


Redefining the β -Phase Stability in Ti-Nb-Zr Alloys for Alloy Design and Microstructural Prediction

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β -Phase stability is a key consideration for the design of β -titanium (β -Ti) alloys and subsequent heat treatment and/or thermo-mechanical processing. The concept of the Mo equivalence (Mo-Eq), proposed by Molchanova (Phase diagrams of titanium alloys, 1965), has been commonly used as a general guideline to gauge the stability of a β -Ti alloy. A critical literature review has shown that all four existing Mo-Eq expressions deviate substantially from experimental observations and the well-established d -electron theory in predicting the β -phase stability of Ti-Nb-Zr alloys. The reasons are that existing Mo-Eq expressions either completely neglect or significantly overestimate the β -stabilizing effect of Zr. In this study, a new Mo-Eq expression, i.e., $(\text{Mo-Eq})_{\text{Ti-Nb-Zr}} = 0.238\text{Nb (wt.\%)} + 0.11\text{Zr (wt.\%)} + 0.97$, has been defined for Ti-Nb-Zr alloys in order to properly address the β -stabilizing effect of Zr. This new Mo-Eq expression showed proven consistency with both experimental observations and the d -electron theory in predicting the β -phase stability of various Ti-Nb-Zr alloys. With necessary modifications, the approach developed is expected to be also applicable to the assessment of the β -phase stability in other Zr-containing Ti alloys.

INTRODUCTION

β -Titanium (β -Ti) alloys that are free from vanadium (V), nickel (Ni) and aluminium (Al) are one of the most promising groups of metallic biomaterials for orthopaedic applications. By definition, β -Ti alloys refer to those which contain sufficient β -stabilizing element(s) to enable the β -phase to be retained in a metastable condition by water quenching.¹ They are generally divided into metastable (age-hardenable) and stable β -Ti alloys (not age-hardenable).² The critical minimum amount of β -stabilizers needed to retain 100% β -phase on water quenching is denoted as β_c , while the minimum content of β -stabilizers needed to form stable β -Ti alloys is referred to as β_s . Metastable β -Ti alloys are those that fall in between β_c and β_s , within which α can precipitate upon ageing. As the values of β_c and β_s depend on alloy chemistry, a parameter known as the Mo equivalent (Mo-Eq) has been defined and commonly used as a general guideline to assess the β -phase stability of a Ti alloy for both

alloy design and processing.^{3–5} It is therefore an important design parameter for Ti alloys. This paper first examines all existing Mo-Eq expressions and then focuses on the development of a new Mo-Eq equation proposed specifically for the Ti-Nb-Zr system. This is followed by a detailed experimental assessment and comparison with Morinaga's d -electron theory for alloy design.

EXISTING MO-EQ EXPRESSIONS AND THEIR DEFICIENCIES

The concept of the Mo equivalence was proposed by Molchanova [Phase diagrams of titanium alloys (Figs. 193 and 194, p. 158)],³ which was a translation from *Atlas Diagramm Sostoyaniya Titanovykh Splavov* (Israel Program for Scientific Translations, Jerusalem, 1965). Boyer et al.⁴ presented the data shown in Fig. 193 of Ref. 3 as the Mo equivalence of a titanium alloy in the well-known *Materials properties handbook: titanium alloys* (1994) in the following form:

$$\begin{aligned}
 &(\text{Mo-Eq})^{\text{Molchanova [3]}} \\
 &= 1.0(\% \text{Mo}) + 0.2(\% \text{Ta}) + 0.28(\% \text{Nb}) \\
 &\quad + 0.4(\% \text{W}) + 0.67(\% \text{V}) + 1.25(\% \text{Cr}) \\
 &\quad + 1.25(\% \text{Ni}) + 1.7(\% \text{Mn}) + 1.7(\% \text{Co}) \\
 &\quad + 2.5(\% \text{Fe})
 \end{aligned} \quad (1)$$

Bania⁵ then further discussed the Mo-Eq concept and expressed it in the form of Eq. 2 in which the prefix coefficient of each β -stabilizer represents the ratio of the β_c value of Mo (i.e., 10 wt.%) to that of respective β -stabilizer. As an α -stabilizer, Al is also included to show its opposing effect when it is present in a β -Ti alloy.

