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3 **Using heat treatments, high-pressure torsion and post-deformation annealing**
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5 **to optimize the properties of Ti-6Al-4V alloys**
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17 **Abstract**
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20 Experiments were conducted to investigate the processing parameters that may be used to
21 optimize the properties of Ti-6Al-4V alloys. The alloy was initially subjected to two different
22 heat treatments leading to the formation of martensitic α' and lamellar $\alpha+\beta$ microstructures and
23 then both materials were processed by high-pressure torsion (HPT) for 10 turns at room
24 temperature. This gave significant grain refinement to the nanometer range in both conditions
25 and the occurrence of an allotropic *hcp* to *fcc* phase transformation in the martensitic alloy.
26 These nanostructured alloys were subjected to post-deformation annealing (PDA) at
27 temperatures in the range of 473 to 1023 K. The results show the hardness increases slightly to
28 773 K due to $\alpha'+fcc \rightarrow \alpha+\beta+fcc$ and $\alpha \rightarrow \alpha+\beta$ phase transformations in the martensitic α' and
29 lamellar $\alpha+\beta$ alloys and then decreases up to 1023 K due to recrystallization and grain growth.
30 An optimum property of a very high yield strength (~1120 MPa) and ultimate tensile strength
31 (~1200 MPa), together with excellent ductility (elongation to failure of ~26%), was achieved in
32 the Ti-6Al-4V martensitic alloy processed by a combination of HPT followed by PDA at 873 K
33 for 60 min.
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48 *Keywords:* High-pressure torsion; Nanostructured materials; Phase transformations; Post-
49 deformation annealing; Ti-6Al-4V alloy.
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1. Introduction

1 Ti-6Al-4V is the titanium alloy used most frequently in commercial and industrial
2 applications. This alloy possesses low density, high strength, good toughness, a general
3 corrosion resistance, excellent bio-compatibility and very good high temperature properties and
4 formability. Therefore, it is used widely for many applications in aerospace and chemical
5 engineering, for power generation and as an implant material in medicine [1-4]. In equilibrium,
6 the alloy consists mainly of an α -phase (*hcp*) with some β -phase (*bcc*) at room temperature.
7 The existence of the α/β transformation means that it is possible to achieve a variety of
8 microstructures and property combinations in the alloy through thermo-mechanical processing
9 thereby permitting the development of properties for specific applications [5].
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23 Depending upon the cooling rate and the prior heat treatment, the microstructure of the
24 alloy may be divided into several types. For very slow cooling rates from high within the $\alpha+\beta$
25 region or above the β -transus temperature (1263 ± 20 K), the β -phase primarily transforms into
26 a globular type of α . Increasing the cooling rate accelerates the α nucleation rate in the β grain
27 boundaries thereby enhancing the formation and growth of α platelets into prior β grains. The
28 β -phase fully or partly transforms into a martensitic type during high cooling rates and this
29 martensite exists in two different forms, α' (*hcp*) and α'' (orthorhombic) [6-8]. The type and
30 amount of α' and α'' formed on quenching depends upon the chemical composition (vanadium
31 enrichment) of the β phase that exists at the temperature prior to quenching [9]. Thus, by
32 controlling the phase transformations occurring during thermal processing, particularly during
33 cooling from elevated temperatures, an optimal mechanical performance may be achieved in
34 the $\alpha+\beta$ Ti alloys.
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52 It is now well established that grain refining is a very effective procedure for improving the
53 mechanical properties of materials. Furthermore, processing through the application of severe
54 plastic deformation (SPD) provides an opportunity for reducing the grain size to the
55 submicrometer or even the nanometer level [10,11]. In practice, high-pressure torsion (HPT) is
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1 an excellent processing method because it produces materials with exceptionally small grain
2 sizes and with a large fraction of grain boundaries having high angles of misorientation [12-
3 14]. In HPT a disk-shaped specimen is deformed by simple shear between two anvils where it
4 is constrained under a high pressure and subjected to concurrent torsional straining [15].
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6 Generally, nanostructured metals and alloys processed by HPT exhibit high strength but their
7 ductility is limited because they have both a low rate of strain hardening and a low strain rate
8 sensitivity [16-18]. Accordingly, post-deformation annealing (PDA) is often an important tool
9 for improving the ductility both for titanium [19] and for other materials [20,21]. Nevertheless,
10 annealing at excessively high temperatures may lead to an acceleration in recovery and a
11 reduction in hardness so that it is important to select an appropriate annealing condition to
12 produce both good ductility and high strength [22].
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25 Several reports are now available describing the processing of the Ti-6Al-4V alloy by HPT
26 [23-29] but no systematic investigations are available describing the effect of the initial
27 microstructure on the structural evolution and on the mechanical behavior of the alloy after
28 PDA. Furthermore, there has been no attempt to date to optimize the properties of Ti-6Al-4V
29 alloys using selected combinations of HPT and PDA. Accordingly, the present investigation
30 was initiated to address this deficiency by subjecting a Ti-6Al-4V alloy to two different heat
31 treatments in order to produce significantly different initial microstructures and then evaluating
32 the significance of these initial conditions on the subsequent microstructural evolution and the
33 mechanical properties attained within the alloy after HPT and PDA.
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47 **2. Experimental material and procedures**

