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Effect of Cold Work on Pseudoelastic Property of Ti-Nb Alloys for Utilizing as Artificial Bone

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Abstracts

Nowadays, there are many materials which are used in medical applications such as stainless steel 316L, Ti-6Al-4V and other Ti alloys. However, these materials are staying high modulus of elasticity when compare with human bone**.** From this reason, this research aims to explore the pseudoelasticity from Martensitic transformation in Ti-Nb alloys in order to obtain a low modulus of elasticity-liked properties. In this study, effect of cold-work on pseudoelasticity of Ti-Nb alloys was investigated by varying reduction ratio of thickness during cold-rolling process. Ti-Nb alloys with Nb-content from 22 to 26 at% were prepared by arc-melting method. Cold rolling processes were carried out at reduction ratio of 90 and 95% for each specimen, respectively in order to apply the internal stress for introducing metastable phase. After cold-rolling, specimens were heat-treated at 873K followed by aging at 573 and 673K, respectively for 3.6 ks in order to obtain precipitation strengthening due to formation of ω phase. Tensile tests were carried out in order to evaluate mechanical properties of each specimen. In order to investigate the pseudoelasticity, load-unloading tests under various strains were performed at room temperature. It is found that the pseudoelasticity can be confirmed in the alloy with Nb-content ranging from 23 to 26.7 at%, while modulus of elastic due to detwining of martensite variant increase with increasing reduction ratio. Excellent recovery pseudoelastic strain ratio is confirmed in a Ti-25.6at%Nb alloy. Moreover the very low modulus of elastic can be obtained during the range of reorientation of martensite variants which is suitable for utilizing as an artificial bone.

Keyword: Pseudoelasticity, Titanium alloy, Artificial Bone and Martensitic transformation

1. Introduction

Ti-based alloys have been widely used as biomaterials because of their high corrosion resistance, good biocompatibility, and high strength per weight. In previous time, Ti-6AL-4V is successfully applied as biomedical materials such as bone plates and artificial hip. However, it has been pointed out that pure V is a toxic element, so it is preferable to develop absolutely safe V-free Ti-base alloys for biomedical applications. [1-4]

In recent years, β-type Ti-base alloys was used as an implant material because of low modulus of elasticity, high formability, and good biocompatibility. Mo, Nb, and Ta was used as β-stabilizing element for fabricating β-type Ti-base alloys which compose of non toxic element [5]. Moreover, many β-type Ti-base alloys exhibit pseudoelastic behavior at room temperature. Pseudoelasticity can be obtained by stress-induced martensitc transformation which shows very low modulus of elasticity. It is reported that martensitic transformation from β (disordered body-centered cubic) to orthorhombic (α") martensite was exhibited in β-type Ti alloys above a critical alloying content, optimum heat treatment process and cold working process. [4]

At the first time, martensitic transformation was discovered in "shape memory effect in Ti-35 wt.% Nb (Ti-21.7 at.%Nb) alloy" which is a phase transformation between α" and β [6]. Nosova GI also explained the mechanism of martensitic transformation from the similarity of twice phase and the transformation of lattice constant from β to α" [7]. After that, there are many researches related to the improvement of pseudoelasticity in Ti-Nb alloy. For example; H.Y. Kim improved the pseudoelasticity by ω addition from aging treatment for increasing strength of microstructure [4]; the addition of third element in Ti-Nb alloy such as Al and Zr for improving mechanical properties of this alloy were reported by H. Hosoda [8] and L.M. Elias, [9] respectively.

In this work, the effect of cold-work on pseudoelasticity of Ti-Nb alloys by varying reduction ratio of thickness during cold-rolling process was investigated. Ti - Nb alloys with Nb-content from 22 to 26 at% were chosen for investigate mechanical properties in each heat treatment condition.

2. Experimental

Ti - Nb alloy with Nb-content from 22 to 26 at% were prepared by arc-melting method under argon (Ar) atmosphere in a water-cooled copper hearth. The ingots were homogenized at 1273 K for 7.2 ks and sliced into a plate with 3 mm. of thickness by EDM wire cut machine. The specimens were cold-rolled with a reduction in thickness of 90% and 95 %. The Electron Probe Micro-Analyzer (EPMA)(JXA 8100) was used in this study for investigating the chemical composition. After cold-rolled process, the specimens were annealed at 873 K for 0.6 ks followed by i) with out aging ii) aging at 573K for 3.6 ks and iii) aging at 673K for 3.6 ks. Load-unloading tests with strain rate of 4.167×10^{-4} s^{-1} under various strains (increase