$$\begin{aligned}
 &(\text{Mo-Eq})^{\text{Bania[5]}} \\
 &= 1.0(\% \text{Mo}) + 0.67(\% \text{V}) + 0.44(\% \text{W}) \\
 &\quad + 0.28(\% \text{Nb}) + 0.22(\% \text{Ta}) + 2.9(\% \text{Fe}) \\
 &\quad + 1.6(\% \text{Cr}) + 0.77(\% \text{Cu}) + 1.11(\% \text{Ni}) \\
 &\quad + 1.43(\% \text{Co}) + 1.54(\% \text{Mn}) - 1.0(\% \text{Al})
 \end{aligned} \quad (2)$$

Equation 2 overrides the original Eq. 1 as a commonly used expression to estimate the Mo-Eq of a Ti alloy. Apart from Eq. 2, three other expressions for Mo-Eq have also been proposed,^{6–8} namely:

$$\begin{aligned}
 &(\text{Mo-Eq})^{\text{Zhou [6]}} \\
 &= 1.0(\% \text{Mo}) + 0.74(\% \text{V}) + 0.5(\% \text{W}) + 0.39(\% \text{Nb}) \\
 &\quad + 0.28(\% \text{Ta}) + 2.2(\% \text{Fe}) + 1.69(\% \text{Cr}) + 0.85(\% \text{Cu}) \\
 &\quad + 1.22(\% \text{Ni}) + 1.57(\% \text{Co}) + 1.69(\% \text{Mn})
 \end{aligned} \quad (3)$$

$$\begin{aligned}
 &(\text{Mo-Eq})^{\text{Mo [7]}} \\
 &= 1.0(\% \text{Mo}) + 0.73(\% \text{V}) + 0.5(\% \text{W}) + 0.31(\% \text{Nb}) \\
 &\quad + 0.24(\% \text{Ta}) + 2(\% \text{Fe}) + 1.69(\% \text{Cr}) \\
 &\quad + 1.29(\% \text{Ni}) + 1.16(\% \text{Co}) + 1.69(\% \text{Mn})
 \end{aligned} \quad (4)$$

and

$$\begin{aligned}
 &(\text{Mo-Eq})^{\text{Wang[8]}} \\
 &= 1.0(\% \text{Mo}) + 1.25(\% \text{V}) + 0.59(\% \text{W}) + 0.28(\% \text{Nb}) \\
 &\quad + 0.22(\% \text{Ta}) + 1.93(\% \text{Fe}) + 1.84(\% \text{Cr}) + 1.5(\% \text{Cu}) \\
 &\quad + 2.46(\% \text{Ni}) + 2.67(\% \text{Co}) + 2.26(\% \text{Mn}) + 3.01(\% \text{Si}) \\
 &\quad + 0.3(\% \text{Sn}) + 0.47(\% \text{Zr}) - 1.47(\% \text{Al})
 \end{aligned} \quad (5)$$

Equations 2–4 are similar and were all formulated on the basis of experimental data, where Zr was treated as a neutral element and therefore excluded. It should be pointed out that no information was given about the sample size used in the water-quenching experiments, which affects the resultant microstructure. However, Eq. 3 considered that the β_c value of Mo should be 11 (rather than 10), which was also mentioned in Ref. 3. The slight difference between Eqs. 2, 3 and 4 resulted from experimental and analytical errors. In contrast, Eq. 5 combines both experimental and phase diagram data. For example, the coefficients for Nb and Ta were taken from Eq. 2, while the coefficient for Zr was derived from the Ti-Zr phase diagram.⁸ As a result, Zr was treated as a β -stabilizer that is more potent than Nb (0.28) and Ta (0.22), but no experimental support was given. Limited evidence suggests^{9,10} that the β -stabilizing effect of Zr depends on the quantity of other β -stabilizers present in the alloy.