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49 The experiments were conducted using a Ti-6Al-4V alloy where the composition is given
50 in wt%. Prior to processing by HPT, the as-received material was divided into two separate
51 batches and these batches were treated using two different heat treatments. The first batch was
52 subjected to a solution annealing at 1273 K for 30 min followed by water quenching to obtain a
53 martensitic microstructure. The second batch was solution annealed at 1223 K for 45 min
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1 followed by air cooling to room temperature and then a stress relief anneal at 873 K for 3 h
2 followed by furnace cooling to obtain a lamellar ($\alpha+\beta$) microstructure. Henceforth, the
3 materials processed by these two procedures are denoted as α' (martensitic) and $\alpha+\beta$ (lamellar),
4 respectively. Following the initial heat treatments, disks with thicknesses of ~ 0.8 mm and
5 diameters of 10 mm were processed by HPT at room temperature under an applied pressure of
6 $P = 6.0$ GPa using a rotation speed of 1 rpm and rotations through totals of 10 revolutions
7 under quasi-constrained conditions [30]. These disks were then processed by PDA at
8 temperatures from 473 to 1023 K for 60 min.
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10 Each processed disk was polished to a mirror-like quality and hardness measurements
11 were taken using a Vickers microhardness tester with a load of 500 gf and a dwell time of 10 s.
12 The average microhardness values, Hv, were measured at 3 mm from the disk centres and at
13 every point the local value of Hv was obtained from an average of five separate hardness
14 measurements. The phase constituents were determined using X-ray diffraction (XRD)
15 employing Cu K α radiation (wavelength $\lambda = 0.154$ nm) at 45 kV with a tube current of 200
16 mA. The XRD measurements were performed over a 2θ range from 30° to 90° using a scanning
17 step of 0.01° and a scanning speed of 2° min^{-1} . The analysis using XRD was conducted over
18 sample areas with diameters of 3 mm located near the edges of the disks. Microstructural
19 characterizations were carried out using optical microscopy (OM), scanning electron
20 microscopy (SEM) and transmission electron microscopy (TEM). Foils for TEM were prepared
21 after HPT processing using a focused ion beam (FIB) Zeiss Nvision 40 FIB facility at 3 mm
22 from the disk centres in the normal sections of the disks so that the normals of the images lay in
23 the shear direction. The TEM micrographs were obtained using a JEOL JEM-3010 microscope
24 operating under an accelerating voltage of 300 kV.
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26 Two miniature tensile specimens with gauge dimensions of $1.1 \times 1.0 \times 0.6 \text{ mm}^3$ were cut
27 from symmetric off-centre positions near the edges of each disk using electro-discharge
28 machining. The mechanical properties were examined in the martensitic and lamellar alloys
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1 after HPT followed by PDA at 773 to 1023 K for 60 min. Stress-strain curves were recorded
2 using an initial strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$ with a Zwick universal testing machine. Two samples
3 were tested for each condition. The stress-strain curves were plotted for each specimen and the
4 ultimate tensile strengths were derived directly from the curves. The elongations were also
5 estimated by carefully measuring the gauge lengths before and after tensile testing using an
6 optical microscope.
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13 **3. Experimental results**

14 *3.1 Microstructures of the Ti-6Al-4V alloy before and after HPT processing*

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18 The microstructure of the alloy after solution annealing at 1273 K for 30 min followed by
19 water quenching is shown in Fig. 1(a). The coarse prior β grains, with an average size of ~ 500
20 μm , were fully transformed to martensite (α') and the microstructure shows martensitic laths
21 distributed throughout the microstructure having different orientations and with an average lath
22 thickness of $\sim 0.8 \mu\text{m}$. Figure 1(b) shows the microstructure after solution annealing at 1223 K
23 for 45 min followed by air cooling to room temperature and then stress relief annealing at 873 K
24 for 3 h followed by furnace cooling. The microstructure reveals a lamellar $\alpha+\beta$ structure having
25 different orientations. In the lamellar $\alpha+\beta$ the retained β -phase lies between the α platelets so
26 that the resulting microstructure consists of average prior- β grains of $\sim 500 \mu\text{m}$, colony size
27 parallel-oriented α -phase lamellae of $\sim 200 \mu\text{m}$ and an average α lamellae lath width of ~ 1.5
28 μm .
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45 TEM micrographs and selected area electron diffraction (SAED) patterns of the HPT
46 processed α' and $\alpha+\beta$ alloys are shown in Fig. 2(a) and (b), respectively, taken at regions of ~ 3
47 mm from the disk centres. The microstructures of both conditions are highly strained from the
48 HPT processing with complex non-uniform contrasts because of the presence of high densities
49 of lattice defects. These images show that many grains have an irregular shape with sharp
50 corners while many of the grain boundaries are wavy and ill-defined. Some equiaxed grains
51 appear to form from the fragmentation of elongated grains and these and other similar images
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1 suggest that the average sizes of the separate fragments of structures are ~30 and ~40 nm for
2 the processed α' and $\alpha+\beta$ alloys, respectively. The diffraction patterns show numerous spots
3 arranged along circles indicating the presence of crystallites separated by high-angle grain
4 boundaries (HAGBs). The appearance of significant streaking of the diffraction spots denotes
5 the presence of high internal stresses and elastic distortions of the crystal lattice. All of these
6 characteristics are typical of materials prepared using SPD techniques and they are consistent
7 with the presence of a large volume of high-energy non-equilibrium grain boundaries [31-34].
8 The observed diffraction patterns correspond to the α/α' -phase and the *fcc* phase for the
9 processed α' samples but only to the α -phase for the processed $\alpha+\beta$ samples. Thus, in the latter
10 material there is no evidence for the presence of a β -phase and this demonstrates the dissolution
11 of the β -phase during the SPD-processing.
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25 Further observations of the microstructure of the HPT-processed α' alloy by TEM and the
26 relevant SAED pattern are given in Fig. 3(a). The dark-field image in Fig. 3(b) is based on the
27 spot corresponding to the *fcc* phase marked by a circle in the diffraction pattern in Fig. 3(a) and
28 it shows the distribution of this phase within the *hcp*-phase matrix and suggests the occurrence
29 of twinning in *fcc*.
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37 *3.2 Mechanical properties after PDA*

