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Influence of Heat Treatment Conditions on Microstructure of Ti-6Al-7Nb Alloy As Used Surgical Implant Materials

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Abstract

The study involves mechanical deformation and heat treatment effect on the microstructure of the Ti-6Al-7Nb alloy which is used as surgical implanted materials. The observed properties of (alpha-beta) Ti-based alloy are strongly dependent on their microstructures. These alloys are heat treated by solution treatment and aging (STA) as an effective strengthening method for (alpha-beta) titanium alloys.

Ti-6Al-7Nb alloy is hot rolled in the (alpha-beta) field and subjected to solution treatment above and below its beta transformation temperature. The solution treatments are applied at three different temperatures (850^{o}C, 930^{o}C and 950^{o}C) for one hour to these treatments. The solution treatment specimens are water quenched (WQ), normalizing [air cooled (AC) and annealed,[furnace cooled(FC)] and subsequently aged the quenched and normalized specimens at 550^{o}C for 4 hours.

Changes in the microstructure were observed from heat treatment action using optical microscopy, Atomic Absorption Spectroscopy (AAS) and phases analysis by X-ray diffraction (XRD). The microstructure examination results for as received alloy indicate alpha grains within a b- **transformed matrix. The results also indicate that the grain size and percent of** a /b **for the heat treated specimens depend on heat treatment type and cooling media. The microstructure of specimens heat treated at 950^{o}C with air cooled shows fine duplex (**a /b**) structures which have excellent properties for surgical implanted field applications.**

Keyword: Titanium heat treatment, titanium alloy, implant material, heat treatment ,surgical materials.

الخلاصة

تتضمن الدراسة تأثير التشكيل الميكانيكي والمعاملات الحراريه على التراكيب المجهرية لسبيكة(Ti-6Al-7Nb) المستخدمة في الزوارع الجراحية. لوحظ بأن خواص سبيكة التيتانيوم ثنائية الطور (a /b) تعتمد بشدة على التراكيب المجهرية هذه. تعامل هذه السبائك بطريقة المعاملة المحلولية والتعتيق كطريقة فعالة لتقوية سبائك التيتانيوم ثنائية الطور (a /b). يتم

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درفلة هذه السبيكة على الساخن في منطقة (الفا - بيتا) وتجري لها معاملة محلولية فوق وتحت درجة حرارة تحول الطور بيتا.
استخدمت المعاملة المحلولية بثلاث درجات حرارية هي (٨٥٠م° , ٩٣٠ م° , ٩٥٠ م°) وبزمن قدره ساعة واحدة لجميع تلك المعاملات . تبرد هذه السبيكة بعد المعاملات اعلاه بثلاث اوساط تبريد هي التبريد السريع (تبريد بالماء) ، والمعادلة (تبريد بالهواء) والتلدين (تبريد داخل الفرن). يتم بعدها اجراء عملية التعتيق عند ٥٥٠ م° لمدة اربعة ساعات للنماذج التي تم تبريدها (بالماء والهواء).
لوحظ حصول تغيرات في البنية المجهرية بفعل هذه المعاملات بأستخدام المجهر الضوئي، طيف الامتصاص الذري وتحليل الاطوار بأستخدام حيود الاشعة (XRD). أظهرت نتائج الفحص الدقيق للسبيكة (كما استلمت) على حبيبات الفا في ارضية بيتا .تبين النتائج ايضاً ان نسبة وحجم حبيبات الفا / حبيبات بيتا للنماذج التي عوملت حرارياً بأنها تعتمد على نوع تلك المعاملة ووسط التبريد. تشير نتائج البنية المجهرية لنماذج السبيكة المعاملة حرارياً عند ٩٥٠ م° وتبريد بالهواء الى وجود تركيب مزدوج دقيق(a /b) الذي يمتاز بأفضل الخواص التطبيقية في مجال الزوارع الجراحية.