Table I summarizes nine different Ti-Nb-Zr alloys reported in the literature with identified phase constituents in the water-quenched state.^{9,11} They provide an experimental basis for a detailed assessment of Eqs. 2 and 5. As can be seen from Table I, despite the high Mo-Eq values predicted by Eq. 5, e.g., 24.12 for Ti-12Nb-30Zr (at.%), α' -martensite still forms on water quenching. In other words, Eq. 5 significantly overestimates the β -stabilizing effect of the alloying elements in these alloys. With

Table I. Metastable β -region for Ti-Nb-Zr alloy systems and their Mo-Eq^{9,11}

Alloy compositions		Phases (water-quenched)	Mo-Eq values by existing expressions (wt.%)					New Mo-Eq by Eq. 7
			Equation 2	Equation 3	Equation 4	Equation 5		
at.%	wt.%							
Ti-12Nb-30Zr	Ti-16.82Nb-41.29Zr	α' ,b	4.71	6.56	5.21	24.12	9.5	
Ti-14Nb-30Zr	Ti-19.4Nb-40.7Zr	β	5.430	7.57	6.01	24.56	10.1	
Ti-8Nb-46Zr	Ti-10.4Nb-58.8Zr	α' ,b	2.91	4.06	3.22	30.55	9.9	
Ti-10Nb-45Zr	Ti-13Nb-57.1Zr	β	3.64	5.07	4.03	30.48	10.1	
Ti-20.9Nb-9Zr	Ti-31.7Nb-13.4Zr	α' ,b	8.88	12.36	9.83	15.17	9.99	
Ti-20.2Nb-12Zr	Ti-30.18Nb-17.6Zr	β	8.45	11.77	9.36	16.72	10.1	
Ti-22Nb-4Zr	Ti-34.4Nb-6.13Zr	β	9.63	13.42	10.66	12.51	9.8	
Ti-22Nb-6Zr	Ti-33.9Nb-9.1Zr	β	9.49	13.22	10.51	13.77	10	
Ti-22Nb-8Zr	Ti-33.4Nb-11.92Zr	β	9.35	13.03	10.35	14.95	10.2	

regards to Eq. 2, it substantially underestimates the overall β -stabilizing effect of the alloying elements in Ti-Nb-Zr alloys. For example, Eq. 2 predicts Mo-Eq values of 3.64 and 5.43 for Ti-10Nb-45Zr (at.%) and Ti-14Nb-30Zr (at.%), respectively, which are much lower than 10. However, the β -phase was fully retained in both alloys upon water quenching. Equations 2 and 4 offer similar predictions, while Eq. 3 also shows a serious underestimation of the β -stabilizing effect. For instance, the Mo-Eq of Ti-14Nb-30Zr (at.%) is predicted to be 7.57 but full β phase is retained. So is the case for Ti-10Nb-45Zr (at.%), where Mo-Eq = 5.07 but full β phase is retained. On the other hand, Eq. 3 overestimates the β -stabilizing effect in Ti-20.9Nb-9Zr (at.%) (clear formation of α'' , although Mo-Eq = 12.36). In summary, none of the existing Mo-Eq equations offers predictions that are consistent with experimental observations for Ti-Nb-Zr alloys. In addition, when all these predictions based on Eqs. 2–5 are compared with the d -electron theory diagram, there is also substantial inconsistency.

NEW MO-EQ EXPRESSION FOR TI-NB-ZR ALLOYS

We first determine the Mo-Eq coefficient of Nb. Three different values were used previously in Eqs. 2–5, namely, 0.28%, 0.31% and 0.39%. Recent experimental work has defined that the β_c value for Ti-Nb binary alloys is 42 wt.%.¹² Consequently, the Mo-Eq coefficient of Nb can be defined as

$$\psi = (10/42) = 0.238 \quad (6)$$

We then need to properly consider the β -stabilizing effect of Zr. As can be seen from Table I, a small increase (e.g., 1–3 at.%) in the content of Zr or Nb + Zr can change the phase constituents from $\alpha'' + \beta$ to single β under water-quenching conditions. For instance, a single β -phase was retained in the Ti-22Nb-4Zr (at.%) alloy but not in the Ti-22Nb-

