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Effects of Nd Content on the Dynamic Elastic Modulus and Mechanical Properties of Titanium-Neodymium Alloys

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The microstructures, dynamic elastic modulus, and mechanical properties of Ti-Nd alloys with Nd contents of 1.5, 3, 4.5, and 6 (mass%) were investigated in this study in order to assess whether Nd was an effective alloying element for decreasing the elastic modulus of a Ti alloy and simultaneously increasing its strength for its potential use in biomedical applications. The microstructures were examined by X-ray diffraction (XRD) analysis and scanning electron microscopy (SEM). The dynamic elastic modulus was measured by the vibration resonance method and the mechanical properties were determined from uniaxial tensile tests. Experimental results indicate that all the Ti-Nd alloys exhibit hexagonal structures of both α Ti and α Nd. An increase in the Nd content decreases the elastic modulus of the Ti-Nd alloys by a small amount and gradually increases their strength. The residual stress caused by cold rolling has a slight effect on both the elastic modulus and the mechanical properties of the Ti-Nd alloys. From this investigation, it can be concluded that Nd is not an effective alloying element for decreasing the elastic modulus of Ti alloys and simultaneously increasing their strength for their potential use in biomedical applications.

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1. Introduction

Ti and its alloys have become the most preferred metallic biomaterials because of their lower elastic modulus, better biocompatibility, and higher specific strength in comparison to conventional implant materials such as stainless steel and Co-Cr alloys.¹⁻³⁾ However, the elastic moduli of the currently used Ti implants, which are in the range of 105~114 GPa,¹⁻³⁾ are significantly higher than the elastic moduli of human bones (10~30 GPa).⁴⁾ As a result, these Ti implants can potentially lead to the resorption of adjacent bone tissues.⁵⁾ Thus, it is necessary to develop novel Ti biomaterials that exhibit better mechanical compatibility for biomedical applications.

An ideal metallic biomaterial should exhibit excellent biocompatibility, superior corrosion resistance, high strength, and low elastic modulus that is close to the elastic modulus of human bones.⁶⁻⁸⁾ A large number of studies have been carried out in order to develop such biomaterials. A previous study⁹⁾ has calculated the binding energies between titanium and various alloying elements, from which the strength and modulus of Ti alloys have then been estimated. It has been suggested that Ta, Nb, Mo, Hf, Zr, etc., have the potential to simultaneously enhance the strength and reduce the elastic modulus of Ti alloys.⁹⁾ We have experimentally investigated the effects of Ta and Hf on the elastic modulus and mechanical properties of Ti-Ta alloys¹⁰⁾ and Ti-Hf alloys,¹¹⁾ respectively, and have confirmed the abovementioned calculated results. Many new Ti alloys composed of elements such as Ta, Nb, Mo, Hf, and Zr have been developed specifically for biomedical applications. These Ti alloys exhibit better mechanical compatibility than the currently used Ti implants.^{2,3)} However, their elastic moduli between 55~85 GPa^{2,3)} are still considerably higher than

elastic moduli of human bones (10~30 GPa)⁴⁾ and their strength is not sufficiently high. Hence, they do not satisfy the criteria of ideal metallic biomaterials, more research should be conducted in an effort to further decrease the elastic modulus of these Ti alloys and simultaneously increase their strength so that they can be used effectively in biomedical applications and provide for a long service life.

It is well known that the rare earth materials (REMs) can improve various properties of some steels and nonferrous metals by modifying their microstructures. For example, a small addition of Nd to steels refines their grains, modifies their inclusions, and purifies steels, their mechanical properties are accordingly improved;^{12,13)} adding 0.3% Nd in Al-25Si alloy results in improvement of mechanical properties by refining primary silicon and eutectic morphology;¹⁴⁾ and grain sizes of Mg alloys are refined with the increase of Nd content, which leads to an increase in both room-strength and hardness of alloys.¹⁵⁾ In addition, Nd has a very low elastic modulus of 41 GPa¹⁶⁾ and no inter-metallic compound forms in the Ti-Nd alloys.¹⁷⁾ Thus, Nd is expected to be a potential alloying element to increase the strength and simultaneously reduce the elastic modulus of a Ti alloy. However, reports about the effects of Nd on the elastic modulus and mechanical properties of a Ti alloy are very deficient.

