

X-ray Diffraction Analysis and Micro Hardness of Ternary Alloy (Ti – 6Al – 4Nb)

Riad Jamal Ibrahim¹, Rasha Hamid Ahmed²

^{1,2}Tikrit University, Collage of Education for Pure Sciences, Physics Department. E-mail: ba230036pep@st.tu.edu.iq

KEYWORDS

Powder Technology,
Annealing,
Mechanical
Properties

ABSTRACT

This research aims to prepare the ternary alloy (Ti-6Al-4Nb) Using powder technology and studying X-ray diffraction analysis and micro hardness of this alloy. Titanium, aluminum, and niobium, powders were mixed together to homogenize the elements, and then the pressing process was carried out under pressure (5Ton) and for a period of time(60sec) For the purpose of obtaining a cohesive alloy in the form of discs, then the heat Treatment process (annealing) was conducted at different temperatures. (650,750,850,950,1050)°C for two hours, the structural properties were studied through X-ray diffraction examination (XRD)And studying the mechanical properties by measuring the micro hardness using the Vickers hardness method before and after annealing, the results of the tests showed (XRD)The phases of the basic materials that make up the alloy, as the crystalline structure of titanium appeared to be of the hexagonal stacked type(HCP)As for aluminum, it appeared in a cubic system with concentric faces(FCC) and niobium in a body-centered cubic system (BCC)A new phase also appeared after the annealing process, which is the phase(AlNbTi₂)Has a right rhombic crystal structure(Orthorhombic)The results of the micro hardness test (Vickers) showed that the annealing process has a positive effect on improving the mechanical properties, as the hardness value increases when temperatures increase.

1. Introduction

Powder technology is one of the most important methods for preparing alloys that are characterized by their ability to produce difficult-to-form pistons with uncomplicated shapes that have distinctive properties and excellent crystal structures and high purity using pressure without using thermal treatments [1]. The chemical composition, purity of the powder, and the shape and size of the powder particles are among the distinctive characteristics of powders that improve the physical properties and control the microstructure to reach the required homogeneity [2,3]. The pistons resulting from mixing the elements are characterized by suitable structural and physical properties that are better than the properties of the pure elements themselves that are relied upon in engineering applications [4]. To improve and develop the properties of the pistons, they are thermally treated by annealing, as heat has an effect on the properties of the microstructure of the annealed alloy [5]. The alloys are heated to a temperature of (0.9-0.7) from their melting point and then left to cool slowly to obtain a dense, solid, cohesive, homogeneous body with a high density [6,7]. The resulting material that includes more than one metal One is called an alloy [8], as an alloy is a mixture of two or more materials (with metallic properties) in which atoms or molecules of one material are randomly distributed throughout another material in different proportions. The alloy resulting from mixing two elements is called a binary alloy (Binary alloys) and may be a mixture of three elements, which is called a ternary alloy (Ternary alloys). Alloys are divided into two main sections, which are (ferrous and non-ferrous alloys). Ferrous alloys have good physical properties [9] and alloys have many wide applications in the field of industry [10,11]. Titanium is one of the metals with good physical properties, such as high tensile strength, density, malleability, corrosion resistance, and its ability to merge with aluminum, niobium, vanadium, nickel, and other metals to produce strong and lightweight alloys [12,13]. As for aluminum, it has many

desirable physical properties, as it is flexible, lightweight, a good reflector of visible light, malleable, malleable, and highly durable and resistant to corrosion, so it forms many strong alloys [14]. As for niobium, it is characterized by its high density and good resistance to corrosion and is added to other metals. To improve the strength and durability of these metals[15].

2. Practical part In this study

the triple alloy was prepared using titanium materials of American origin with a purity of 99.95% and a percentage of 90%, aluminum of German origin with a purity of (99.98%) and a percentage of 6%, niobium of Indian origin with a purity of (99.99%) and a percentage of 4%, as the materials were weighed in the mentioned proportions and mixed manually with an agate mortar for (½ h) to obtain the required homogeneity, 6 identical alloys were prepared with the same components and mentioned proportions and the pressing process was carried out with a hydraulic press type (HONMAKSAN) of Turkish origin, using cold pressing technology and using the unidirectional pressing method for the mixed powder, as a pressing pressure of (5 Ton) was applied for a period of time (60sec) to obtain six cohesive and strong samples of the alloy, which were (20mm) in diameter and (15mm) in thickness. The alloys are introduced into the electric furnace (Muffle Furnace) with Chinese origin, its temperature reaches °C (1200) by placing each sample in a ceramic crucible and exposing it to the following temperatures: °C (650, 750, 850, 950, 1050) for two hours. Thus, the samples will be ready for laboratory tests. X-ray diffraction analysis was performed using an American-made (PANALYTICAL'SAERIS) device at room temperature for the purpose of identifying the crystalline structure and new phases formed in the alloy. As for the results of the microhardness test (Vickers), they showed that the annealing process has a positive effect on improving the mechanical properties, as the hardness value increases with increasing temperatures.