0.2%strain in each cycle) (Fig. 1) were performed at room temperature for investigate the pseudoelasticity of Ti-Nb alloy. Biocompatibility test was carried out by MTT Assay. Similar tests were also done on Ti-Mo and Ti-Ni for comparison. L929 cell (mice connective tissue cell) was used in biocompatibility test and Dulbecco's Modified Eagle Medium (DMEM) was used for cell culture in the test. Every material was immersed with DMEM for 5 day before testing. After that DMEM taken from each material was used for cell culture for 3 day. Surviving cell was counted for evaluating biocompatibility of materials

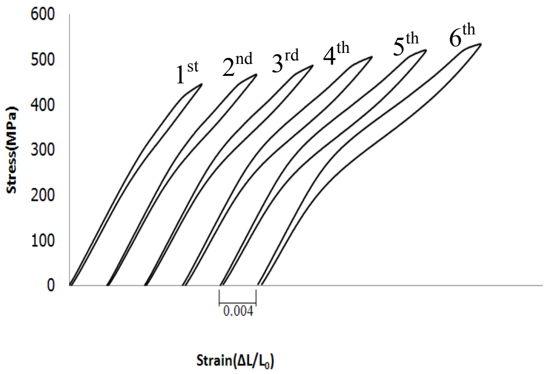

Fig. 1 Load-unloading diagram of Ti-25at% Nb alloy annealed at 873 K for 0.6 ks followed by aging at 573K for 3.6 ks with thickness reduction ratio of 89.85% under various strains (increase 0.2%strain in each cycle)

3. Result and Discussions

3.1 Chemical composition

The illustration of the comparison between analytical composition from EPMA testing and nominal composition is shown in Fig. 2. Analytical composition was slightly more than nominal composition in every alloy composition because of their different melting point between Ti and Nb (more 899K), so some of Ti would be loss from the evaporation during melting process. However, the difference of alloy composition which shown in Fig. 2 seems to be not significant because the chosen compositions in this study cover interested range.

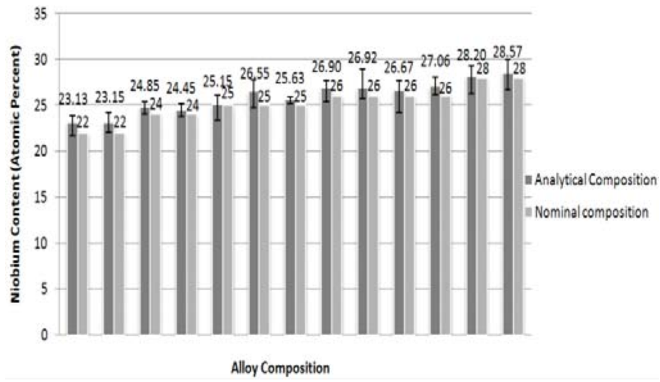

Fig. 2 Analytical composition from EPMA testing and nominal composition

3.2 Cyclic test

In order to elucidate the effect of cold work on pseudoelastic behavior, cyclic tensile tests were carried out at room temperature. Fig. 3 shows the schematic stress–strain curves obtained from the test.

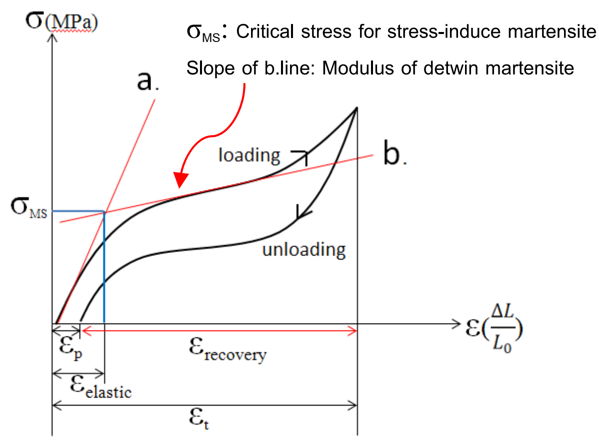

Fig. 3 Schematic figure for cyclic test

3.2.1 Effect of cold work on maximum strain

Pseudoelasticity in this study is defined as a recovery ratio (ratio between recovery strain and tensile strain) more than 95%. Fig. 4 shows maximum recovery strain of each pseudoelastic Ti-Nb alloy with 90 and 95% reduction ratio of thickness. As a result, it show Ti-Nb alloys cold-rolled at reduction ratio of 90% show higher maximum strain than those of the alloys cold-rolled at reduction ratio of 95% irrespective of alloy composition and heat-treatment process.

