



# Effect of room-temperature aging on shape memory characteristics of Ti–10Nb–10Zr–11Ta (at.%) alloy



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## ABSTRACT

In this study, the effect of room-temperature (RT) aging on the shape memory characteristics of the Ti–10Nb–10Zr–11Ta (at.%) alloy was investigated by tensile tests. Ingots were prepared using the arc melting method and then cold-rolled at a reduction of up to 95%. After cold-rolling, the plates were solution-treated at 1173 K for 1.8 ks, followed by aging at RT and temperatures up to ~573 K for various periods of times. Superior superelasticity was observed at RT in the solution-treated specimen. The critical stress for inducing martensitic transformation ( $\sigma_{SIM}$ ), tensile strength, and critical stress for slip ( $\sigma_S$ ) of specimens aged at RT increased with increasing aging time up to 60 days, showing no noticeable changes with further increases in the aging time. On the other hand, in the specimens aged at 373 K, 423 K, and 473 K for 3.6 ks, the values of  $\sigma_{SIM}$  and the tensile strength increased with increasing aging temperature, while the specimen aged at 573 K exhibited mature fractures. There were little change in  $\sigma_{SIM}$  and  $\sigma_S$  of the specimen that was solution-treated followed by aging at 373 K for 3.6 ks during RT aging. This result indicated that aging at 373 K resulted in good resistance against the effect of RT aging.

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## 1. Introduction

Ti–Ni shape memory alloys have been widely applied as biomedical materials, smart materials, and aerospace materials because they have excellent shape memory characteristics, corrosion resistance, and workability [1–3]. However, some reports pointed out that pure Ni causes allergic and hypersensitive reactions in the human body [4]. Therefore, a variety of Ni-free Ti-based shape memory alloys have been developed for biomedical applications [5–14].

Among Ni-free Ti-based shape memory alloys,  $\beta$ -type Ti-based alloys are well known for their superior biocompatibility, high strength, and low elastic modulus.  $\beta$ -type Ti-based alloys exhibit two stable phases—the  $\beta$  phase (BCC, austenite) at higher temperatures and the  $\alpha$  phase (HCP, martensite) at lower temperatures—and three metastable phases— $\alpha'$  phase (hexagonal martensite),  $\alpha''$  phase (orthorhombic martensite), and  $\omega$  phase (hexagonal structure). Quenching from the  $\beta$  phase leads to transformation from the  $\beta$  phase to martensite ( $\alpha'$  or  $\alpha''$  phase). It has been reported that the reverse transformation from the  $\alpha''$  phase to  $\beta$  phase causes shape recovery [15,16]. The transformation temperature can be controlled by adjusting the amount of alloying

elements. The  $\omega$  phase is formed by either quenching from high temperatures (athermal  $\omega$ ) or heat-treatment at intermediate temperatures (isothermal  $\omega$ ). Both athermal and isothermal  $\omega$  phases have hexagonal structures. Moffat and Kattner reported the metastable relationship between  $\beta$  phase and  $\omega$  phase thermodynamically in Ti–Nb, Ti–V and Ti–Mo alloy systems and they reported that  $\omega$  phase is Ti-rich phase [17].

It has been reported that  $\omega$  precipitates are formed during aging. In general, it is well known that the thermal  $\omega$  phase formed by aging at a low or intermediate temperature causes deleterious effects such as brittleness on the mechanical properties of Ti-based alloys. However, it was also reported that fine and dense isothermal  $\omega$  precipitates formed during aging in the temperature range between 573 and 673 K, and they were effective in increasing the critical stress for slip deformation and the critical stress for inducing martensite in Ti–26Nb (at.%) alloys [18]. Recently, Al-Zain et al. [19] reported that the critical stress for inducing martensitic transformation ( $\sigma_{SIM}$ ) increased considerably because of room-temperature (RT) aging caused by the formation of an isothermal  $\omega$  phase. They also reported that the RT-aging effect was suppressed by annealing at 973 K or solution treatment followed by aging at 773 K.