3. Results and discussion

Structural properties (X-ray diffraction (XRD)): -

X-ray diffraction (XRD) was studied for the basic materials that make up the prepared ternary alloy (Ti-6Al-4Nb) and to know the crystal structure of the materials, the results showed the phases of the basic materials that make up the alloy, the appearance of the crystal structure of titanium of the hexagonal packed type (HCP) and of aluminum of the face-centered cubic type (F.C.C) and of niobium of the body-centered cubic type (B.C.C). The difference in the crystal structures of the materials involved in preparing the alloy led to the emergence of a new phase after the annealing process that took place at different temperatures °C (650,750,850,950,1050) for a period of (2h), which is the phase (AlNbTi₂) with the orthorhombic crystal structure (Orthorhombic) According to the standard card of the American Society for Testing Materials (ASTM) numbered (00-152-2558), Figure (1) shows the appearance of diffraction peaks for the alloy components before and after annealing at different temperatures. We notice that with increasing the annealing temperature, the intensity level of the peaks

decreases for the base material (titanium) after the annealing process. As for the rest of the components (aluminum, niobium), there is a variation in their intensity between rising and falling according to the temperature. This is due to their proportion in the prepared alloy. The appearance of the (AlNbTi₂) phase indicates the presence of phase changes resulting from the interaction of the materials with each other due to good mixing and the similarity of the grain sizes of the materials used and the exposure of the model to high and different temperatures, which led to new bonds that formed the (AlNbTi₂) phase [16,17].