In this study, we investigate the effects of Nd on the elastic modulus and mechanical properties of Ti-Nd alloys in order to assess whether Nd is an effective alloying element for decreasing the elastic modulus of a Ti alloy and simultaneously increasing its strength for its potential use in biomedical applications.

2. Experimental Procedure

2.1 Material preparation

The most recent phase diagram of Ti-Nd is shown in Fig. 1.¹⁷⁾ It can be noticed that the solubility of Nd in Ti is very limited. Thus, the studied Ti-Nd alloys with low Nd

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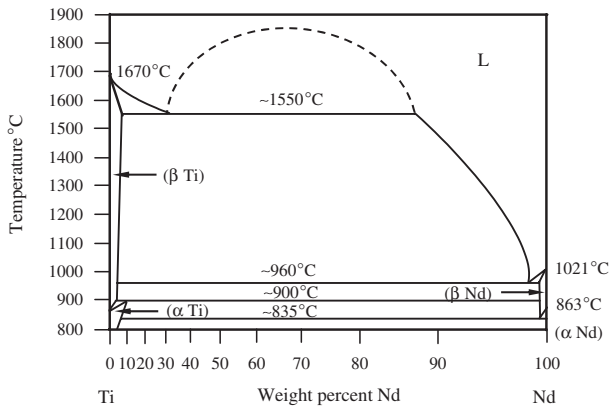


Fig. 1 Phase diagram of Ti-Nd alloy.¹⁷⁾

contents of 1.5, 3, 4.5, and 6 (mass%) were prepared by mixing appropriate amounts of high-purity sponge Ti (99.5%) and a chip of Nd (98.5%). The mixtures were melted under a high-purity argon atmosphere in a tri-arc furnace. Before melting, a definite amount of oxygen getter was melted in the furnace, which had been evacuated and flushed five times with purified argon. The melting operation was carried out five times for each alloy, and each time it was held in the molten state for 3~4 min. Simultaneously, the melted mixtures of Ti-Nd alloys were inverted before each melting to improve their chemical homogeneities. The same reduction in thickness was carried out for all the ingots, which were cooled in the tri-arc furnace and then cold rolled (CR) into plates with a thickness of 3 mm. The samples machined from the CR plates of the Ti-Nd alloys were used to measure their dynamic elastic modulus and mechanical properties. In order to verify the effects of residual stress caused by cold rolling on the dynamic elastic modulus and mechanical properties of the Ti-Nd alloys, some samples of the Ti-4.5Nd alloy were subjected to annealing treatment at 723 K for 7.2 and 14.2 ks, respectively, and then cooled down in air.

As a representative of all Ti-Nd alloys, the Ti-4.5Nd alloy was subjected to wet chemical and gas analysis. The result of the chemical analysis was 4.67 mass% Nd, 0.113 mass% O and balance Ti. Hence, the chemical composition of the Ti-4.5Nd alloy is close to its nominal composition, indicating that the preparation of Ti-Nd alloys was successful.

2.2 Material characterization

The microstructures of the Ti-Nd alloys were observed by scanning electron microscopy (SEM) at 20 kV. The samples for the SEM observation were ground, polished, and etched in a solution composed of 5 vol% HF, 10 vol% HNO₃, and 85 vol% H₂O. The phase constitutions of the Ti-Nd alloys were determined by X-ray diffraction (XRD) analysis using Cu-K α radiation in the typical range $2\theta = 30^\circ$ – 80° at an accelerating voltage of 40 kV, current of 250 mA and scanning speed of $1^\circ/\text{min}$.

2.3 Measurement of elastic modulus and mechanical properties

The dynamic elastic modulus was measured at room temperature along the rolling direction by the resonance

vibration method using rectangular plates having dimensions of 10 mm \times 2 mm \times 55 mm. This method has been reported in a previous study.¹⁰⁾ Uniaxial tensile tests were carried out at a crosshead speed of 8.33×10^{-6} m/s at room temperature using a specimen with a gage length of 12 mm and a cross section of 3 mm (width) \times 2 mm (thickness). A strain gage was attached to the gage section of each specimen to measure the strain change during each test. The ultimate tensile strength (UTS), 0.2% offset yield strength (YS), and elongation at fracture (EI) were determined accordingly. Three specimens were used for the measurement for each alloy in order to minimize experimental errors.

