

M. MOTYKA,* J. SIENIAWSKI*, W. ZIAJA*, G. MRÓWKA-NOWOTNIK*

MICROSTRUCTURAL CHARACTERIZATION OF QUENCHED AND PLASTICALLY DEFORMED TWO-PHASE $\alpha+\beta$ TITANIUM ALLOYS

CHARAKTERYZACJA MIKROSTRUKTURY PRZECHŁADZANYCH I ODKSZTAŁCANYCH PLASTYCZNIE DWUFAZOWYCH STOPÓW TYTANU $\alpha+\beta$

Development of microstructure in two-phase $\alpha+\beta$ titanium alloys is realized by thermomechanical processing – sequence of heat treatment and plastic working operations. Analysis of achieved results indicates that hot plastic deformation – depending on deformation degree – causes significant elongation of α phase grains. Following heat treatment and plastic deformation processes lead to their fragmentation and spheroidization. Characterization of microstructure morphology changes during thermomechanical processing of quenched Ti-6Al-4V and Ti-6Al-2Mo-2Cr alloys is presented in the paper. The effect of martensitic phase α' (α'') on microstructure development in plastic deformation process was confirmed.

Keywords: titanium alloys, microstructure, quenching, plastic deformation, spheroidization of α phase.

Kształowanie mikrostruktury dwufazowych stopów tytanu $\alpha+\beta$ realizowane jest w procesie cieplno-plastycznym będącym sekwencją operacji obróbki plastycznej i przeróbki plastycznej. Analiza uzyskanych wyników badań wskazuje, że odkształcanie plastyczne na gorąco – w zależności od stopnia odkształcenia - powoduje wydłużanie ziarn fazy α . Kolejne operacje obróbki cieplnej lub odkształcania plastycznego prowadzą do ich fragmentacji i sferoidyzacji. W pracy przedstawiono charakterystykę zmian morfologii składników mikrostruktury stopów Ti-6Al-4V oraz Ti-6Al-2Mo-2Cr poddanych przechłodzaniu na początkowym etapie procesu cieplno-plastycznego. Potwierdzono oddziaływanie fazy martenzytycznej α' (α'') w badanych stopach na przebieg procesu kształtowania ich mikrostruktury podczas odkształcania plastycznego.

1. Introduction

Development of microstructure in two-phase $\alpha+\beta$ titanium alloy is usually realized in several plastic working and heat treatment operations – i.e. in thermomechanical processing (TMP) [1,2]. Experimental results obtained for Ti-6Al-4V alloy indicate that uniformity, size and morphology of its microstructural constituents depend mainly on [3]:

- plastic deformation conditions;
- recrystallization's effects;
- stereological parameters of phases in initial microstructure (before plastic deformation).

Structural defects, generated during plastic deformation, control nucleation process and nuclei growth during recrystallization. Therefore increase of deformation degree and strain rate causes higher grain refinement after TMP. It was found that time of recrystallization has a stronger influence on final grain size than its temperature [3].

Phase morphology of two-phase titanium alloys determines their applications. Two principal types of

microstructure are usually developed: lamellar and equiaxed. Development of lamellar microstructure is mainly controlled by cooling rate during $\beta \Rightarrow \alpha+\beta$ phase transformation whereas formation of equiaxed microstructure requires contribution of plastic deformation process [2,4,5].

Due to high strength titanium alloys are deformed in hot plastic working process causing deformation and elongation of α phase grains. Evolution of their microstructure on following stages of thermomechanical processing is principally related to spheroidization of α phase lamellae [6]. It was found that in Ti-6Al-4V alloy such microstructural transformation is caused by recrystallization process during hot deformation and following heat treatment <Fig. 1> [7]. Recrystallization occurs initially in α -phase lamellae and leads to formation of subgrains and successively grains of α phase <Fig. 1b>. Surface tension of α/α grain boundaries and their orientation regarding α/β interphase cause local β -phase growth into α/α grain boundary and lead to migration of α/β interphases <Fig. 1c>. Therefore it results in grain growth of recrystallized α_R phase which reach size higher than lamella thickness and touch adjacent α lamellae <Fig. 1d>.