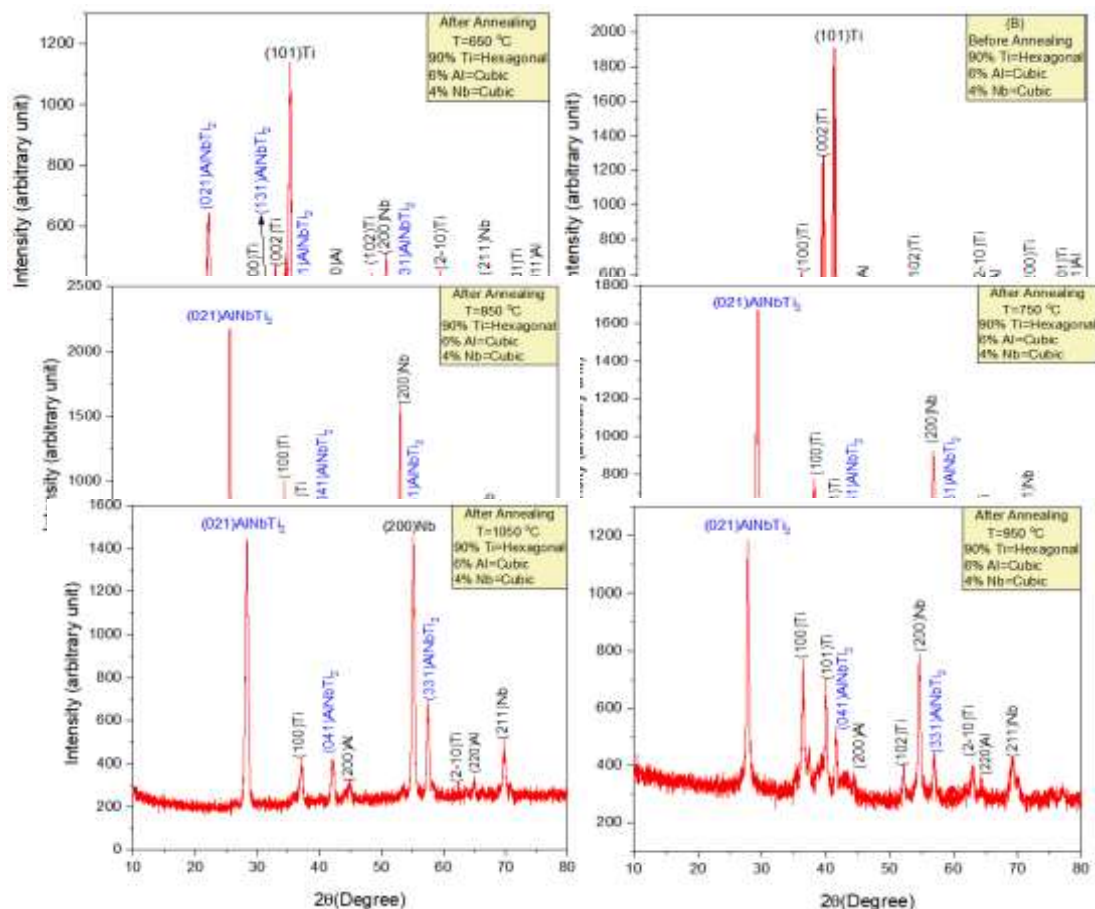


table (1) shows the results of X-ray diffraction of the alloy (Ti-6Al-4Nb). We see the values of the grain size, the angle at which the diffraction peaks appeared, Miller coefficients, and the width of the midpoint of the peak (FWHM). Obtaining this information shows the extent of homogeneity of the alloy components with each other and the possibility of forming new phases. The phase (AlNbTi₂) appeared at a temperature of °C (650) for the first time at the angle (27.392°, 37.0117°, 42.1505°), while at a temperature of °C (750) the phase appeared at the angles (27.8314°, 42.6406°, 57.0052°), while at a temperature of °C (850) the phase appeared at the angles (27.4865°, 42.0611°, 57.4206°), and at a temperature of °C (950) it appeared at the angles (27.8489°, 42.589°, 57.9319°), at a temperature of °C (1050) it appeared at the angles (27.2776°, 42.1121°, 57.434°). We noticed the repetition of the same angles for temperatures (750°, 850°, 950°, 1050°). I concluded that the annealing temperatures affected

the alloy to form the new phase with almost the same effect, with differences in the grain sizes and the width of the midpoint of the peak at those angles. If we compare the appearance of the phase at different temperatures at each angle, we see a small displacement of some peaks as a result of the stresses resulting from the annealing process and the densities of the materials entering into the alloy composition and the mixing ratios [18,19].