1- Introduction

Ti-6Al-4Vand Ti-6Al-7Nb alloys have been relatively popular biomaterials for surgical implants due to their biocompatibility characterization but the major problem in using them for bio-application is their poor wear resistance [1]. Though extensive research works on titanium alloy have been made to develop the relationship between microstructure and mechanical properties [2,3,4]. Over the past decades, quantitative analysis of basic biomaterials properties have been utilized to better optimizes biocompatibility profiles for surgical implant devices [5].

Several titanium alloys of technological interest contain a two-phase mixture of alpha (h.c.p) and beta (b.c.c) titanium polymorphs.

The relative phase percentages depend mainly on two factors: thermal history of the material and alloying elements [6, 7]. In pure titanium, the alpha phase is stable up to 882^{o}C, whereas the beta phase is stable from this temperature to melting temperature (1670^{o}C). The alpha to beta transition temperature is influenced by the presence of alloying elements; Aluminum stabilizes the alpha phase, whereas V, Mo, Nb and Ta stabilize the beta phase [8,9,10].

Alpha and near alpha titanium alloys can be stress relieved and annealed, but high strength can not be developed in these alloys by any type of heat treatment. The commercially beta alloys are, in reality, metastable alloys. When these alloys are exposed to selected elevated temperatures, the retained beta phase decomposes and strengthening occurs [11]. For beta alloys, stress relieving and aging treatments can be combined and annealing and solution treating may be identical operations [12].

Hot rolling accompanied with post heat treatment is the most effective method; this method can be used for strengthening of alpha-beta titanium alloys and to refine its morphology [11].

In any case, knowing the exact polymorphic phase composition is very important. Unfortunately, in many alloys used in technological applications, including Ti-6Al-4V and Ti-6Al-7Nb alloys, the beta phase is in minority with respect to the alpha phase, and its Bragg reflections are weak and mostly overlap with those of the alpha phase [7].

This study is an attempt to point out some of the microstructural features of (α+β) Ti-6Al-7Nb alloy after different heat treatments. The main target is to obtain the so-called duplex microstructure and compared with other microstructures developed by post heat treatment after hot rolling.

2- Experimental Parts

2-1 Alloy Preparation

Ti-6Al-7Nb alloy was prepared by DMRL (Defence Metallurgical Research Laboratories, Hyderabad-India). Alloy preparation consisted of melting by non-consumable vacuum arc melting and casting in a water-cooled copper crucible under vacuum (< $1x10^{-3}$ mbar). Raw materials were used for melting consisted of titanium sponge, and aluminum-niobium master alloy (6%Al-7%Nb). The ingot was obtained in the form of pancake of 600 gm in weight (as cast alloy). Then the ingot was subjected to deformation (hot rolling) in the α+β phase field (950^{o}C) to obtain 4-mm thickness from a 12 mm thick sheet. The composition of the alloy is determined by chemical analysis.

2-2 Elemental Analysis

To determine the contents of all major and minor alloying elements, chemical analysis, was done on specimens taken from the as cast ingot and the rolled sheet using Atomic Absorption Spectroscopy (AAS 670, Shimadzu, Japan). 22 mg of each specimen was weighed and 10 ml of 1:1 H_2SO_4 (by volume) was added and heated to dissolve the specimens. The solution was made up to 25 ml in a standard flask by the addition of 5% (by volume) of H_2SO_4. Using the similar procedure, blank acid specimen was prepared.

2-3 Phase Analysis

2-3-1 X-Ray Diffraction (XRD)

The phases present in the specimens of Ti-6Al-7Nb and alloys were identified using XRD. The bulk specimen was polished up to mirror finish before the XRD experiment was carried out. Philips 3121 powder X-ray Diffractometer using Cu Kα radiation was used for this analysis.

2-4 Heat Treatment

The thermal treatments were performed in a tubular furnace and the temperature accuracy of the furnace was controlled to within ± 2^{o}C, and the specimens were introduced at room temperature and heat treated along with the furnace to the required temperature. A number of different heat treatments were given to specimen cut from the rolled sheet. A solution annealing in α+β phase field followed by aging was carried out. Three solution temperatures (namely 850^{o}C, 930^{o}C and 950^{o}C) were used. After one hour, the specimens were cooled at various rates, water quenching, air-cooling and slow furnace cooling . All the heat treatments were carried out in inert argon atmosphere. The water-quenched and air cooled specimens were subjected to aging treatment in open-air furnace at 550°C for interval time of 4 hours.