* RZESZOW UNIVERSITY OF TECHNOLOGY, DEPARTMENT OF MATERIALS SCIENCE, 12 POWST. WARSZAWY AV., 35-959 RZESZOW, POLAND

* Corresponding author: motyka@prz.edu.pl

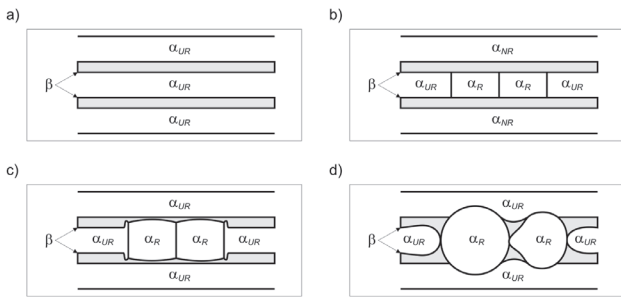


Fig. 1. Scheme of dynamic or metadynamic recrystallization in α -phase lamellae in Ti-6Al-4V alloy: a) unrecrystallized α -phase lamellae - a_{UR} , b) formation of nuclei of new grains - a_R , c, d) grain growth of a_R phase [7]

Spheroidization of α lamellae can be also realized through their fragmentation caused by plastic deformation in the temperature range of $\alpha+\beta \rightarrow \beta$ transformation. It is assumed that stress caused by pile-up of dislocations leads to shearing and rotation of grains – initial stage of fragmentation. Formation of low-angle and consequently high-angle boundaries in the smallest cross-section of α lamellae causes their partition and spheroidization <Fig. 2a> [6,8,9]. It is generally accepted that fragmentation of α phase lamellae can be caused by shear bands forming during plastic deformation <Fig. 2b> [9].

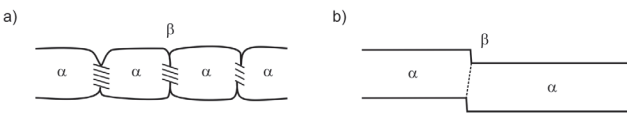


Fig. 2. Fragmentation mechanism of α phase lamellae: a) formation of dislocation substructure, b) localized shear [6,9]

According to microstructural evolution mechanisms mentioned before size of newly formed equiaxed α grains is related to the thickness of fragmented lamellae. Development of fine-grained equiaxed microstructure by fragmentation and spheroidization is possible in two-phase $\alpha+\beta$ titanium alloys having fine-grained lamellar microstructure. Lamellae refinement in these alloys is achieved by fast cooling from $\alpha+\beta$ or β regions [2-5]. H. Inagaki introduced β quenching before hot rolling operation in thermomechanical processing of Ti-6Al-4V alloy [10,11]. Developed microstructure was composed of thin and highly elongated α phase grains separated by thin film of β phase. It was particularly interesting that examined alloy exhibited high superplasticity despite its microstructure was not equiaxed. Based on the own results [12] it was revealed that such microstructure is able to transform into equiaxed one during initial stage of superplastic deformation.

It seems that martensitic transformation $\beta \rightarrow \alpha'$ (α'') in thermomechanical processing of two-phase titanium alloys has particular influence on phase morphology in developed microstructure. The aim of presented paper is to describe microstructural transformation during thermomechanical processing of Ti-6Al-4V and Ti-6Al-2Mo-2Cr alloys including β quenching and following open die forging at the temperature of $\alpha+\beta \rightarrow \beta$ range.

2. Materials and research methodology

Martensitic two-phase $\alpha+\beta$ Ti-6Al-4V and Ti-6Al-2Mo-2Cr titanium alloys bars (diameter of 16 mm) were examined. Alloys contain different β -stabilizers – V in Ti-6Al-4V and Mo, Cr in Ti-6Al-2Mo-2Cr <Tab 1>.

On the basis of dilatometric results and previous investigations conditions of heat treatment and plastic deformation were defined and thermomechanical processing (TMP) was worked out. Preliminary heat treatment – quenching – was carried out from the temperature of 1050°C – stable β phase range. Plastic deformation in the $\alpha+\beta \leftrightarrow \beta$ phase transformation range (900°C) was performed in WSK “PZL Rzeszow” S.A. using open die forging process with forging reduction of about 50% <Fig. 3>.

