens was examined after polishing up to 6 μm alumina polish followed by etching in a solution of H2O:HNO3: HF (10:5:2) at ambient temperature. An AXIOPLAN2 optical

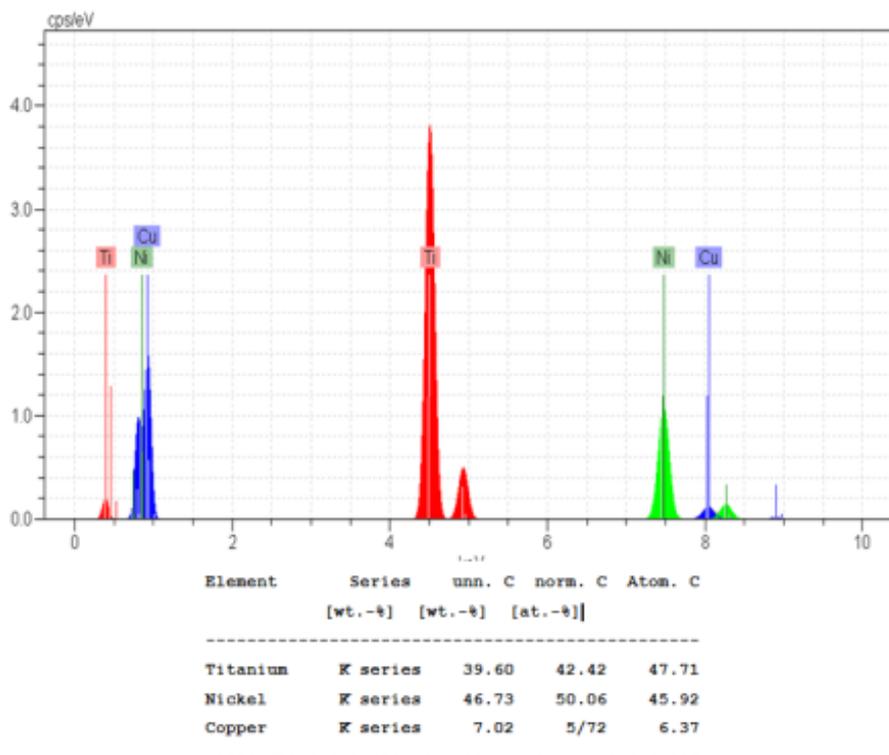
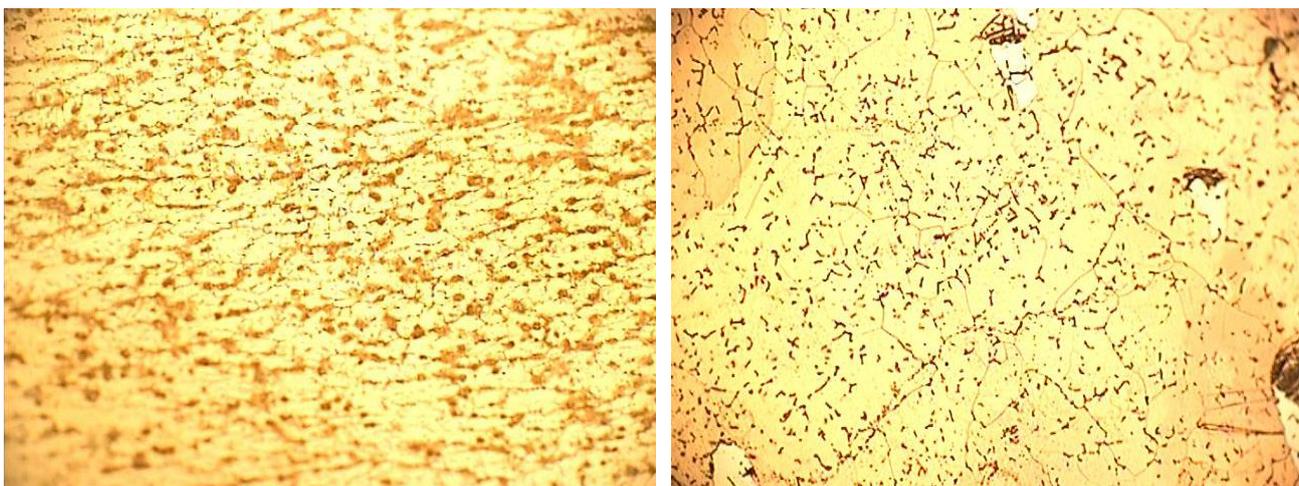