2-5 Metallographic Techniques

Specimens of 10-mm diameter with thickness of 4 mm were cut out from the rolled sheet Ti-6Al-7Nb alloy. These specimens were mounted and then polished with SiC emery papers with different grits started from 80 grit, and continued by 120, 230, 400, 600, 800 and 1000 grit to get flat and scratch free surface. The final polishing was carried out using alpha alumina 1μm, 0.3μm and 0.05μm. These specimens were used for microstructure examinations. The microstructural evolution was investigated by means of optical microscopy using Nikon Type 120, Japan optical microscope.

3- Results and Discussion

3-1-Elemental Analysis

Table 1 lists the weight percentage of the elements contained in the composition of Ti-6Al-7Nb alloy.

3-2 Phase Analysis

Though there is no standard for Ti-6Al-7Nb alloy, we have fitted our results obtained from this analysis according to those reported for the hexagonal α Ti and cubic β Ti [13]. Also, the results have been compared with the reported diffraction data for CPTi and Ti-6Al-4V alloy [7,14]. Figure 1 shows the XRD patterns of CP Ti, Ti-6Al-4V and Ti-6Al-7Nb alloys. From this quantitative fitting, the CP Ti, Ti-6Al-4V and Ti-6Al-7Nb were indexed, as shown in Table 2.The XRD analysis of Ti-6Al-7Nb alloy shows slight change in the 2θ value of α phase reflections of Ti-6Al-7Nb alloy with respect to those in pure Ti and Ti-6Al-4V alloy. These variations are due to the replacement of V atom by Nb of lesser diameter.

Table 1: Chemical Composition (wt.%) of Ti-6Al-7Nb alloy.

Element	Al	Nb	Ta	Fe	Mo	Ti
Ti-6Al-7Nb	6.0	7.2	0.46	0.22	0.005	Balance

Table 2: Miller indices *hkl* and Bragg angle (*2q*), of *a* and *b* phases in CP Ti, Ti-6Al-4V and Ti-6Al-7Nb alloys obtained from Figure 1

Ti-6Al-7Nb		Ti-6Al-4V		CPTi			
2q	d (Å)	2q	d (Å)	2q	d (Å)	hkl-a	hkl-b
35.333	2.583	35.234	2.545	35.073	2.55 6	100	
38.485	2.337	38.319	2.347	38.474	2.337	002	
40.425	2.229	40.201	2.241	40.325	2.234	101	
53.325	1.716	53.048	1.724	53.319	1.716	102	
57.267	1.607	57.141	1.610	-	-		200
63.639	1.461	63.683	1.460	63.13 5	1.471	110	

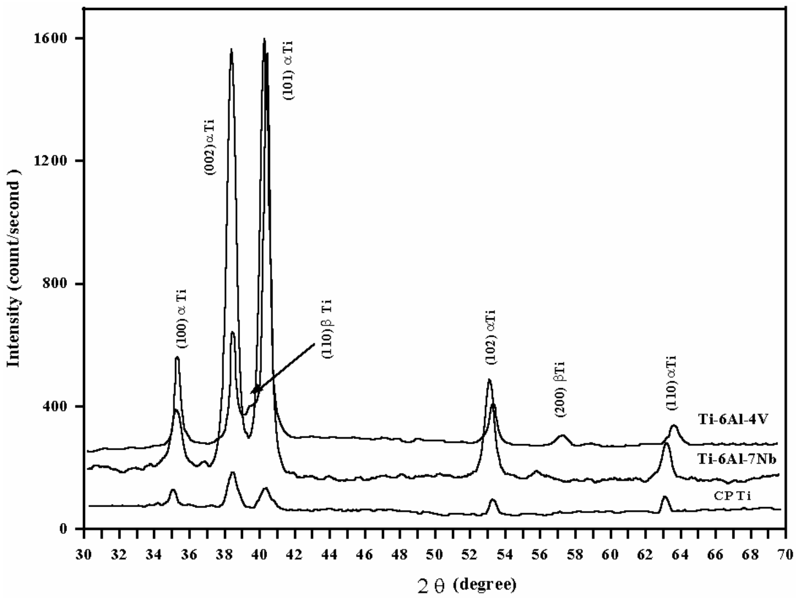

Figure 1: XRD patterns of CPTi, Ti-6Al-4V and Ti-6Al-7Nb alloys.