Meanwhile, it was also reported that the amount of the  $\omega$  phase formed during aging decreased with increasing Ta content in Ti–Ta alloys [20]. For practical applications, it is important to investigate the RT-aging effect caused by formation of the  $\omega$  phase and search for an excellent method to enhance the resistance against the effect

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of RT aging. Since the addition of Ta is expected to suppress the formation of the  $\omega$  phase, the Ti–10Nb–10Zr–11Ta (at.%) alloy used in this study was designed for that purpose.

There have been no studies on comparisons between RT aging with low-temperature aging. Therefore, in this study, the effects of RT and low-temperature aging (temperature range between 373 and 573 K) on the shape memory characteristics in the Ti–10Nb–10Zr–11Ta (at.%) alloy were investigated.

## 2. Experimental procedure

The Ti–10Nb–10Zr–11Ta (at.%) alloy was prepared using the arc melting method in an Ar atmosphere. The ingot was cold-rolled to sheets of 0.4 mm in thickness, with a final cold working ratio of 95%. Specimens with dimensions of 1 mm  $\times$  40 mm were cut from the sheets for tensile tests using an electro-discharge machine.

After cold-rolling, the plates were solution-treated at 1173 K for 1.8 ks in an Ar atmosphere in a quartz tube, followed by quenching into water. The lightly oxidized surface layer of each specimen after solution treatment was removed by pickling. After solution

treatment, some specimens were aged at temperatures between 373 and 573 K and some were aged at room temperature for different periods of time (30–120 days). The effects of RT aging and low-temperature aging on the shape memory characteristics of the Ti–10Nb–10Zr–11Ta (at.%) alloy were investigated by tensile tests.

## 3. Results and discussion

Fig. 1(a) shows the stress–strain curves of the specimens that were aged at room temperature (RT) for 0, 30, 60, and 120 days after solution treatment (ST) at 1173 K for 1.8 ks. Hereafter, the specimen aged at RT for 30 days after ST is abbreviated as the ST-RTA30 specimen, and specimens aged at RT for 60 days and 120 days after ST are abbreviated as the ST-RTA60 specimen and ST-RTA120 specimen, respectively. All specimens exhibited two-stage yielding. It was assumed that all specimens exhibited the shape memory effect or superelasticity at room temperature. The critical stress for inducing martensitic transformation ( $\sigma_{\text{SIM}}$ ) and yielding stress for plastic deformation are indicated by black-headed and white-headed arrows, respectively in Fig. 1(a). The value of  $\sigma_{\text{SIM}}$  increased with increasing aging time and the tensile strength also increased up to a value of  $\sim 1090$  MPa with increasing aging time, implying that the mechanical properties changed during aging at RT without heat treatment. The tensile strength and  $\sigma_{\text{SIM}}$  were evaluated using the stress–strain curves of specimens that were solution-treated followed by aging treatment at RT for 0–120 days; the results are plotted against the aging time in Fig. 1(b). The tensile strength and  $\sigma_{\text{SIM}}$  increased with increasing aging time to the maximum value observed in the ST-RTA60 specimen. The values stabilized thereafter and hardly increase with further increases in aging time, i.e., RT aging hardly progress with further aging after 60 days.

In order to evaluate the shape memory characteristics, cyclic tensile tests were carried out for all specimens, followed by aging at RT. Fig. 2 shows the stress–strain curves of the ST specimen. Each stress–strain curves were obtained by elongating the specimen up to 2.0% strain and then removing the load followed by heating. The test was carried out with an increase of 0.5% strain for each following cycle. Two types of strain were defined to characterize the shape memory properties of the alloys, namely the permanent residual strain (or plastic strain),  $\epsilon_p$ , and the strain recovered elastically and superelastically upon unloading after heating,  $\epsilon_A$ . Almost perfect superelastic behavior was observed in the first