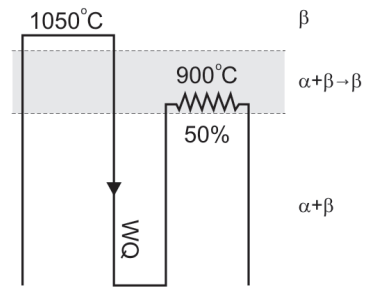


Fig. 3. Scheme of thermomechanical processing (TMP) of Ti-6Al-4V and Ti-6Al-2Mo-2Cr alloys (WQ - water quenching)

Light microscope (LM) Nikon Epiphot 300 equipped with digital camera Nikon DS-1U and transmission electron microscopes (TEM) Tesla BS540 were employed for microstructural observation. Metallographic specimens were etched using Kroll’s reagent. Evaluation of stereological parameters of microstructural constituents was performed on longitudinal etched microsections using quantitative metallography methods and image analysis software Aphelion 3.2. Following parameters were determined:

- Ti-6Al-4V alloy with equiaxed initial microstructure: grain size of α phase expressed by length of sides of rectangular

TABLE 1
Chemical composition of titanium alloys

Alloy	Al	V	Mo	Cr	Fe	Si	Ti
	wt. %						
Ti-6Al-4V	6.78	4.38	-	-	0.18	0.33	bal.
Ti-6Al-2Mo-2Cr	6.87	-	3.16	1.57	0.45	0.65	bal.

circumscribed on grain section – a_α and b_α , elongation factor of α phase grains – f_α and volume fraction of α phase – $V_{V\alpha}$.

- Ti-6Al-2Mo-2Cr alloy with lamellar microstructure: grain size of primary β phase expressed by length of sides of rectangular circumscribed on grain section – $a_{\beta prim}$ and $b_{\beta prim}$, elongation factor of primary β phase grains – $f_{\beta prim}$, size of the colony of parallel α lamellae – R , thickness of α -lamellae – g and volume fraction of α phase – $V_{V\alpha}$.

3. Results and discussion

In initial state - before TMP - equiaxed microstructure in Ti-6Al-4V alloy was observed – spheroidal α phase grains separated by thin layer of β phase <Fig. 4a>. The initial microstructure of Ti-6Al-2Mo-2Cr is lamellar – colonies of parallel α and β lamellae formed in the primary β phase grains - β_{prim} - elongated in plastic flow direction <Fig. 4b>.

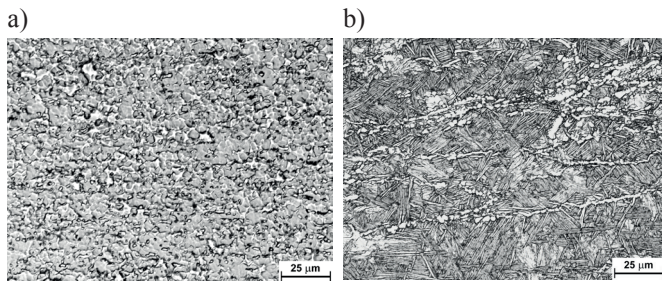


Fig. 4. Initial microstructure (longitudinal cross-section of rods) of examined titanium alloys: a) Ti-6Al-4V, b) Ti-6Al-2Mo-2Cr

Water quenching from the temperature of β range in TMP leads to martensitic transformation $\beta \rightarrow \alpha'$ (α'') in both titanium alloys. Developed microstructures are composed of martensitic α' (α'') phase having similar morphology but various lamella size and orientation <Fig. 5>. Literature data [13-15] indicate that phase composition of quenched titanium alloys depends on type and content of β -stabilizers. Alloys containing Mo has higher propensity to martensitic transformation $\beta \rightarrow \alpha''$. Therefore volume fraction of martensitic α'' phase in microstructure of Ti-6Al-2Mo-2Cr – difficult for evaluation due to its similarity to α' phase – should be higher comparing

with Ti-6Al-4V alloy. Morphology of thicker martensite lamellae <Fig. 5b> is reported to correspond to α'' phase [13].

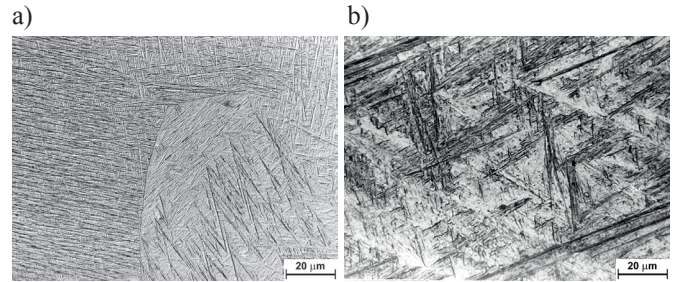


Fig. 5. Microstructure (DIC) of water quenched titanium alloys: a) Ti-6Al-4V, b) Ti-6Al-2Mo-2Cr

Plastic deformation in the temperature range of $\alpha + \beta \rightarrow \beta$ transformation (forging in thermomechanical processing) of β quenched Ti-6Al-4V alloy leads to development of microstructure composed of elongated and deformed α phase grains – denoted as α_D - in the matrix of transformed β phase containing small fraction of spheroidal α_S phase precipitations <Fig. 6a, Tab 2>.