Figure 3: 50 and 7.5 weight percentage of Nickel and Copper.



a)

b)

Figure 4: a) As-cast; b) Homogenized microstructure of Ni-42.5 wt.% Ti-7.5 wt.% Cu in 100X.

microscope was used to study the morphology of alloys. Furthermore, samples were investigated using a TESCAN scanning electron microscope (SEM) equipped with energy dispersive X-ray detector (EDX). ASTM E8-08 standard for doing tensile test was employed.

Results and Discussion

Figure 2 shows the EDX result of as-cast NiTiCu. It shows that the alloying is really near to purpose for fabricating Ni-42.5 wt.% Ti-7.5 wt.% Cu shape memory alloy. The EDX results of base alloy for all of heat treated alloy at 400 °C at different times show almost identical composition the same as the as-cast alloy. 50 and 7.5 weight percentage of Nickel and Copper was observed, respectively as it is shown in Figure 3.

Observation of the as-cast microstructure as it is shown in Figure 4a illustrates a uniform micro-

structure and the precipitation was dispersed un-homogenizedly. So, the homogenization at 1000 °C for 1 hour and cooling at furnace was done for uni-

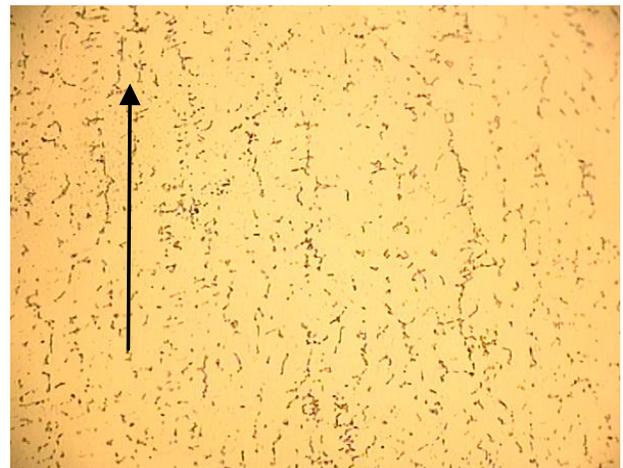
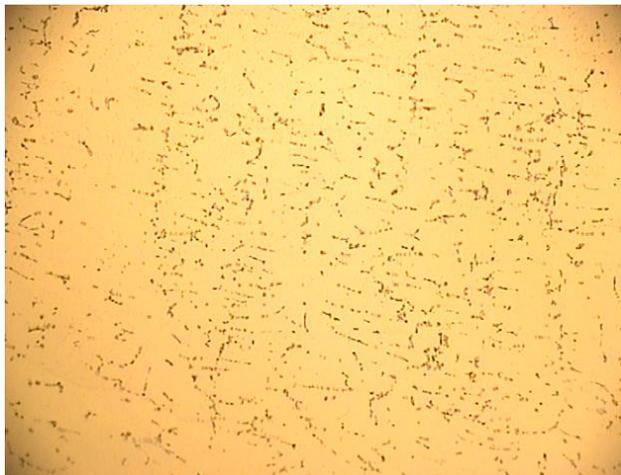


Figure 5: As-rolled Microstructure of Ni-42.5 wt.% Ti-7.5 wt.% Cu in 100X.