3. Results and Discussion

3.1 Microstructures

The XRD patterns of CR samples of Ti-Nd alloys are shown in Fig. 2. The XRD peaks reveal that all the Ti-Nd alloys exhibit hexagonal structures of both α Ti and α Nd. The intensity of α Ti is considerably stronger than that of α Nd, suggesting that the volume fraction of α Ti is considerably larger than that of α Nd according to the XRD technique described by Lopata and Kula.¹⁸⁾ The intensity of α Nd becomes stronger with the Nd content, which indicates the volume fractions of α Nd in the Ti-Nd alloys increase with the Nd content. No intermetallic or other phases such as β , α'' , α' , and ω have been detected by XRD analysis, which is in agreement with the result of a previous study.¹⁷⁾

The SEM microstructures of the Ti-Nd alloys are shown in Fig. 3, which obviously exhibit elongated structures formed by cold rolling. Combining with the above XRD results, it can be observed that the amount of the α Nd (indicated by arrows) increases with the Nd content. Since a large difference in corrosion resistance exists between Ti and Nd, and the etching solution used in this study is a heavy corrosive for α Nd, the phases of α Nd were over etched. This is the reason why the phases of alpha-Nd shown in Fig. 3 look like pores in the matrix.

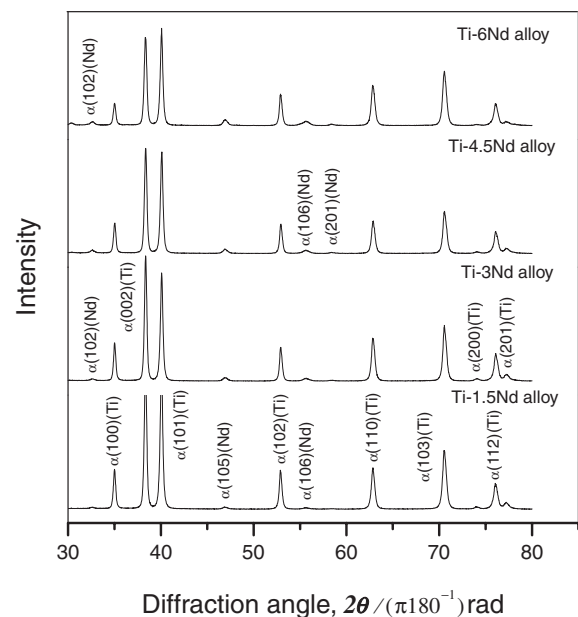


Fig. 2 XRD patterns of CR Ti-Nd alloys.