Temperatures (°C)	2Theta (Degree)	FWHM (Deg)	d _{hkl} Exp (°A)	C.S (nm)	hkl	Phase	System	Card No
Before Annealing	35.5609	0.2758	2.5225	35.7877	(100)	Ti	Hexagonal	00-900-8517
	38.8933	0.3166	2.3137	32.5850	(002)	Ti	Hexagonal	00-900-8517
	40.614	0.1512	2.21956	69.9563	(101)	Ti	Hexagonal	00-900-8517
	44.7843	0.2177	2.00511	51.9652	(200)	Al	Cubic	00-150-2689
	52.4211	0.359	1.71374	36.6746	(102)	Ti	Hexagonal	00-900-8517
	62.3687	0.4214	1.46657	41.0845	(2-10)	Ti	Hexagonal	00-900-8517
	65.5289	0.2057	1.42335	94.2333	(220)	Al	Cubic	00-150-2689
	70.5264	0.447	1.32607	53.8825	(200)	Ti	Hexagonal	00-900-8517
	76.5686	0.4526	1.24329	76.3759	(201)	Ti	Hexagonal	00-900-8517
	78.6167	0.2406	1.21595	169.0847	(311)	Al	Cubic	00-150-2689
650	27.392	0.5015	3.14101	18.0327	(021)	AlNbTi ₂	Orthorhombic	00-152-2558
	35.917	0.5674	2.49831	17.4736	(100)	Ti	Hexagonal	00-900-8517
	37.0117	0.4742	2.42689	21.2052	(131)	AlNbTi ₂	Orthorhombic	00-152-2558
	38.4898	0.556	2.33703	18.4503	(002)	Ti	Hexagonal	00-900-8517
	40.0523	0.4646	2.24938	22.5780	(101)	Ti	Hexagonal	00-900-8517
	42.1505	0.6465	2.14214	16.7522	(041)	AlNbTi ₂	Orthorhombic	00-152-2558
	44.3808	2.2386	2.03953	5.0186	(200)	Al	Cubic	00-150-2689
	52.0552	0.4994	1.7247	26.1475	(102)	Ti	Hexagonal	00-900-8517
	55.2325	0.4572	1.66175	30.7975	(200)	Nb	Cubic	00-151-2524
	57.4975	0.4033	1.60156	37.0519	(2-10)	Ti	Hexagonal	00-900-8517
	62.4251	0.5048	1.4654	34.3614	(220)	Nb	Cubic	00-151-2524
	70.0301	0.971	1.34247	24.2126	(200)	Ti	Hexagonal	00-900-8517
	76.0164	0.667	1.25094	49.8176	(201)	Ti	Hexagonal	00-900-8517
	78.5006	0.5324	1.23065	75.6508	(311)	Al	Cubic	00-150-2689
750	27.8314	0.21	3.20298	43.2368	(021)	AlNbTi ₂	Orthorhombic	00-152-2558
	35.4761	0.446	2.46129	22.1072	(100)	Ti	Hexagonal	00-900-8517
	40.1371	0.4568	2.24483	22.9921	(101)	Ti	Hexagonal	00-900-8517
	42.6406	0.4125	2.16718	26.4612	(041)	AlNbTi ₂	Orthorhombic	00-152-2558
	44.444	0.4351	2.03677	25.8486	(200)	Al	Cubic	00-150-2689
	52.3823	0.3319	1.74526	39.6342	(102)	Ti	Hexagonal	00-900-8517
	55.6991	0.425	1.67668	33.5252	(200)	Nb	Cubic	00-151-2524
	57.0052	0.4079	1.61421	36.1479	(331)	AlNbTi ₂	Orthorhombic	00-152-2558
	62.0392	0.4659	1.47344	36.7572	(2-10)	Ti	Hexagonal	00-900-8517
	65.4012	0.4117	1.44553	46.8530	(220)	Al	Cubic	00-150-2689
	69.3004	0.5686	1.35481	39.9510	(211)	Nb	Cubic	00-151-2524
	78.4081	0.6496	1.25952	52.5971	(311)	Al	Cubic	00-150-2689
	27.4865	0.1633	3.24239	55.4264	(021)	AlNbTi ₂	Orthorhombic	00-152-2558
850	35.9087	0.1994	2.43343	50.3605	(100)	Ti	Hexagonal	00-900-8517
	40.0026	0.4871	2.25206	21.5194	(101)	Ti	Hexagonal	00-001-1292
	42.0611	0.1891	2.14648	57.1923	(041)	AlNbTi ₂	Orthorhombic	00-152-2558
	44.8639	0.2035	2.01868	55.6681	(200)	Al	Cubic	00-150-2689
	55.1021	0.2212	1.66537	63.4477	(200)	Nb	Cubic	00-151-2524
	57.4206	0.19	1.60352	78.4822	(331)	AlNbTi ₂	Orthorhombic	00-152-2558
	62.4531	0.2198	1.46482	78.9895	(2-10)	Ti	Hexagonal	00-900-8517
	65.7787	0.2371	1.43801	82.5452	(220)	Al	Cubic	00-150-2689