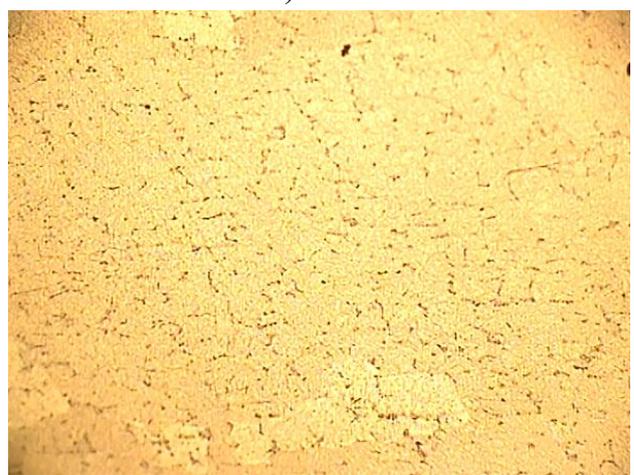
a)



b)



c)



d)

Figure 6: Microstructure of aging of Ni-42.5 wt.% Ti-7.5 wt.% Cu in 100X at 400 °C for a) 10 min; b) 1 h; c) 8 h; d) 16 h, 100X.

form microstructure [11,12]. Figure 4b shows the microstructure of homogenized alloy with properly uniform precipitation into grains.

Figure 5 shows the microstructure of as-rolled samples. By comparing between Figure 4b and Figure 5, it can be derived that grains have been deformed and the rolling direction are observed in microstructure. The microscopic examination in aging treated alloys at a temperature of 400 °C can be seen in Figure 6. In Figure 6a, aging treatment for 400 °C and 10 minutes shows no significant changes in the alloy microstructure and the precipitation dispersion. Moreover, the grains have kept the rolling direction. Thus, there is no expecting of changes in mechanical properties of alloy for 400 °C and 10 minutes aging treatment. With concentrating in Figure 6b, Figure 6c and Figure 6d, it is obvious that increasing time of aging has effect on amount and coarsening of precipitation. The volume fraction of precipitation increased and then in long aging time, it decreased with increasing the aging time where for 16 hours aging time, the precipitation are very coarse rather than other aging times with less volume fraction rather than 8 hour aging. The amount of precipitation will be increase by increasing aging time due to long time and enough time for diffusion. These changes in morphology of alloy can affect the mechanical properties.

Composition analysis of the precipitates in all samples is shown in Figure 7. As it is shown, the weight percentage of nickel in precipitates are much than titanium and copper but the weight percentage of nickel in precipitates is less than base alloy.

Figure 8 depict the metallographic and SEM of aging samples at 400 °C temperatures for 1 and 16 h. with comparing the microstructure and composition of precipitations in this figure, as it is mentioned earlier, it is known the composition of precipitation for different aging time is the same but amount and dimension of precipitation are different.

Figure 9 shows the metallographic image of aged samples at 700 °C for 10 minute and 1, 8, and 16 hours. For 10 minutes aging time as the same as occurred for aging at 400 °C, there is no significant changes in microstructure of sample as comparing to as-rolled sample as it is shown in Figure 9a and also, the rolling direction did not get rid of. By increasing the aging time, the direction of rolling was eliminated. The precipitations are very fine for 1 hour aging time. By increasing the aging time, the precipitation will grow and become thicker as it is shown in Figure 9b, Figure 9c and Figure 9d that type and size of precipitation and morphology of