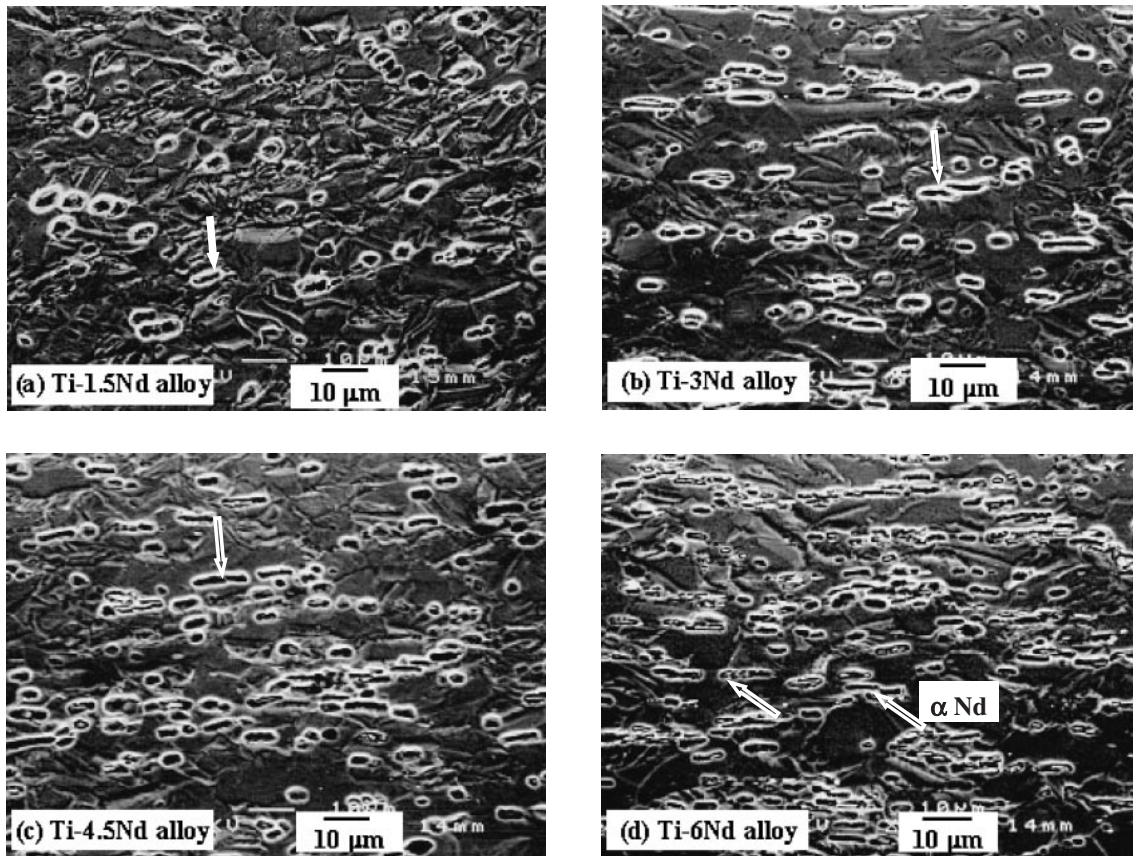


Fig. 3 SEM microstructures of CR Ti-Nd alloys.

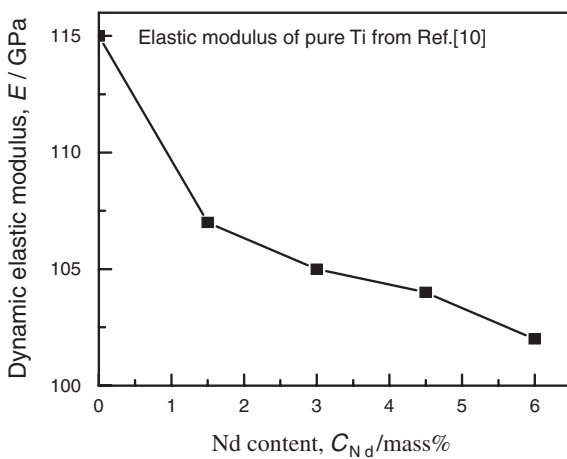


Fig. 4 Dynamic elastic modulus of CR Ti-Nd alloys with the Nd content.

3.2 Effects of Nd on the dynamic elastic modulus and mechanical properties

The measured dynamic elastic modulus of the CR Ti-Nd alloys is shown in Fig. 4. It can be observed that the dynamic elastic modulus of the Ti-Nd alloy decreases slightly with the Nd content. The elastic modulus is one of the intrinsic natures of a metallic material, which is determined by a bonding force among atoms. This bonding force is related to not only the crystal structure but also interatomic spacing, and it can be affected by alloying addition, heat treatment, and plastic deformation.^{10,11,19–22} A different phase has a different

elastic modulus, and the elastic modulus of a multiphase alloy is mainly determined by the elastic modulus of each constituent phase and the individual volume fraction according to the law of mixtures.^{19,20,23} For a Ti-Nd alloy with phases of α Nd and α Ti in this study, its elastic modulus is mainly determined by the elastic modulus of these two phases and their volume fractions. The elastic modulus of the α Nd is 41 GPa,¹⁶ which is considerably lower than that of the α Ti. And the volume fraction of the α Nd increases slightly with the Nd content, as shown by the results of XRD analysis and SEM observation. Those are related to a slight decrease in the elastic modulus of the Ti-Nd alloy with the Nd content. This behavior is in agreement with the law of mixtures.

The mean values of the UTS, YS, and El of the CR Ti-Nd alloys are shown in Fig. 5. It can be observed that both the UTS and YS values of all the Ti-Nd alloys increase gradually with the Nd content. The mechanical properties of an alloy are mainly dependent on its microstructure that is determined by its chemical composition, heat treatment and plastic deformation which it has been subjected to. Since the same plastic deformation and no heat treatment were performed on all the Ti-Nd alloys, it is considered that variations in mechanical properties of the Ti-Nd alloys are due to their different microstructures caused by the Nd content. The volume fraction of the α Nd in the Ti-Nd alloy increases with the Nd content as mentioned above, which is related to an increase in strength of the Ti-Nd alloy due to the improvement in strengthening effects by the second phase (α Nd). Contrary to the change in the trend of strength, the elongation