3-3 Microstructure Evolution

The as-rolled microstructures of Ti-6Al-7Nb and Ti-6Al-4V alloys are shown in Figure 2 a&b respectively. Hot rolling of Ti-6Al-7Nb alloy at 950°C shows microstructure consisted of globular and acicular α grains (white grains) within a β-transformed matrix containing equiaxial grains (dark grains). In region where deformation was intense, the microstructure of α was elongated with flow lines and this is also confirmed by [15]. The mechanical properties of these alloys are very sensitive to the microstructure. The XRD analysis of Cp Ti, Ti-6Al-4V and Ti-6Al-7Nb alloys indicates the presence of α and β phases as shown in table 2. .

Duplex microstructures (primary α in lamellar (α+β) matrix) are superior to fully lamellar microstructures regarding to tensile ductility and resistance to microcrack growth [16]. The cooling rate from homogenization temperature determines the width of α-lamellae in the lamellar structure within the β gain boundaries.

Heat treatment in α+β region resulted in recrystallization of the α into an equiaxed morphology designated as primary α in transformed β. The corresponding microstructures of the deformed and solution treated specimens at 850°C to 950°C with different cooling rates are shown in Figures 3-5. The heat treatments at 850°C and 930°C and water quenched produced equiaxed primary α with fine discontinuous β (Figures 3 a & 4 a), whereas air-cooling reduces the volume fraction of primary α and modified the transformed β matrix (Figures 3 b & 4b). Specimens quenched from 950°C produced structure with less primary α embedded in martensitic α' (Figure 5 a), and air-cooling from the same temperatures shows primary α plus acicular α (Widmanstätten) (Figure 5 b). The furnace cooling resulted in increase of the volume fraction of primary α grains and reduces the transformed β (Figures 3 c, 4c &5 c) which was even less continuous than that observed in the specimens water quenched from 850°C and 930°C.

Thus, in the water quenched and air-cooled specimens, when the temperature was raised from 850°C to 950°C, it tends to decrease the volume fraction of primary α and modify the matrix morphology. Borradaile et.al.[12] confirmed that both solution treatment temperature and the cooling rate from the solutionizing temperature determine the primary α volume fraction matrix. The cooling rate also determines the morphology of the transformed β structure. The β phase could transform to martensite by water quenching while by air cooling the transformation occurred by diffusional mechanism to Widmanstätten α lath structure.

Corrosion resistant and high strength titanium alloys for structural applications are generally two-phase (α+β) alloys [17,18]. Therefore, further studies on the electrochemical behavior for the above alloy are concerned later in the proceeding paper later.