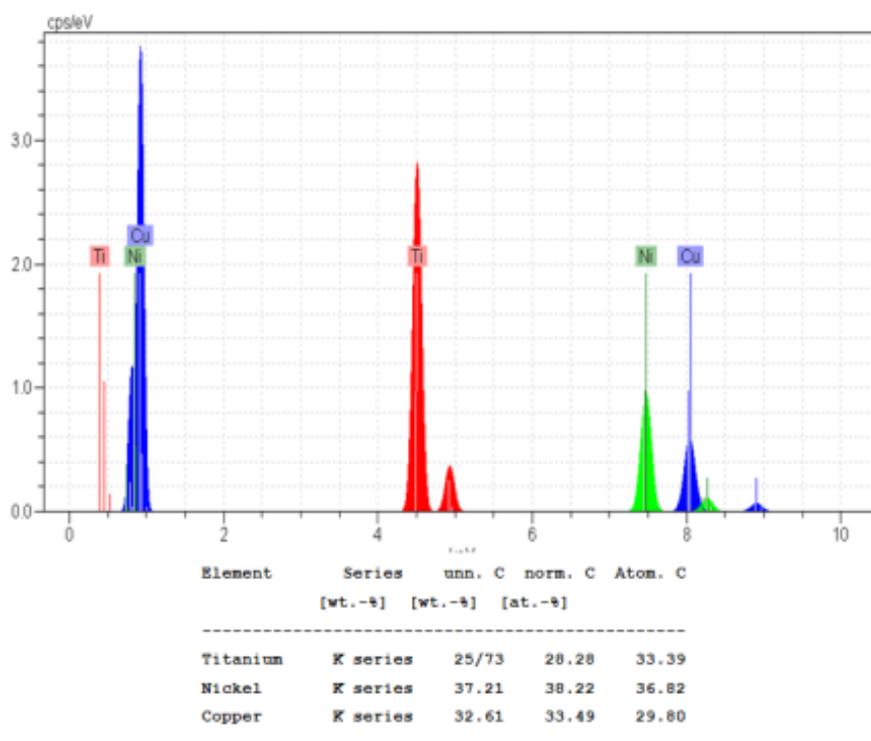


Figure 7: Composition analysis of the precipitates in all samples.

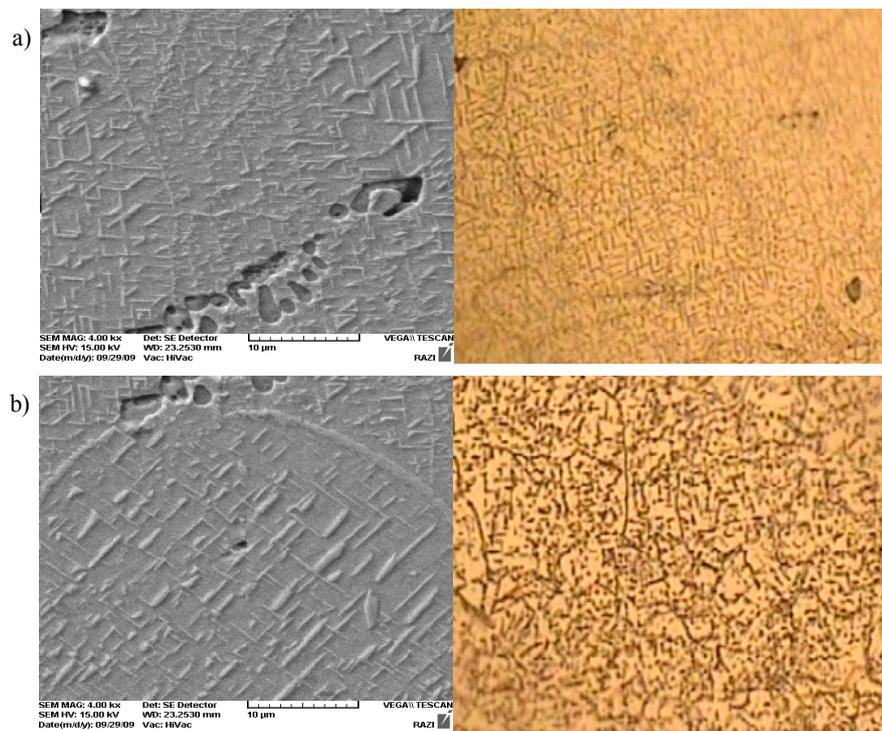


Figure 8: SEM micrograph of heat treated alloy at 400 °C for a) 1 h; b) 16 h aging time.