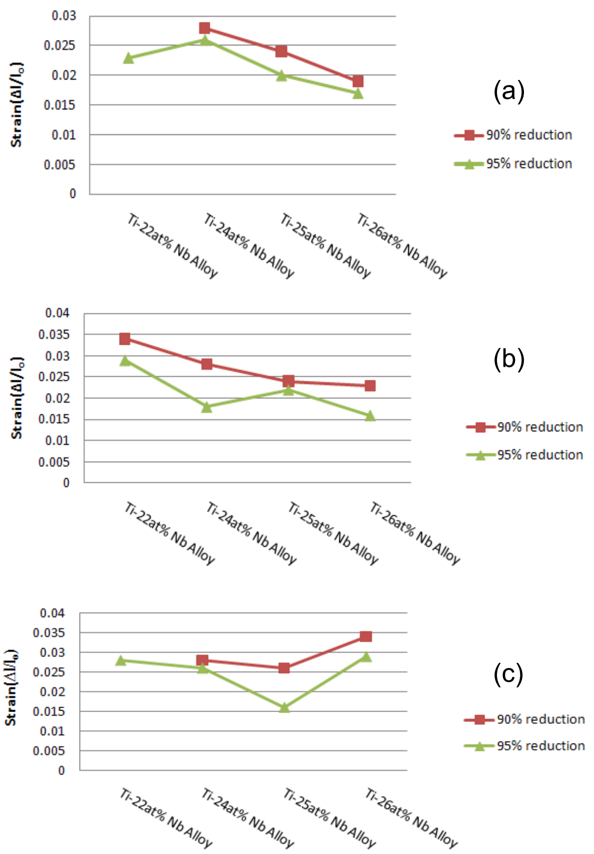

Fig. 4 Maximum strain of Ti-Nb alloys annealed at 873 K for 0.6 ks followed with a) no aging, b) aging at 573 K for 3.6 ks, and c) aging at 673 K for 3.6 ks

This can be explained by the cold-work hardening effect from alloy with 90% reduction is less than that of alloy with 95% reduction. From the result, the highest value of maximum strain was confirmed in a Ti-26at%Nb annealed at 873 K for 0.6 ks followed with aging at 673 K for 3.6 ks (0.034 strain)

3.2.2 Effect of cold work on Critical stress for stress-induced martensite σ_m

Critical stress for stress-induced martensite (σ_m) is a stress that induces martensitic transformation from β to α''. Normally stress-induced martensite depends

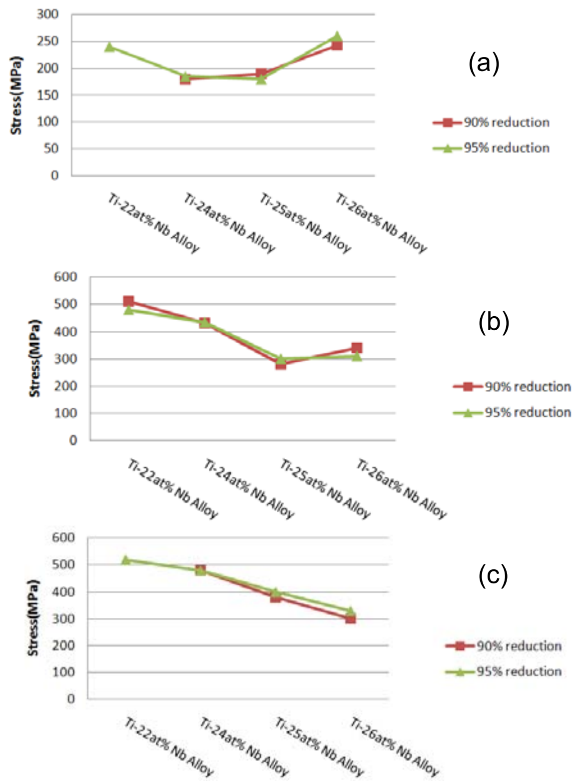

Fig. 5 Critical stress for Stress-induced martensite of Ti-Nb alloys with various alloy compositions annealed at 873 K for 0.6 ks followed with a) no aging, b) aging at 573 K for 3.6 ks, and c) aging at 673 K for 3.6 ks

on martensitic start temperature (M_s) and internal stress. However, it is seen that cold-work (reduction ratio) dose not affect the value of critical stress as shown in Fig 5.