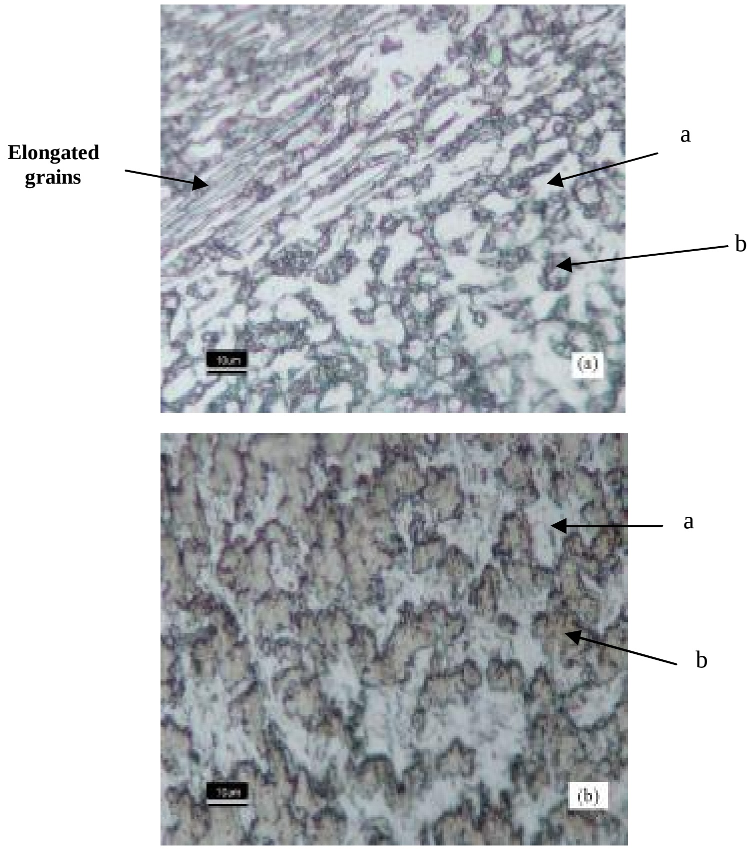

Figure 2 Optical micrographs showing the microstructure of alloys etched by Kroll's reagent containing 10ml of HF, 5ml of HNO3 and 85ml of water :
(a) As-rolled Ti-6Al-7Nb and (b) Ti-6Al-4V alloys.

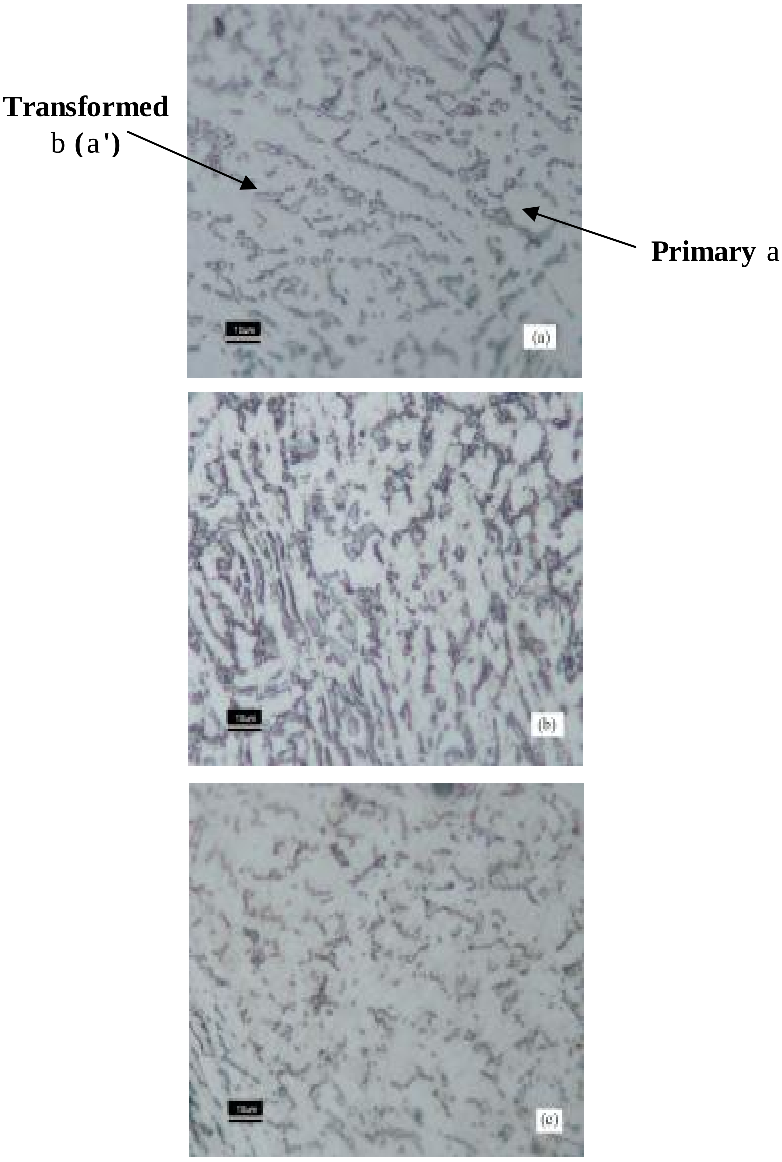

Figure 3 Optical micrographs of Ti-6Al-7Nb rolled alloy solution treated for 1 hour at 850^{o}C and cooled at different rates and etched by Kroll's reagent containing 10ml of HF, 5ml of HNO3 and 85ml of water :
(a) WQ, (b) AC& (c) FC.