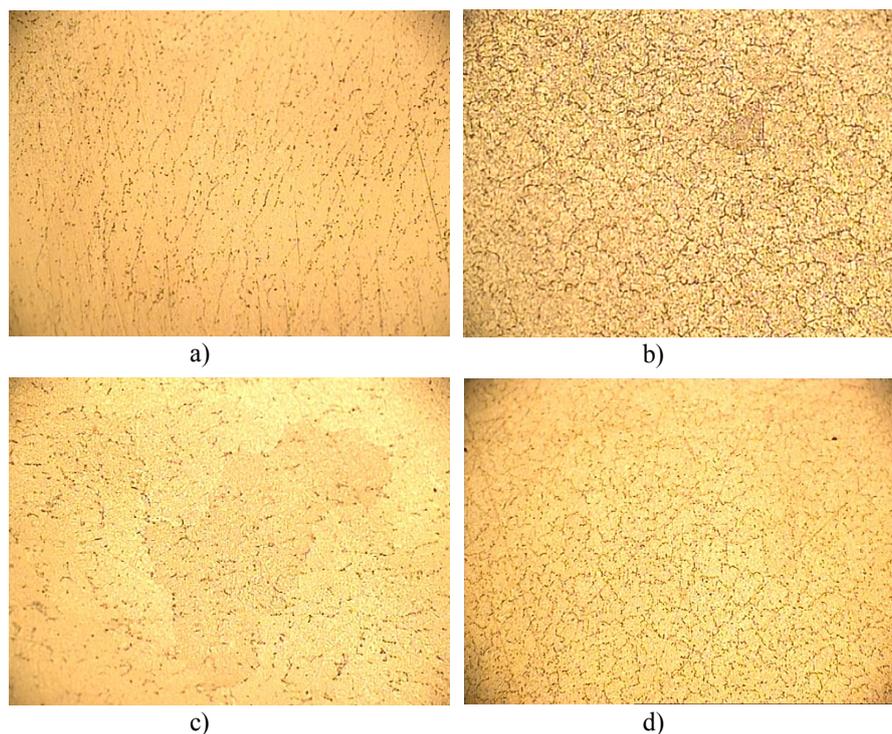


Figure 9: Microstructure of aging of Ni-42.5 wt.% Ti-7.5 wt.% Cu in 100X at 700 °C for a) 10 min; b) 1 h; c) 8 h; d) 16 h, 100X.

precipitation have effect on mechanical properties.

By comparing the Figure 6 and Figure 9, it is obvious that the precipitations are thicker for 700 °C

aging temperature than 400 °C aging temperature for the same time due to high temperature tends to increase the diffusion rate and increase the growth rate of precipitation.

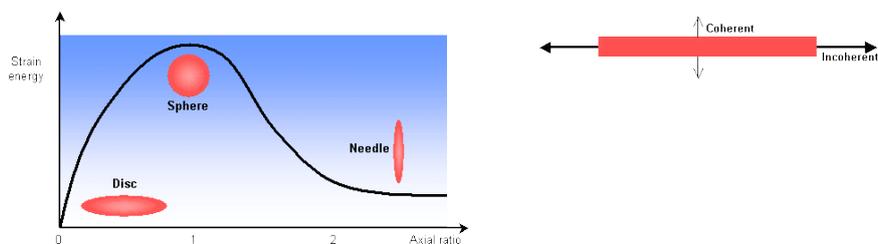


Figure 10: Effect of precipitation shape on mechanical properties.