2Zr (at.%) alloy. Similarly, a single β -phase was retained in the Ti-14Nb-30Zr (at.%) alloy but not in the Ti-12Nb-30Zr (at.%) alloy. Considering that Nb and Zr are generally weak β -stabilizers, it can be assumed that the Mo-Eq values of the Ti-Nb-Zr alloys listed in Table I are all in the vicinity of the critical value, i.e., 10 wt.%. Consequently, the β -stabilizing effect of Zr in these Ti-Nb-Zr alloys can be estimated by the quantity, $\Delta = 10 - \psi \times \text{Nb (wt.\%)}$. Figure 1 shows the dependence of the quantity, $\Delta = 10 - \psi \times \text{Nb (wt.\%)}$, on the Zr content for all the Ti-Nb-Zr alloys listed in Table I. The resultant significant linear relationship identifies the contribution of Zr in stabilizing the β phase in Ti-Nb-Zr alloys considered in Table I. Accordingly, a new Mo-Eq for Ti-Nb-Zr alloys can be defined from Fig. 1 as

$$(\text{Mo-Eq})_{\text{Ti-Nb-Zr}} = 0.238\text{Nb (wt.\%)} + 0.11\text{Zr (wt.\%)} + 0.97 \quad (7)$$

Equation 7 was applied to all the Ti-Nb-Zr alloys listed in Table I. Good consistency was observed with the experimental observations, where, when the new Mo-Eq parameter is close to or above 10, the alloy contains β -phase as the primary phase (along with some other secondary phases) after water quenching, while, when it is clearly below 10, martensite is the dominant phase.

APPLYING THE NEW MO-EQ TO THE DESIGN OF TI-NB-ZR ALLOYS AND VALIDATION

Table II summarizes the Mo-Eq values predicted by Eqs. 2–5 and 7 for six newly designed Ti-Nb-Zr alloys in this study (the first six alloys in Table II) and those available in the literature. Ingot samples of these alloys were made by arc-melting of a mixture of pure metals (purity > 99.9 wt.%) in a Ti-gettered high-purity argon atmosphere. The ingots were remelted four times to ensure chemical homogeneity. Then, the melted alloys were cast into a copper mold with a cylindrical cavity of 10 mm in diameter and 60 mm in depth. The as-cast samples with a diameter of 10 mm and thickness of 2 mm were homogenized at 1000°C for 4 h and then solution-treated at 900°C for 0.5 h, followed by water quenching. Samples were ground with SiC grinding papers of up to 4000 grit for microstructural characterization. Final polish was performed with a mixture of colloidal silica and H₂O₂. The samples were subsequently cleaned in an ultrasonic bath using ethanol and etched with Kroll's reagent. Phase identification was conducted using a Bruker D8 Advance x-ray diffractometer (XRD) with a Cu K_α radiation source at room temperature, and the resultant XRD data was analyzed by MAUD software.

Figure 2 shows the XRD data of six water-quenched Ti-Nb-Zr alloys and optical micrographs of two of them. Ti-10Nb-26Zr (at.%) was predicted to have martensite due to its low Mo-Eq (8.6,

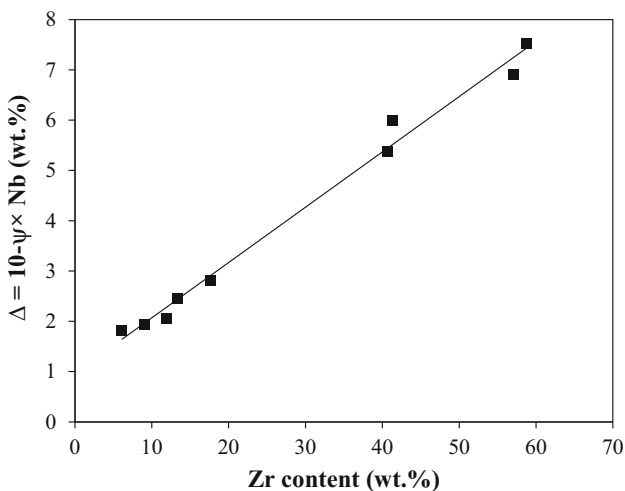


Fig. 1. Dependence of the quantity of $\Delta = 10 - \psi \times \text{Nb (wt.\%)}$ on Zr content in Ti-Nb-Zr alloys.