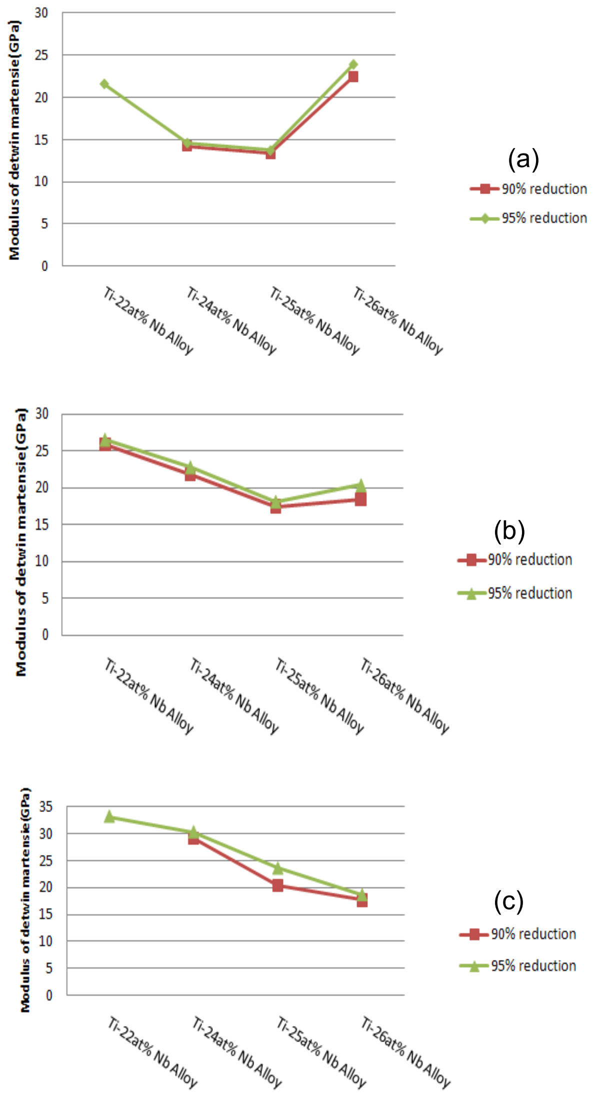

Fig. 6 Modulus of detwin martensite of Ti-Nb alloys with various alloy compositions annealed at 873 K for 0.6 ks followed with a) no aging b) aging at 573 K for, 3.6 ks, and c) aging at 673 K for 3.6 ks.

3.2.3 Effect of cold work on modulus of detwin martensite

Fig.6 shows the relationship between modulus of detwin martensite and reduction ratio. It is obviously seen that modulus of detwin martensite shows similar tendency to critical stress for stress-induced martensite (σ_m). Though modulus of detwin martensite slightly increase with increasing reduction ratio. This can be explained by the mobility of martensitic variant which is suppressed by dislocation density.

3.3 Biocompatibility

In order to investigate the biocompatibility of Ti alloy, MTT assay were carried out for Ti- Nb, Ti-Mo and Ti-Ni alloy. The result is shown in Fig.7. It shows surviving cell from MTT assay in each material compare with 50% surviving cell of control that is standard of MTT assay for arbitrate the biocompatibility. From the quantity of surviving cell, it shows these materials are good biocompatibility.

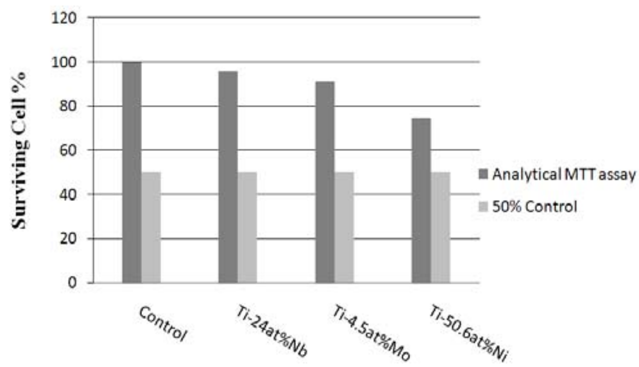

Fig. 6 Biocompatibility test by MTT Assay with L929 (mice connective tissue cell) cell culture.

4. Conclusion

1. Pseudoelasticity can be confirmed by this study.
2. Cold working from 90 to 95% reduction ratio did not affect critical stress for stress-induced martensite (σ_m)
3. Cold-rolling at appropriate reduction ratio can decrease modulus of detwin martensite which can suitably apply for artificial bone.
4. Good biocompatibility was confirmed in Ti-Nb alloys.
5. Maximum recovery strain more than 0.02 was obtained in this alloy system.

5. Acknowledgment

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