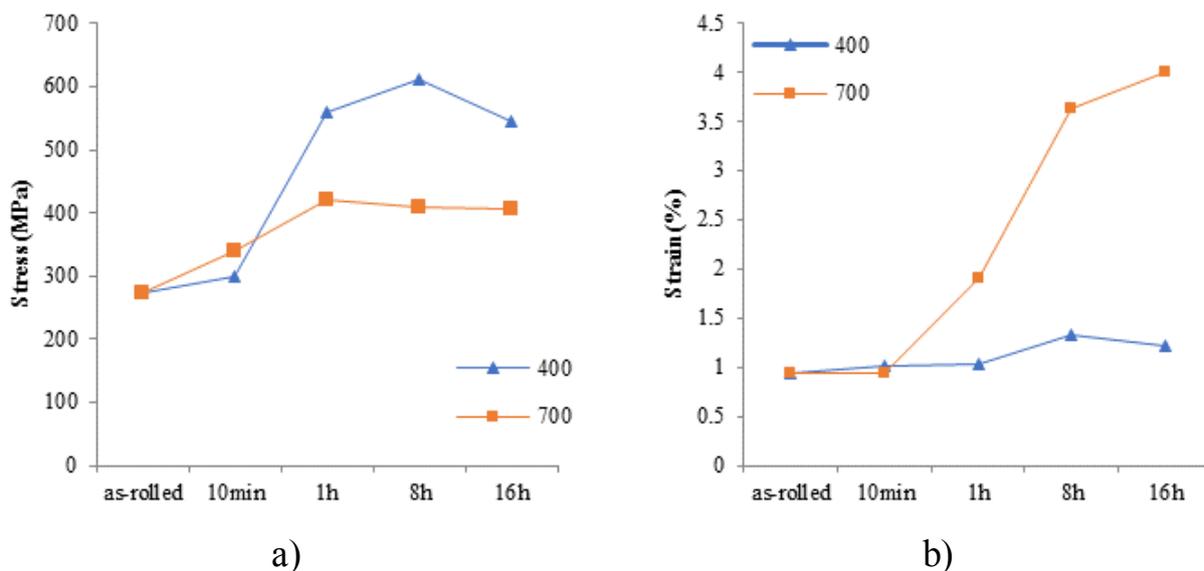


Figure 11: The result trends for aging at 400 °C demonstrate that increasing time of aging tends to increase fracture stress and fracture strain.

Table 1: Summary of mechanical properties of Ni-42.5 wt.% Ti-7.5 wt.% Cu at different time and temperature aging.

	As-rolled	Aging	10 min	1 h	8 h	16 h
		Time Aging Temp.				
Fracture stress (MPa)	272.3	400 °C	300.14	559.74	611.05	545.38
Fracture strain (%)	0.95		1.02	1.04	1.33	1.22
Fracture stress (MPa)	272.3	700 °C	339.25	421.31	409.78	406.33
Fracture strain (%)	0.95		0.95	1.9	3.63	4.01

Observation of electron microscopic image which aged at 700 °C showed that composition of base alloy and precipitations in different times experimenting is the same as composition of base and precipitation of aging samples at 400 °C. The difference between aging samples at 400 °C and 700 °C is the size of precipitations. The stresses present in the parent matrix as the precipitate grows strongly influences the shape of the precipitate as it is shown in Figure 10. By modeling the precipitate as an ellipsoid of revolution, Figure 10 shows how the strain energy is related to the shape. Growth

as discs or plates is clearly preferred. A precipitate particle will likely have some coherent and some incoherent interfaces with the matrix. The greater mobility of the incoherent interfaces leads to faster growth in these directions. This anisotropic growth leads to plate and disc morphologies. The bounding coherent interfaces will be parallel to crystallographic planes in the matrix.

The results of mechanical properties of aged samples are summarized in Table 1. By comparing the mechanical properties of as-rolled and aged

samples for 10 min and by comparing Figure 5 with Figure 6a and Figure 9a, it is obvious that there is no significant improving in mechanical properties for 400 °C and 700 °C aging temperature as it is mentioned earlier because of no changing in microstructure of these samples.

The fracture stress and fracture strain variations are presented in Figure 10a and Figure 10b, respectively. The result trends in Figure 11 for ag-

ing at 400 °C demonstrate that increasing time of aging tends to increase fracture stress and fracture strain by 8 hours, but over time, such as 16 hour aging treatment, decreasing of mechanical properties is evident. However, for aging treatment at 700 °C, stress to failure increases for 1 hour and at over time like 8 and 6 hours, it decreases by increasing the aging time. On the other hand, the fracture strain increase gradually and there is no