Microstructure of Ti-6Al-2Mo-2Cr alloy after TMP also contains elongated and deformed α_D grains in the matrix of transformed β phase <Fig. 6b>. Precipitations of α phase were observed on grain boundaries of β_{prim} phase. The colonies of parallel α and β lamellae identified in initial microstructure <Fig. 4b> were not found after TMP <Fig. 6b>. Grain growth of β_{prim} phase was detected <Tab 2> caused by dynamic recrystallization during plastic deformation.

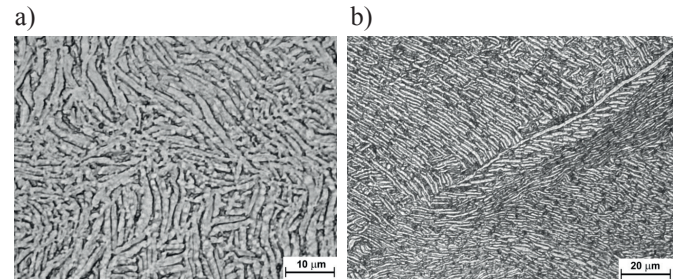


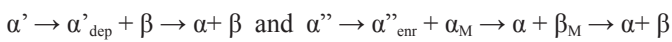
Fig. 6. Microstructure of Ti-6Al-4V (a) and Ti-6Al-2Mo-2Cr (b) alloys after β quenching and plastic deformation in the $\alpha + \beta \rightarrow \beta$ range (longitudinal cross-section)

TABLE 2

Stereological parameters of Ti-6Al-4V and Ti-6Al-2Mo-2Cr alloys before and after TMP

Ti-6Al-2Mo-2Cr									
Alloy condition	Volume fraction of α , α_D and α_S phases %			Length a and width b of rectangular circumscribed on grain section of α and α_D phases, μm				Elongation factor for α and α_p grains	
	V_α	$V_{\alpha D}$	$V_{\alpha S}$	a_α	b_α	$a_{\alpha D}$	$b_{\alpha p}$	f_α	$f_{\alpha D}$
Initial state	82	-	-	4.1	5.3	-	-	0.77	-
After TMP	76	62	14	-	-	1.9	14.3	-	7.5
Ti-6Al-4V									
Alloy condition	Length a and width b of rectangular circumscribed on grain section of β_{prim} phase, μm		Elongation factor for β_{prim} phase $f_{\beta prim}$	Size of colonies of α/β lamellae R , μm	Thickness of α lamellae / α_D grains g , μm				
	$a_{\beta prim}$	$b_{\beta prim}$							
Initial state	137	42	3.1	12	1.3				
After TMP	276	111	2.5	-	1.2				

Metallographic examinations confirmed that during plastic deformation in the $\alpha+\beta\rightarrow\beta$ range following microstructural processes occur: deformation and elongation of α grains, recrystallization of β_{prim} phase and precipitation of spheroidal α_s <Fig. 6>. It is worth to notice that thickness of elongated α_D grains in both alloys do not exceed 2 μm <Tab 2>. Based on results of own studies [16] it was established that α_D grains in microstructure of Ti-6Al-4V alloy thermomechanically processed without preliminary quenching have significantly lower elongation factor ($f_{\alpha D} \sim 2$) and size. The presence of α' (α'') phase before deformation seems to favour elongation of α_D grains and restrict their transverse growth. Even if alloy is quenched before deformation, decomposition of α' (α'') phase during heating up to the deformation temperature should be considered. It is generally accepted [3,4,17] that metastable martensitic α' and α'' phases decompose into mixture of $\alpha+\beta$ phases as follow:



where: α'_{dep} – α' phase depleted in β -stabilizers, α''_{enr} – α'' phase enriched in β -stabilizers, α_M and β_M – metastable phases.