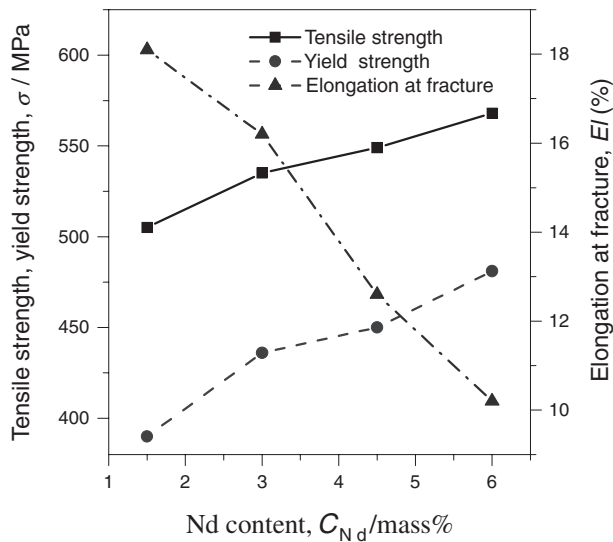


Fig. 5 Mechanical properties of CR Ti-Nd alloys with the Nd content.

at the fracture of the Ti-Nd alloy decreases with the Nd content, which is consistent with the ordinarily observed strength-ductility relationship, i.e., when the yield strength increases, the ductility decreases accordingly.

It should be pointed out that the Ti-6Nd alloy has a higher strength than the Ti-10Ta alloy¹⁰⁾ and the Ti-10Hf alloy¹¹⁾ (mass%). This, however, does not indicate that the element Nd has a greater effect on the strength of Ti alloys as compared to the elements Ta or Hf, because the CR Ti-6Nd alloy, which has not been subjected to heat treatment, is strengthened by a solid solution, second phase, and work hardening, while the solution-treated Ti-10Ta alloy and Ti-10Hf alloy with a single phase of α' are strengthened only by a solid solution.

3.3 Effects of residual stress on the dynamic elastic modulus and mechanical properties

In order to understand the effects of residual stress caused by cold rolling on the elastic modulus and mechanical properties of the Ti-Nd alloys, some samples of the CR Ti-4.5Nd alloy were subjected to an annealing treatment at 723 K for 7.2 and 14.2 ks, respectively, and then cooled down in air. The measured dynamic elastic modulus and mechanical properties of the annealed samples of Ti-4.5Nd alloy are shown in Figs. 6(a) and 6(b), respectively. It can be observed that the annealing at 723 K increases the dynamic elastic modulus of the Ti-4.5Nd alloy and its elongation at fracture by a small amount and gently decreases its strength. An increase in annealing time gently decreases the strength of the Ti-4.5Nd alloy and does not alter its elastic modulus. It is considered that the variations in elastic modulus and mechanical properties of the annealed Ti-4.5Nd alloy are due to changes in its microstructure caused by annealing treatment. The SEM microstructure of the annealed Ti-4.5Nd alloy, however, is similar to that shown in Fig. 3(c), and no noticeable change in the microstructure has been observed.

Generally, an annealing varies the microstructure of a plastically deformed metallic material. Recovery, recrystal-