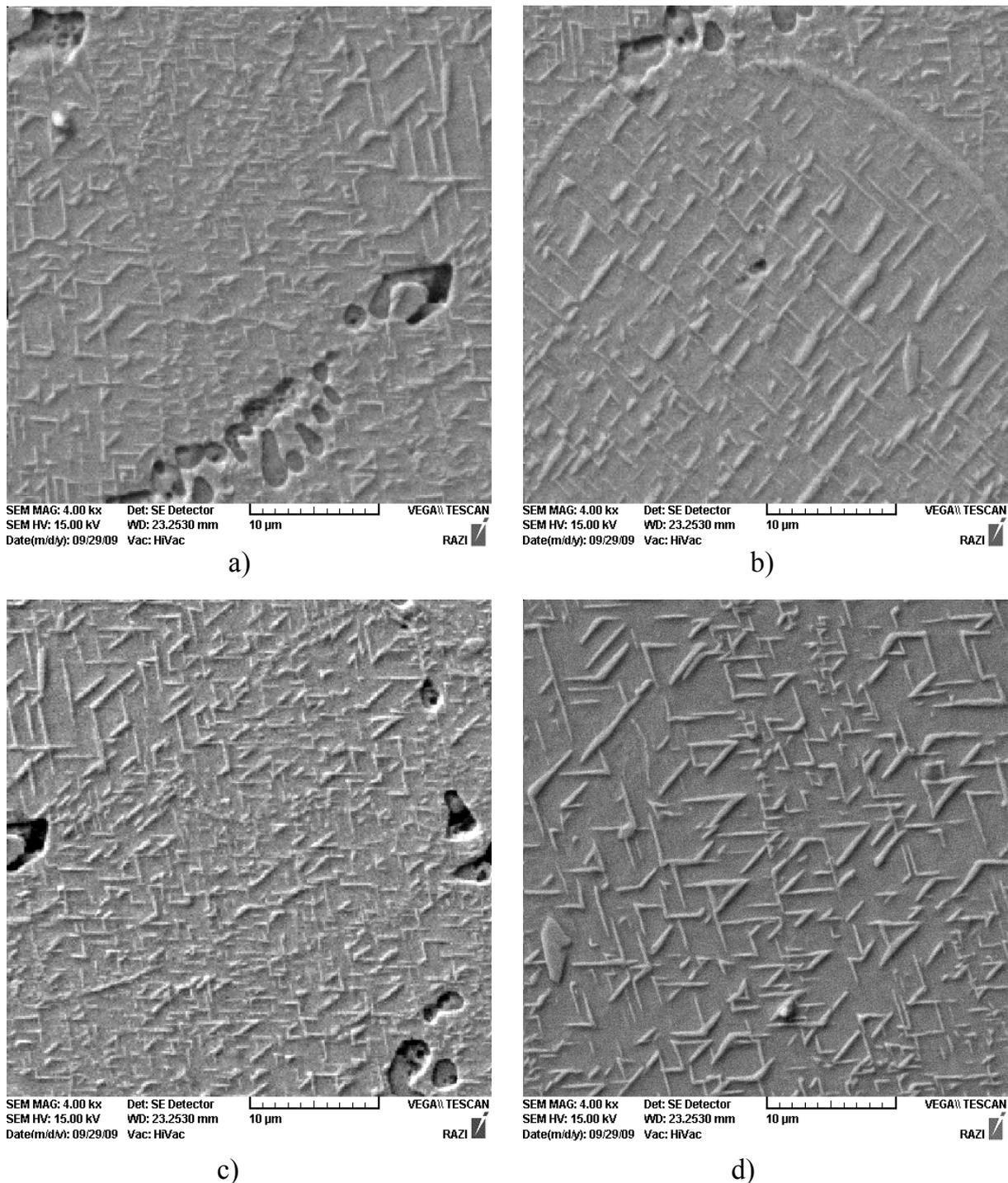


Figure 12: Comparing the size of precipitation: a) 400,1 h; b) 400,8 h; c) 700,1 h; d) 700,8 h.

changing trend by increasing aging time. This trend is well seen in Figure 11. The reason for decreasing the mechanical properties after a specific time is overaging and cause to fabricate coarse precipitate as you it is shown in Figure 6 and Figure 9. The temperature of aging has effect on overaging time whereas with increasing aging temperature, reaching overaging case dropped.

The reason for high overaging time for 400 °C and low overaging time for 700 °C is that this is due to at low temperature; the diffusion in solid is low and need more time for aging to reach ideal size of precipitation to achieve maximum tension while at higher temperature, 700 °C, due to high diffusion as a result of high temperature, the ideal precipitation size will reach at less time rather than low temperature such as 400 °C as it is shown in Figure 12. So, the loss of mechanical properties will occur in less time at higher aging time. In other words, higher aging temperatures to attain better properties can be achieved in a shorter time, but overaging occurs sooner while at lower aging temperatures, overaging time increases. Totally, the strength of samples aged at 400 °C is better than 700 °C while ductility of aged samples at 700 °C have better properties rather than aged samples at 400 °C.