Effectivity of decomposition process in quenched titanium alloy depends on temperature and time [18,19]. Hence additional investigation was performed and water quenched Ti-6Al-4V alloy <Fig. 7a> was heated up to deformation (forging) temperature (900°C). Such annealing for 0.5 h (similar to used for heating up to deformation temperature in TMP) caused increase of lamellae thickness <Fig. 7b>. It can be assumed that during forging in TMP deformed α grains have morphology similar to martensite lamellae. Coagulation of α lamellae is observed after 1 h annealing <Fig. 7c>. Therefore hot deformation with low strain rates should favour growth of α lamellae. Open die forging used in TMP is characterized by rather high strain rate ($\sim 10^2 \text{ s}^{-1}$) then obtained microstructures are composed of highly elongated and deformed fine lamella-like α_D grains <Fig. 6>.

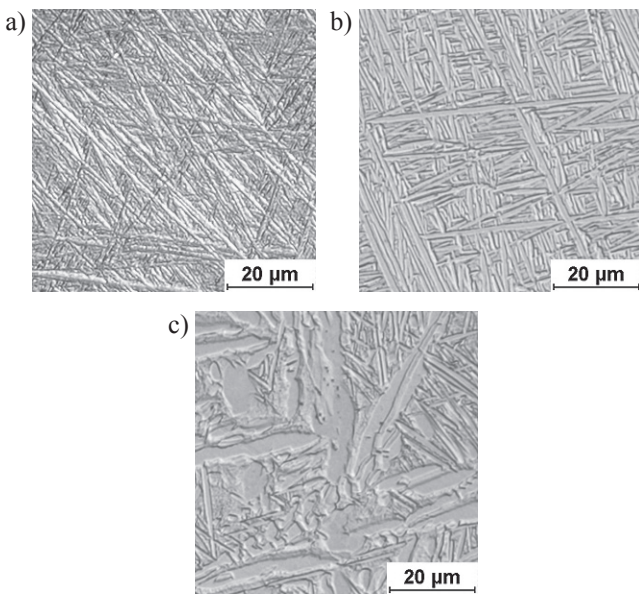


Fig. 7. Microstructure of Ti-6Al-4V alloy (DIC) after water quenching from the temperature of β range (a) and following annealing at 900°C for 0,5 h (b) and 1 h (c)

TEM observations of Ti-6Al-4V alloy after water quenching and following open die forging in $\alpha+\beta\rightarrow\beta$ range revealed high dislocation density in α_D grains <Fig. 8a,b>. It was also found that

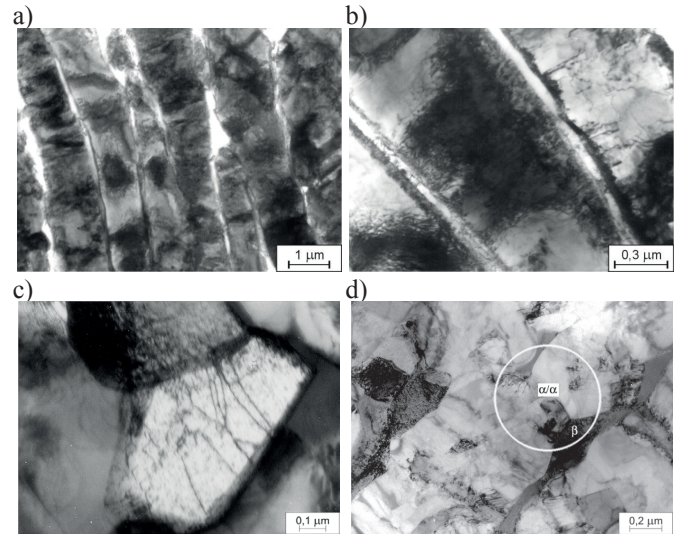


Fig. 8. Microstructure (TEM) of Ti-6Al-4V alloy after TMP - elongated α_D grains with high dislocation density (a, b), fragmentation of α_D grains by high-angle boundary formation (c) and „grooving” of α/β interface (d)

Fragmentation of α_D grains started – mainly by dynamic or metadynamic recrystallization process leading to formation of high-angle boundaries inside deformed areas <Fig. 8c>. Results of TEM investigations indicated possibility of fragmentation by “grooving” of α/β interface <Fig. 8d>. Classical model of “grooving” - proposed by W.W. Mullins [20,21] – is based on grain growth of one phase (β) into interface (α/β) causing grain boundary formation (α/α) in adjacent grain of the second phase (α) <Fig. 9>.