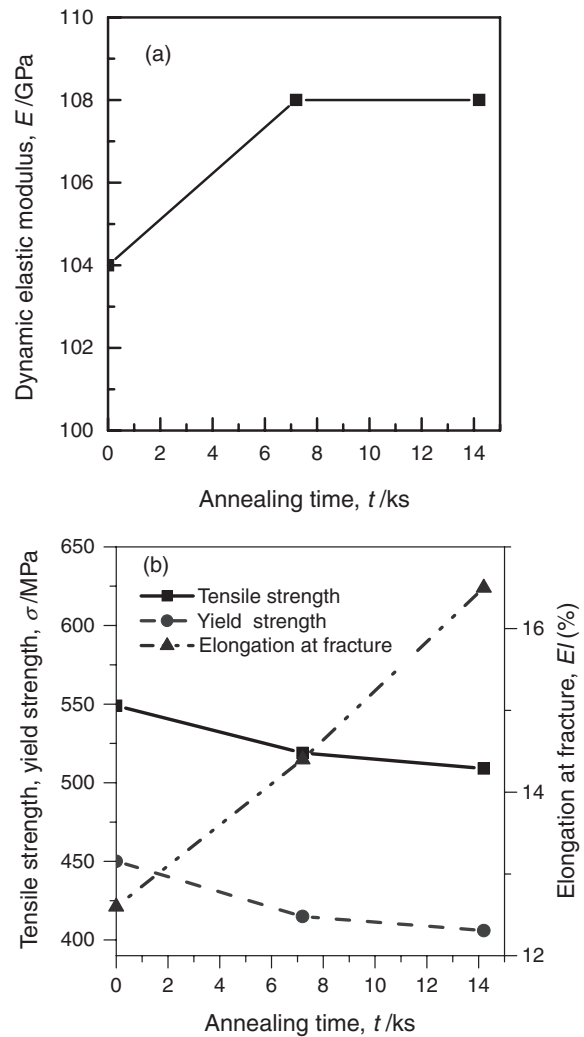


Fig. 6 (a) Dynamic elastic modulus and (b) mechanical properties of Ti-4.5Nd alloy with the annealing time.

lization, grain growth, or a combination of these actions occurs in the microstructure, which depends upon the annealing temperature. Recovery starts almost immediately on heating the metallic material and reaching its maximum temperature. Recrystallization, on the other hand, requires an incubation period and starts gradually.²⁴⁻²⁸⁾ The recrystallization temperature depends upon the duration for which annealing is carried out, the chemical composition, and the amount and type of plastic deformation.²⁴⁻²⁸⁾ The lowest recrystallization temperature for Ti and its alloys can be estimated by the empirical formula of $T_{\text{recrystallization}} \approx 0.4 T_{\text{melting point}}$.²⁴⁾ The melting point of the Ti-4.5Nd alloy is calculated to be 1933 K (1660°C) by the phase diagram of Ti-Nd alloy,¹⁷⁾ and the lowest recrystallization temperature of the Ti-4.5Nd alloy is estimated to be 773 K by the abovementioned empirical formula. Hence, the annealing temperature of 723 K used in this study is lower than the lowest recrystallization temperature of the Ti-4.5Nd alloy, which indicates that neither recrystallization nor grain growth occurs in the annealed Ti-4.5Nd alloy. The stress-relief temperature for pure Ti is often between 773 K~873 K,^{27,28)} which indicates the annealing at 723 K is suitable for the Ti-4.5Nd alloy to be stress relieved because the Ti-

4.5Nd alloy has a lower melting point than pure Ti. In fact, according to previous studies,^{24–26} a slight reduction in strength of a plastically deformed metallic material indicates that a recovery rather than a recrystallization or grain growth occurs in its microstructure, and only the densities of dislocations and point defects decrease during recovery.^{24–26} This is the real reason why no obvious change in microstructure of the annealed Ti-4.5Nd alloy can be observed. Hence, further investigation of the microstructure of annealed Ti-4.5Nd alloy using the transmission electron micrograph (TEM) analysis, which is more accurate than SEM analysis, is necessary.

Previous studies^{29–34} have investigated the effects of plastic deformation on the elastic modulus of metallic materials. They have concluded that the movement of the mobile dislocation and dislocation pileup are the main sources of the decrease in the elastic modulus after plastic deformation. Thus, the change in the dislocation arrangement after recovery is related to the small increase in elastic modulus of the annealed Ti-4.5Nd alloy. The elastic modulus of the Ti-4.5Nd alloy remains unchanged with the annealing time maybe because the recovery reaches its maximum at 723 K^{24–26} and the elastic modulus of a metallic material is not sensitive to its microstructure.²⁰ Since the same plastic deformation was carried out for all the Ti-Nd alloys, the effects of residual stress caused by cold rolling on the elastic modulus and mechanical properties of all the Ti-Nd alloys should be the same. Thus, it can be judged that the residual stress caused by cold rolling has a slight effect on both the dynamic elastic modulus and mechanical properties of all the Ti-Nd alloys.

4. Conclusions

On the basis of the results obtained from this investigation of the effects of Nd content on the dynamic elastic modulus and mechanical properties of Ti-Nd alloys, the following conclusions can be drawn.

All the Ti-Nd alloys exhibit hexagonal structures of both α Ti and α Nd. An increase in the Nd content decreases the elastic modulus of the Ti-Nd alloys by a small amount and gently increases their strength. The residual stress caused by cold rolling has a slight effect on both the elastic modulus and mechanical properties of Ti-Nd alloys. Nd is not an effective alloying element for decreasing the elastic modulus of Ti alloys and simultaneously increasing their strength for their potential use in biomedical applications.

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