Figure 12 illustrate the SEM micrograph of heat treated alloy at 400 °C and 700 °C for 1 and 8 hours. By comparing the size and morphology of precipitation at the same time, it is evident that precipitation are finer for 400 °C aging temperature than 700 °C aging temperature that it is evidence for changing the mechanical properties at several aging time and aging temperature. Precipitations act as obstacles to the glide of the dislocations and thus reduce their mobility. Dislocations can overcome these obstacles either by shearing these particles or bypassing them. The path a dislocation takes is predominantly dictated by the nature of precipitation, precipitation size distribution and the inter-precipitation spacing. Aging of precipitation hardened alloys leads to precipitation coarsening. Coarsening of precipitation from a very small size at a constant volume fraction helps in increasing the stress required to shear them. These precipitates hinder the movement of dislocations and substantially strengthen the alloy.

Equation 1 and Equation 2 shows the proportion of precipitations size (r) in distance between precipitations (λ) with mechanical shear stress [33].

$$\lambda = \frac{4(1-f)r}{3f} \quad (1)$$

$$\tau = \frac{Gb}{\lambda} \quad (2)$$

Where λ is the distance between the reinforcement particles, f is the particles volume fraction, assuming f is constant, and r is the size of precipitation. By increasing the aging time, there is enough time for nucleation and growth of precipitation that nucleation of new precipitations lead to decrease the distance between precipitations as it is shown in Figure 12, so, based on Eq. (1) and (2), the force for deformation will be increase that it cause to increase the strength. On the other hand, at over time that overaging will be occurred, the precipitation will join together and become thicker due to more diffusion. According to Eq. (1) and (2), the distance between precipitations will be increase that it tends to decrease the force for deformation and following reduction in strength. In general, in overaging condition, the distance between the precipitations will be increased that it tends to decrease strength of alloy against deformation.

As it is shown in Figure 13, the results of hardness depict the same trend as fracture strain as the hardness where hardness decrees by 8 and 1 hour aging at 400 °C and 700 °C, respectively, qua it was expected from our observation in fracture stress.

Figure 13 is the SEM images of fracture surface. The SEM fractography images shows shear bands in the failure of these samples that the shear bands represent a ductile failure of this alloy.

Conclusion

In the present paper, the processing steps required to make ternary NiTiCu ingots produced using Vacuum Induction Melting (VIM) with high

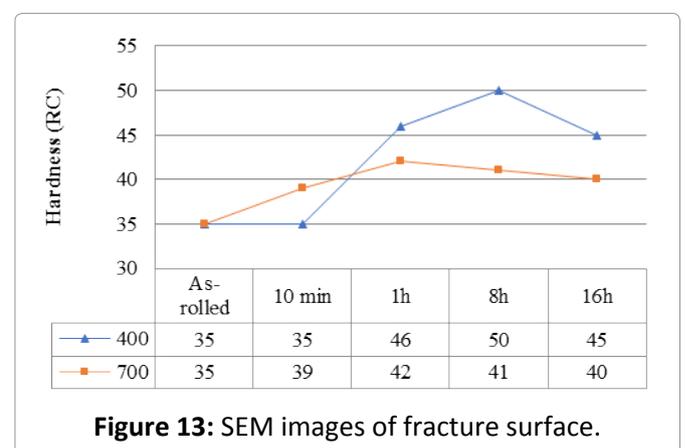


Figure 13: SEM images of fracture surface.

frequency. The processing steps include homogenizing, rolling, and aging treatment. From the results obtained from the present study, the following conclusions can be drawn:

1. Temperature can effect on morphology of precipitation where by increasing the temperature at the same aging time, the precipitation became coarse because of high coefficient diffusion at higher temperature.
2. Time of aging can change the morphology of precipitation. When the aging time increasing, time for diffusion increases and precipitation size increase and become coarse.
3. The fracture strength decrease by increasing the aging temperature at the same time due to precipitation coarsening while fracture strain increase by increasing the aging temperature.
4. The fracture strain of sample that aged at higher temperature is better that aged on lower temperature.
5. The fracture stress improve by increasing the aging time until a critical point and after that the fracture stress decrease.

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