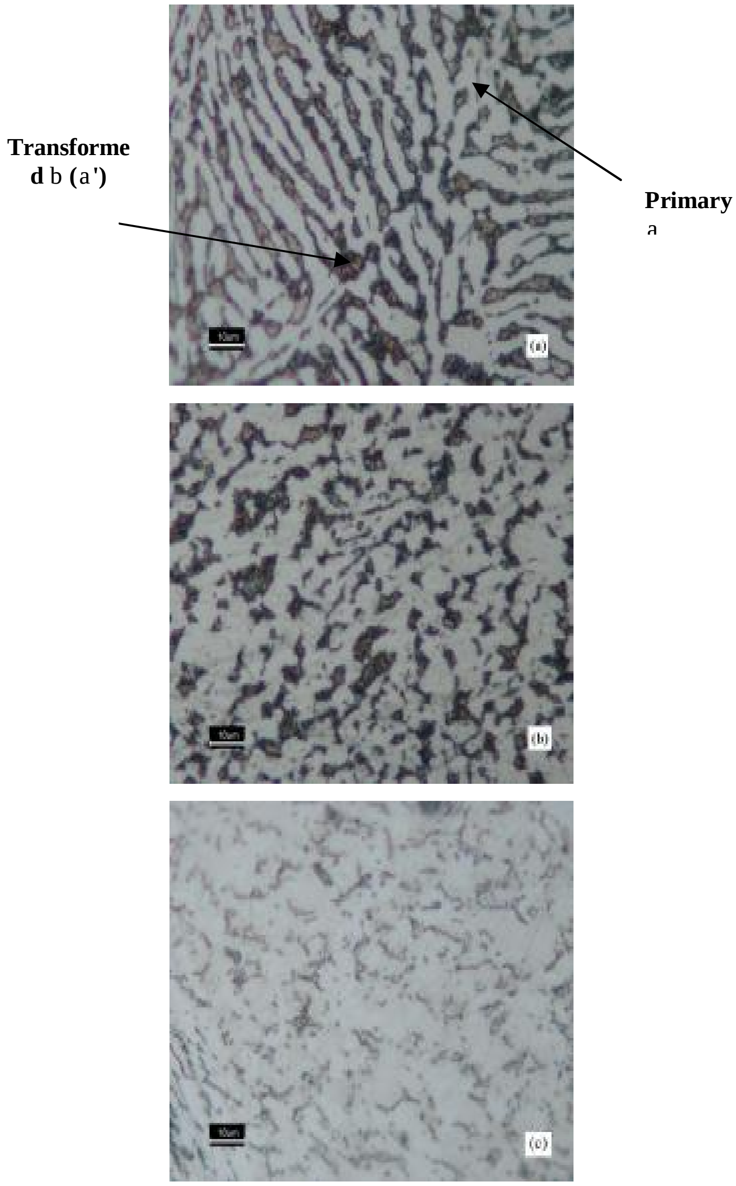

Figure 4 Optical micrographs of Ti-6Al-7Nb rolled alloy solution treated for 1 hour at 930^{o}C and cooled at different rates and etched by Kroll's reagent containing 10ml of HF, 5ml of HNO3 and 85ml of water:
(a) WQ, (b) AC& (c) FC.