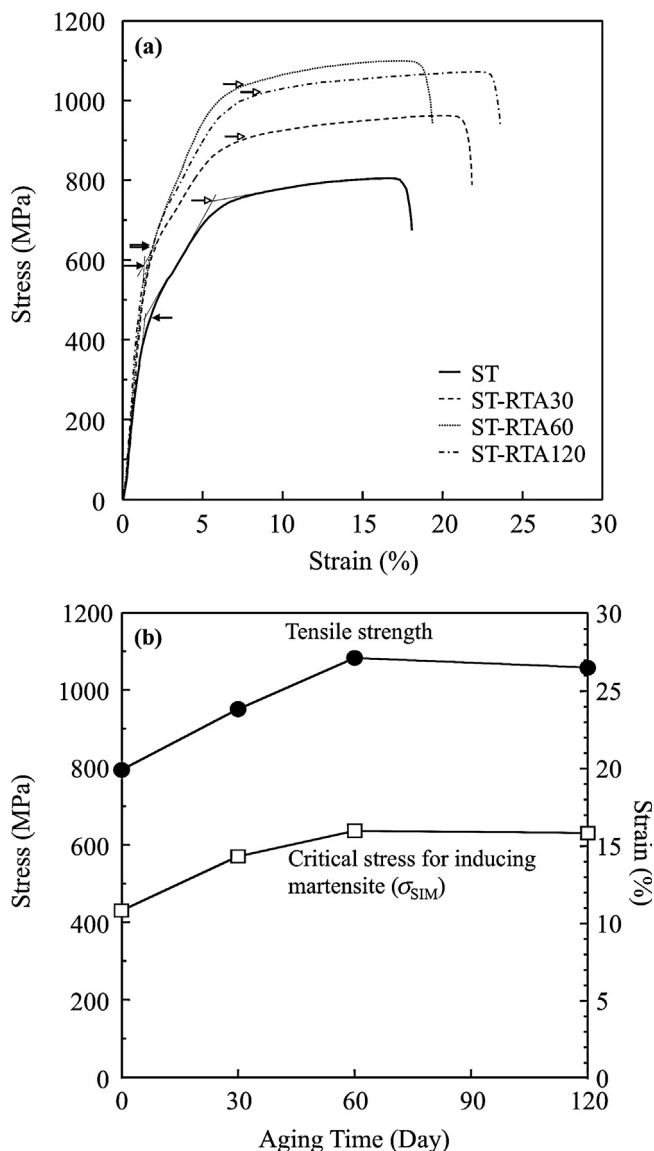


Fig. 1. (a) Stress–strain curves obtained in specimens aged at room temperature (RT) for 0–120 days. (b) Effect of aging time on the tensile strength, critical stress for inducing martensite formation ( $\sigma_{\text{SIM}}$ ), and fracture strain in the specimens.

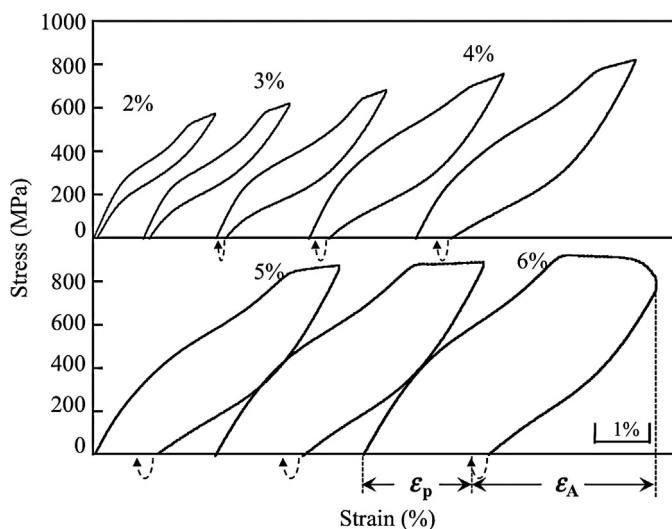


Fig. 2. Stress–strain curves obtained by cyclic loading–unloading tensile tests for the specimen that was solution-treated (ST) at 1173 K for 1.8 ks.

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