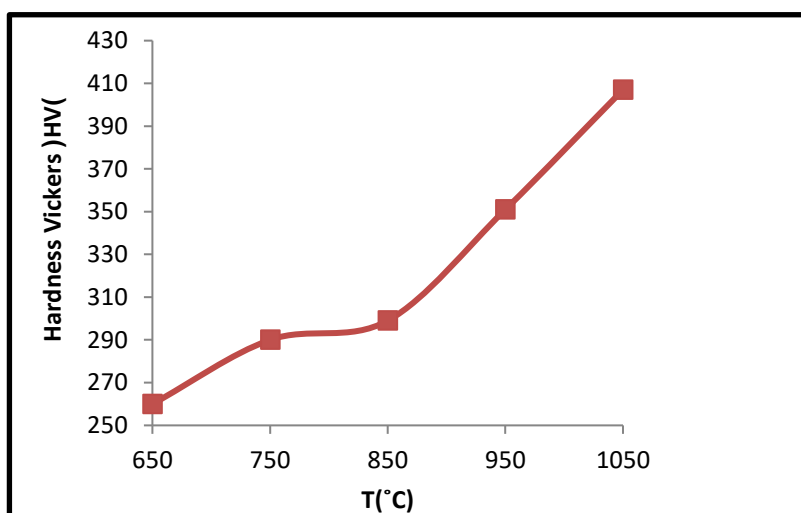
	69.7063	0.298	1.34791	77.6871	(211)	Nb	Cubic	00-151-2524
	70.481	0.2241	1.33498	107.2361	(200)	Ti	Hexagonal	00-900-8517
	78.2272	0.2957	1.23433	133.0873	(311)	Al	Cubic	00-150-2689
950	27.8489	0.2909	3.20101	31.2176	(021)	AlNbTi ₂	Orthorhombic	00-152-2558
	35.4127	0.3628	2.46543	27.1556	(100)	Ti	Hexagonal	00-900-8517
	40.9748	0.4719	2.25357	22.5366	(101)	Ti	Hexagonal	00-001-1292
	42.589	0.3567	2.16975	30.5752	(041)	AlNbTi ₂	Orthorhombic	00-152-2558
	44.3182	0.3283	2.04226	34.1839	(200)	Al	Cubic	00-150-2689
	52.1304	0.2909	1.7531	44.9642	(102)	Ti	Hexagonal	00-900-8517
	55.6194	0.3458	1.67894	41.1198	(200)	Nb	Cubic	00-151-2524
	57.9319	0.3469	1.61611	43.5959	(331)	AlNbTi ₂	Orthorhombic	00-152-2558
	62.9503	0.5084	1.47531	34.7291	(2-10)	Ti	Hexagonal	00-900-8517
	65.318	0.513	1.4472	37.4823	(220)	Al	Cubic	00-150-2689
	69.2284	0.9775	1.35605	23.1620	(211)	Nb	Cubic	00-151-2524
	27.2776	0.5501	3.15345	16.4226	(021)	AlNbTi ₂	Orthorhombic	00-152-2558
1050	38.0391	0.594	2.42516	17.1632	(100)	Ti	Hexagonal	00-900-8517
	42.1121	0.5451	2.144	19.8565	(041)	AlNbTi ₂	Orthorhombic	00-152-2558
	44.7861	0.6179	2.022	18.3091	(200)	Al	Cubic	00-150-2689
	52.4077	0.6214	1.71414	21.1815	(102)	Ti	Hexagonal	00-001-1292
	55.0977	0.4733	1.66549	29.6494	(200)	Nb	Cubic	00-151-2524
	57.434	0.3941	1.60317	37.8510	(331)	AlNbTi ₂	Orthorhombic	00-152-2558
	62.8699	0.3376	1.43621	52.1562	(2-10)	Ti	Hexagonal	00-900-8517
	65.2511	0.2265	1.40958	84.6786	(220)	Al	Cubic	00-150-2689

4. Mechanical tests (Vickers hardness)

The conditions for preparing the alloy greatly affect the properties of the resulting alloy. The powder technology method followed by the annealing process was a positive indicator of improving the studied mechanical property, which is Vickers hardness, as well as improving its physical properties, as what enhances the increase in the Vickers hardness values of the alloy is the decrease in its porosity and grain size, as The lower these two values, the higher the hardness values, that is, the relationship is inverse, because the distribution of the components of the alloy will be better in this case, as shown in Table (2) and Figure (2). The results of the Vickers hardness examination confirmed that the hardness increases with the increase in the annealing temperature, and that temperature affects. It changes the crystalline arrangement and makes it more regular, which leads to an increase in the bonding strength of the atoms in the material. The formation of a new compound is (AlNbTi₂) as this compound works to strengthen the material and reduce its porosity and grain size, which increases the hardness values and improves the mechanical properties of the alloy [20,21].

Table (2). Vickers hardness values before and after annealing

Temperature °C	Hardness HV
Before annealing	244
650	260
750	290
850	299
950	351
1050	407



The Shape (2) The Relationship Between Annealing

5. Conclusion and future scope

We conclude from the study that the powder technology technique is a successful method for preparing alloys with good mechanical qualities and properties, and that the annealing time of two hours helped improve all the studied properties of the alloy prepared at the temperatures. (650,750,850,950,1050)°C, and through studying the structural characteristics in the Temperature and Vickers hardness values, the best annealing temperature was(1050) °C for properties (structural, mechanical).