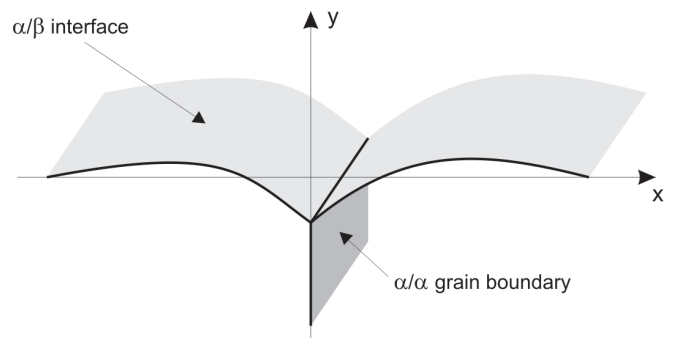
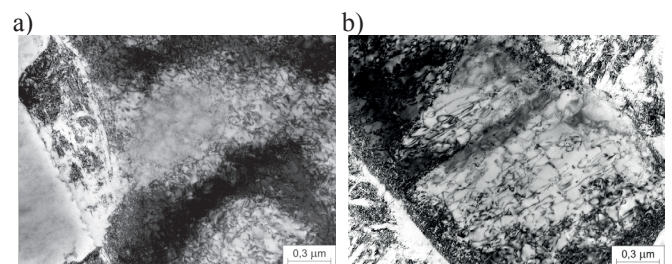


Fig. 9. Scheme of β phase grain growth by „grooving” of α/β interphase [6,20,21]



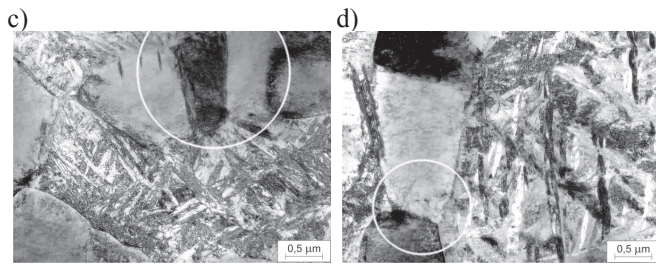


Fig. 10. Microstructure (TEM) of Ti-6Al-2Mo-2Cr alloy after TMP – dislocation substructures in α_D grains (a, b), fragmentation of α_D grains and α phase precipitations in transformed β phase (c, d)

Microscopic examinations using TEM methods of Ti-6Al-2Mo-2Cr alloy revealed high dislocation density in α_D grains <Fig. 10a,b> - similarly to Ti-6Al-4V alloy <Fig. 8a,b>. Also effects of fragmentation process were observed – caused by formation of high-angle boundaries inside deformed areas <Fig. 10c>, often combined with “grooving” phenomena <Fig. 10d>. The main difference between thermomechanically processed Ti-6Al-4V and Ti-6Al-2Mo-2Cr alloys is in morphology of transformed β phase. In Ti-6Al-2Mo-2Cr alloy it contains acicular precipitations of α phase <Fig. 10c,d>. Differences of β transformed phase morphology, formed in the same conditions, results from various β -stabilizers in both titanium alloys.

Obtained results indicate high ability of α_D grains formed from α' (α'') lamellae for transformation during hot plastic deformation. Further investigations including tensile tests at elevated temperature of thermomechanically processed alloys (presented elsewhere [5,12,16]) confirmed their high superplasticity reported by H. Inagaki [10,11]. It was also found that elongated α_D grains are fragmented and spheroidized statically – during heat treatment (recrystallization annealing) or dynamically – during hot deformation (tensile test) <Fig. 11>.