Table II. Newly designed metastable Ti-Nb-Zr alloys and corresponding Mo-Eq values

References	Alloy compositions		Mo-Eq values by different expressions				
	at.%	wt.%	Equation 2	Equation 3	Equation 4	Equation 5	Equation 7
Zr-based alloys (this study)	Ti-15Nb-40Zr	Ti-19.3Nb-50.7Zr	5.44	7.53	5.98	29.23	11.1
	Ti-10Nb-55Zr	Ti-12.2Nb-65.8Zr	3.42	4.76	3.78	34.34	11.1
Ti-based alloys (this study)	Ti-10Nb-26Zr	Ti-15Nb-37Zr	4.2	5.85	4.65	21.59	8.6
	Ti-15Nb-25Zr	Ti-21.3Nb-34.8Zr	5.96	8.31	6.6	22.32	9.9
	Ti-15Nb-22Zr	Ti-22Nb-31.3Zr	6.16	8.58	6.82	20.87	9.6
	Ti-20Nb-21Zr	Ti-28Nb-29Zr	7.84	10.92	8.68	21.47	10.8
13	Ti-24.5Nb-18.3Zr	Ti-34Nb-25Zr	9.52	13.26	10.54	21.27	11.8
	Ti-22Nb-24Zr	Ti-30Nb-32Zr	8.4	11.7	9.3	23.44	11.6
	Ti-20.7Nb-26.7Zr	Ti-28Nb-35.4Zr	7.84	10.92	8.68	24.48	11.5
	Ti-18.6Nb-31.1Zr	Ti-24.8Nb-40.7Zr	6.94	9.67	7.69	26.07	11.3
14	Ti-20Nb-12Zr	Ti-29.9Nb-17.6Zr	8.4	11.7	9.3	16.86	10
	Ti-17Nb-21Zr	Ti-24.4Nb-29.6Zr	6.72	9.36	7.44	20.82	10
	Ti-11Nb-38Zr	Ti-14.7Nb-50Zr	4.2	5.85	4.65	27.7	10
	Ti-6Nb-53Zr	Ti-7.6Nb-65.7Zr	2.24	3.12	2.48	32.79	10
15	Ti-22.6Nb-4.6Zr	Ti-35Nb-7Zr	9.8	13.65	10.85	13.09	10

Table II) while the rest should essentially be single β -phase in the water-quenched state. Both the XRD data and microstructural observations have confirmed these predictions, where the β -phase is predominant in each of the other five alloys (e.g., Figure 2c) plus a small amount of secondary phases (α' , ω) while Ti-10Nb-26Zr (at.%) contains significant α' -martensite (Fig. 2d). The newly defined Mo-Eq expression by Eq. 7 is in good agreement with the experimental observations of each alloy.

With regard to the alloys from the literature (Table II), they were all reported to be nearly single β -phase alloys (i.e., may contain a small amount of α' and/or ω).^{13–15} By the definition of the Mo-Eq, their Mo-Eq values should be equal to or above 10 wt.%. As can be seen from Table II, the new Mo-Eq (Eq. 7) also shows excellent consistency with experimental observations.

THE NEWLY PROPOSED MO-EQ AND THE D-ELECTRON THEORY

The d -electron theory describes the potential relationship between phase stability and elastic properties of Ti alloys through two electronic properties, bond order, B_o and d -orbital energy level, M_d .^{12,16} It was developed by Morinaga¹⁶ on the basis of the DV- $X\alpha$ molecular orbital calculations of electronic structures. The theory has become an essential fundamental tool for the design of β -Ti alloys.^{1,12} In this approach, B_o denotes the covalent bond strength between Ti and a specific alloying element and M_d correlates the electronegativity and metallic radius of that element. B_o and M_d values for all the alloying elements of Ti can be found in Ref. 16. Using these B_o and M_d values, the

compositional average of B_o and M_d can be calculated based on alloy composition through the following equations.¹⁶

$$\overline{B_o} = \sum X_i(B_o)_i \quad (8)$$

$$\overline{M_d} = \sum X_i(M_d)_i \quad (9)$$

where X_i , $(B_o)_i$ and $(M_d)_i$ are the atomic fraction, bond order and d -orbital energy level of the given element, respectively. Then, a $\overline{B_o}$ - $\overline{M_d}$ diagram can be plotted by taking the $\overline{B_o}$ and $\overline{M_d}$ values of different Ti alloys. Several sets of Ti alloys are used to define the three regions, α , $\alpha + \beta$, β , of this $\overline{B_o}$ - $\overline{M_d}$ diagram. Along the $(\alpha + \beta)/\beta$ boundary in the $\overline{B_o}$ - $\overline{M_d}$ diagram, the β -phase is considered metastable.