Reference

- [1] Harmer E. Davis, George Earl Trowel and George F. W. Hauck “The Testing of Engineering Materials”, Mc Grew-Hill, Inc., Fourth Edition, 1982.
- [2] 2 - Polmear, Ian, Light alloys: metallurgy of the light metals. Butterworth-Heinemann, 2017
- [3] 3 - Evans, W.J. Optimizing mechanical properties in alpha+ beta titanium alloys. Materials Science and Engineering: . (1998) A, 243(1-2), 89-96
- [4] 4 - K. G. Budinski “Engineering Materials Properties and Selection” 5th ed., Prentice Hall of India (1996).
- [5] 5 - Eng. Materials Technology, W. Bolton, 3ed, British Library Cataloging Pub.Data,1998.
- [6] 6 - Papp, John F. “Niobium (Columbium) and Tantalum” USGS Minerals Yearbook)2006(.
- [7] 7 - Verevkin, A. “Ultrafast superconducting single-photon detectors, for near-infrared-wavelength quantum

- communications.” Journal of Modern. (2004).
- [8] 8- Krebs, Robert E. The History and Use of Our Earth's Chemical Elements: A Reference Guide (2). Westport, CT: Greenwood Press. (2006).
- [9] 9- Lutjering and J. C. Williams, Titanium (springer Verlag. Germany, 2007).
- [10] 10- Ismaeel, Adam, and Cun-shan Wang. "Effect of Nb additions on microstructure and properties of γ -TiAl based alloys fabricated by selective laser melting." Transactions of Nonferrous Metals Society of China 29.5 (2019): 1007-1016.
- [11] 11- Chlebus, Edward; Kuźnicka, Bogumiła; Kurzynowski, Tomasz; Dybała, Bogdan "Microstructure and mechanical behavior of Ti—6Al—7Nb alloy produced by selective laser melting". Materials Characterization. (1 May 2011). 62(5): 488–495.
- [12] 12- Pozzobon, V.; Levasseur, W.; Do, Kh.-V.; et al. "Household aluminum foil matte and bright side reflectivity measurements: Application to a photobioreactor light concentrator design". Biotechnology Reports 25. (2020).
- [13] 13- Craig, W.; Leonard, A. (2019). Manufacturing Engineering & Technology. Scientific e-Resources. p. 215. ISBN:978-1-83947-242-8. Archived from the original on 2021-04-15.
- [14] 14- Frank, WB, Haupin, WE, Vogt, H., Bruno, M., Thonstad, J., Dawless, RK, ... & Taiwo, OA Aluminum. Ullmann's Encyclopedia of Industrial Chemistry. (2000).
- [15] 15- Sivakandhan, C., Loganathan, G. B., Murali, G., Prabhu, P. Marichamy, S., Krishnan, G. S., & Pradhan, R. Material characterization and unconventional machining on synthesized Niobium metal matrix. Materials Research Express, (2020). 7(1), 015018
- [16] 16- Nowak, Isabella; Ziolk, Maria "Niobium Compounds: Preparation, Characterization, and Application in Heterogeneous Catalysis". Chemical Reviews. (1999). 99. 12: 3603–3624. D.
- [17] 17- Patel, Zh.; Khul'ka K. "Niobium for Steelmaking". Metallurgist. (2001). 45. 11–12: 477–480.
- [18] 18-M. G. Buffalo Vic LJ.J. Ivanovic E.R, Radmilovic V and popov KI, “The Effect of particle Structure on Apparent Density, of Electrolytic Copper Powder”, Serbian Chemical Society, 2001 pp. (923-933).
- [19] 19- M. B. Bever, "Encyclopedia of Materials Science and Engineering", 1st edition, Vol. 5, 1986.
- [20] 20 - Lopes, ESN, et al. “Effects of double aging heat treatment on the microstructure, Vickers hardness and elastic modulus of Ti–Nb alloys.” Materials characterization 62.7 (2011): 673-680
- [21] 21 - Cardoso, Giovana Collombaro, Pedro Akira Bazaglia Kuroda, and Carlos Roberto Grandini. "Influence of Nb addition on the structure, microstructure, Vickers microhardness, and Young's modulus of new β Ti-xNb-5Mo alloy system." Journal of Materials Research and Technology 25 (2023): 3061-3070.