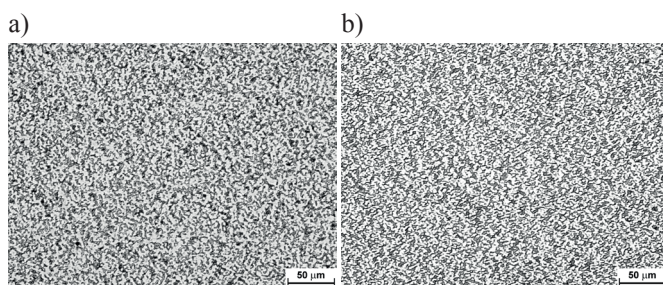


Fig. 11. Microstructure of Ti-6Al-4V (a) and Ti-6Al-2Mo-2Cr (b) alloys after TMP (β quenching and open die forging at 900°C) and tensile test at 850°C at the strain rate of 10^{-2} s^{-1} (longitudinal cross-section) [16]

4. Summary

Plastic deformation in the temperature range of $\alpha+\beta \rightarrow \beta$ transformation of β quenched Ti-6Al-4V and Ti-6Al-2Mo-2Cr titanium alloys leads to development of microstructure composed of elongated and deformed α phase lamellae-like grains.

Developed microstructure is similar in both thermomechanically processed titanium alloys despite their

various initial microstructure – equiaxed in Ti-6Al-4V and lamellar in Ti-6Al-2Mo-2Cr alloy. Preliminary β quenching in proposed TMP minimizes the influence of initial microstructure morphology.

Morphology of α phase grains on the initial stage of plastic deformation at 900°C slightly differ from martensite α' (α'') lamellae.

Elongated α phase grains deformed during plastic deformation in the $\alpha+\beta$ range have dislocation substructure enabling further transformation of their morphology.

Principal mechanism of fragmentation and spheroidization of elongated α grains is formation of high-angle boundaries caused by recrystallization process.

REFERENCES

- [1] M. Motyka, Thermomechanical processing for development of phase constituents morphology and plasticity of two-phase titanium alloys, Rzeszow 2015 (in Polish).
- [2] K. Kubiak, Technological plasticity of hot deformed two-phase titanium alloys, Rzeszow 2004 (in Polish).
- [3] M. Peters, G. Lütjering, G. Ziegler, Z. Metallkd. **74**, 274 (1983).
- [4] A. Bylica, J. Sieniawski, Titanium and its alloys, Warsaw 1985 (in Polish)
- [5] M. Motyka, K. Kubiak, J. Sieniawski, W. Ziąja, Phase transformation and characterization of $\alpha+\beta$ titanium alloys, in: S. H a s h m i (Ed.): Comprehensive Materials Processing, Elsevier (2014).
- [6] S.L. Semiatin, D.U. Furrer, Modeling of microstructure evolution during the thermomechanical processing of titanium alloys, in: ASM Handbook – Vol. 22. Fundamentals of Modeling for Metals Processing. ASM International (2009).
- [7] S.L. Semiatin, V. Seetharaman, I. Weiss, JOM **49**, **6**, 33 (1997).
- [8] T. Seshacharyulu, S.C. Medeiros, J.T. Morgan, J.C. Malas, W.G. Frazier, Y.V.R.K. Prasad, Scripta Mater. **41**, 3 ,283 (1999).
- [9] I. Weiss, F.H. Froes, D. Eylon, G.E. Welsch, Metall. Trans. **17A**, 1935 (1986).
- [10] H. Inagaki, Z. Metallkd. **86**, 643 (1995).
- [11] H. Inagaki, Z. Metallkd. **87**, 179 (1996).
- [12] M. Motyka, Mat. Sci. Eng. **A599**, 57 (2014).
- [13] J.I. Qazi, O.N. Senkov, J. Rahim., F.H. Froes, Mat. Sci. Eng. **A359**, 137 (2003).
- [14] C. Lin, G. Yin, Y. Zhao, P. Ge, Z. Liu, Mater. Chem. Phys. **125**, 411 (2011).
- [15] O.M. Ivasishin, A.I. Ustinov, V.S. Skorodzievskii, M.S. Kosenko, Scripta Mater. **37**, 6 ,883 (1997).
- [16] M. Motyka, J. Sieniawski, Arch. Mat. Sci. Eng. **41**, 2, 95 (2010).
- [17] L. Zeng, T.R. Bieler, Mat. Sci. Eng. **A392**, 403 (2005).
- [18] F.X. GilMur, D. Rodríguez, J.A. Planell, J. Alloy Comp. **234**, 287 (1996).
- [19] J.I. Qazi, O.N. Senkov, J. Rahim, F.H. Froes, Mat. Sci. Eng. **A359**, 137 (2003).
- [20] W.W. Mullins, J. Appl. Phys. **28**, 333 (1957).
- [21] W.W. Mullins, Transactions TMS-AIME **218**, 354 (1960).