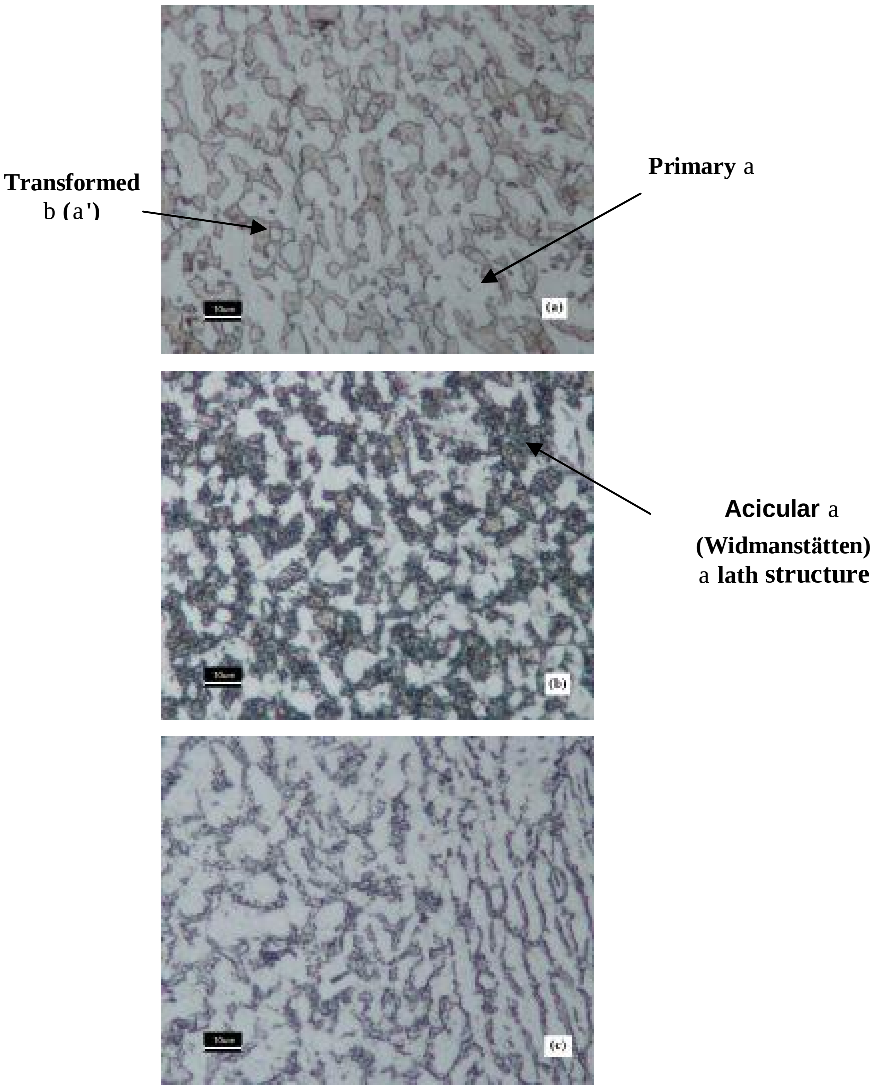

Figure 5 Optical micrographs of Ti-6Al-7Nb rolled alloy solution treated for 1 hour at 950^{o}C and cooled at different rates and etched by Kroll's reagent containing 10ml of HF, 5ml of HNO3 and 85ml of water :
(a) WQ, (b) AC& (c) FC.

4 Conclusions

Heat treatment of Ti-6Al-7Nb alloy components have a marked influence on technologically relevant properties, including residual stress and composition of alpha- beta titanium polymorphous.

The XRD analysis of Ti-6Al-7Nb alloy shows slight changes in the 2q value of alpha phase reflections of Ti-6Al- 7Nb alloy with respect to those in pure Ti and Ti-6Al-4V alloy. These variations are due to the replacement of V atom by Nb of lesser diameter.

The heat treatment of the rolled specimens below the alpha-beta transformation temperature develops the duplex morphology. The globular alpha (primary alpha) decreases and modified the transformed beta matrix as the temperature increases towards the beta transus.

The most common microstructure relevant to Ti-6Al-7Nb alloy are the transformed beta (Widmanstatten) and equiaxed (alpha-beta) region which have been widely used as an effective strengthening method for alpha-beta alloys.

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