Figure 3 shows the $\overline{B_o}$ - $\overline{M_d}$ diagram plotted for this study on the basis of Refs. 12 and 14. A triangle is drawn by connecting the B_o and M_d values for pure Ti, Nb and Zr, which define the regime of the Ti-Nb-Zr alloys, similar to ternary phase diagrams. Each side of this triangle indicates the compositional average values of B_o and M_d of the Ti-Nb, Ti-Zr and Zr-Nb binary alloys. The blue dotted line represents the boundary of critical compositions for metastable β alloys according to the inventor of the d -electron theory.¹² This boundary may shift upwards or downwards depending on the exact alloying compositions. In the Ti-Nb-Zr system, adding Zr was found to shift this boundary downwards,¹⁴ as shown by the black dashed line in Fig. 3. The Mo-Eq values predicted by Eq. 7 are superimposed onto Fig. 3, which indicates that the newly developed Mo-Eq expression is also in good agreement with the d -electron theory in predicting the

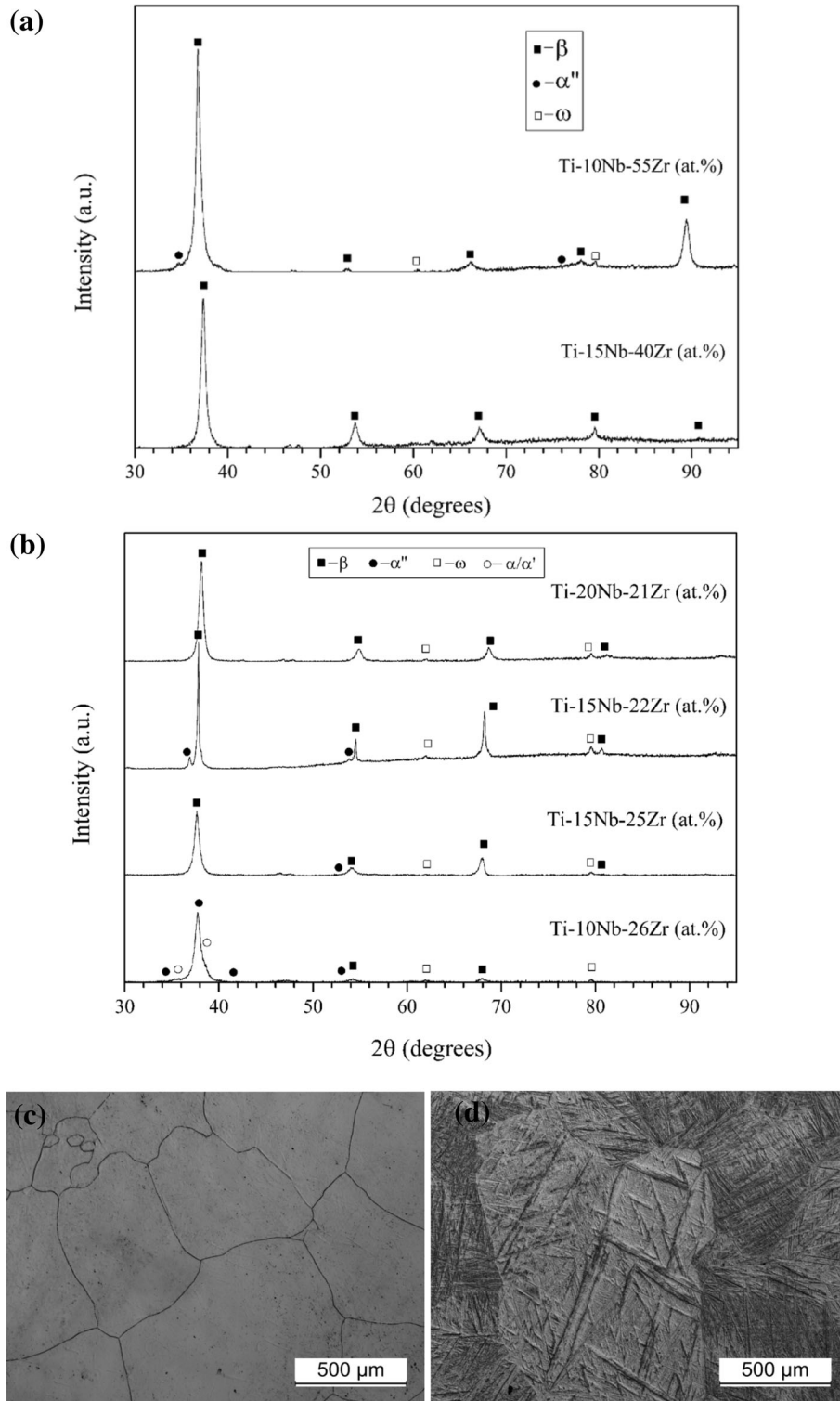


Fig. 2. XRD results of (a) two Zr-based Ti-Nb-Zr alloys and (b) four Ti-based Ti-Nb-Zr alloys. (c) Water-quenched Ti-15Nb-40Zr (at.%), nearly single β . (d) Water-quenched Ti-10Nb-26Zr (at.%), noticeable α'' -martensite.

β -phase stability. For example, Ti-Nb-Zr alloys having Mo-Eq values higher than 10 wt.% are situated above the black dashed line and vice versa.

The new Mo-Eq is thus consistent with the d -electron theory for Ti-Nb-Zr alloys, which was not the case for all the other four Mo-Eq expressions.

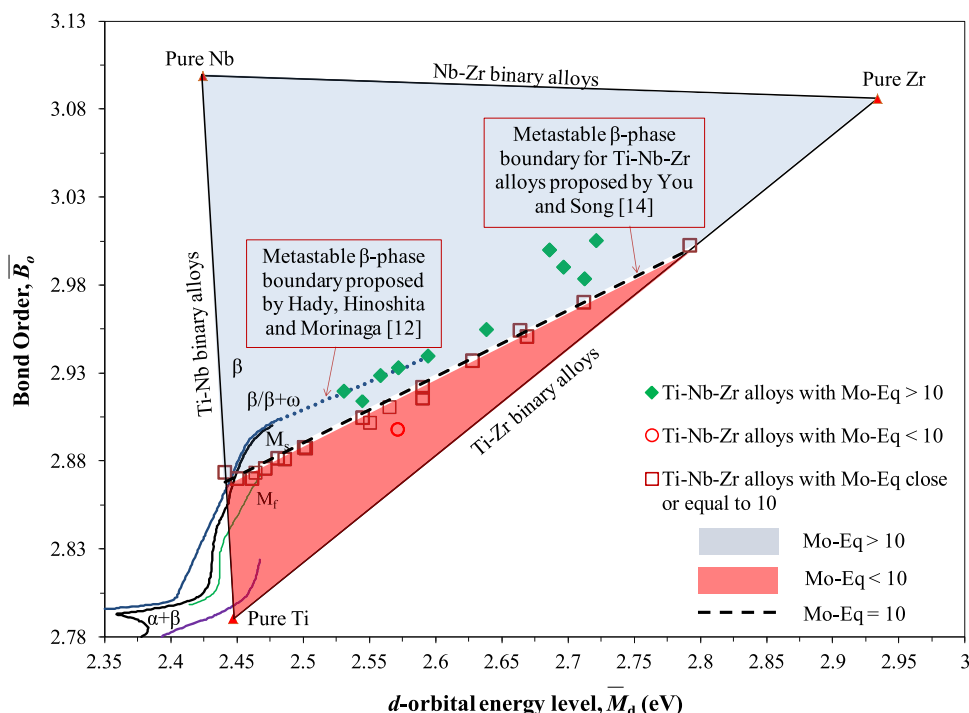


Fig. 3. Ti-Nb-Zr alloys in the $\overline{B}_0-\overline{M}_d$ map.¹³⁻¹⁶

SUMMARY

A new Mo-Eq expression has been formulated for the Ti-Nb-Zr system, i.e., $(\text{Mo-Eq})_{\text{Ti-Nb-Zr}} = 0.238\text{Nb (wt.\%)} + 0.11\text{Zr (wt.\%)} + 0.97$, which shows good consistency with experimental observations as well as literature data. In addition, it is highly consistent with the well-established d -electron theory in predicting the β -phase stability of various Ti-Nb-Zr alloys. In contrast, none of the existing four Mo-Eq expressions is consistent with the d -electron theory, as well as experimental observations. However, caution should be exercised when the alloy composition goes significantly beyond the bounds of the chemistries discussed in this study. It is proposed that the approach developed in this work can also be applied to the assessment of the β -phase stability in other Zr-containing Ti alloys.

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