38 Figure 4 shows the measured values of the microhardness for the HPT-processed samples
39 before and after annealing at temperatures from 473 to 1023 K for a period of 60 min. The
40 lower dashed lines in Fig. 4 correspond to the initial hardness values before HPT processing of
41 ~330 and ~290 for the α' and $\alpha+\beta$ alloys, respectively, thereby demonstrating that the hardness
42 of the martensitic alloy is initially significantly higher than the lamellar alloy. Inspection of Fig.
43 4 shows that, with reference to the initial conditions, the hardness near the edges of the α' and
44 $\alpha+\beta$ disks increases after 10 turns to ~390 and ~350, respectively. The results show that the
45 hardness of the nanocrystalline α' and $\alpha+\beta$ alloys increases slightly up to ~410 and ~395,
46 respectively, after annealing at 773 K and then decreases rapidly with increasing annealing
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1 temperatures up to 1023 K. At this latter temperature, the hardness is close to the value for the
2 initial $\alpha+\beta$ alloy.

3 Representative plots of engineering stress against engineering strain are shown in Fig. 5
4 using an initial strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$ for initial conditions for (a) α' and (b) $\alpha+\beta$ samples
5 and after PDA for 60 min at temperatures from 773 to 1023 K. Table 1 provides a
6 comprehensive summary of the mechanical properties data derived from the various samples
7 including the hardness, Hv, the yield stress (YS), the ultimate tensile strength (UTS) and the
8 elongation to failure, $\delta\%$.
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10 Figure 5(a) shows results for the initial martensitic sample with high UTS of ~ 1190 MPa
11 and an elongation to failure of $\sim 20\%$ with a flow curve exhibiting strain hardening. Annealing
12 of the α' HPT-processed sample at 773 K gives a reasonable UTS of ~ 1250 MPa that is a direct
13 consequence of the HPT processing but there is only a very limited elongation of $\sim 1\%$ and
14 without any strain hardening. The elongations to failure of samples subjected to PDA above
15 873 K reveal a significant improvement but with corresponding reductions in the UTS. Thus,
16 PDA at temperatures of 873 and 923 K appear to give reasonable strength coupled with a good
17 ductility. The results show the strength decreases and the elongation to failure increases with
18 increasing annealing temperature up to 973 K but with a reduction in the elongation at 1023 K.
19 From these results it is concluded that PDA at 873 K for 60 min leads to excellent mechanical
20 properties with a UTS of ~ 1200 MPa and an elongation of $\sim 26\%$.
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45 Representative plots of engineering stress against engineering strain are shown in Fig. 4(b)
46 for the $\alpha+\beta$ alloy where the initial lamellar sample shows significant strain hardening with a
47 low UTS of ~ 730 MPa and an elongation to failure of $\sim 23\%$. Nevertheless, it is apparent that
48 HPT through 10 turns followed by PDA leads to a very significant improvement in the
49 mechanical properties with a maximum UTS of ~ 1180 MPa and an elongation of $\sim 15\%$ in the
50 $\alpha+\beta$ alloy after PDA at 873 K for 60 min. Inspection of all curves shows that the strength
51 decreases and the elongation increases with increasing annealing temperature except at the
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highest temperature of 1023 K where, similar to the martensitic alloy, there is a slight decrease in the elongation to failure.

3.3 Microstructure after PDA

Figure 6 shows the X-ray diffraction patterns of (a) the α' and (b) the $\alpha+\beta$ alloys in the initial condition, after HPT through 10 turns and after HPT + PDA at 773, 873 and 973 K at the edge areas of the disks. As anticipated, the microstructure of the martensitic alloy consists only of martensite with a main peak of $(101)_{\alpha/\alpha'}$ (Fig. 6(a)). This is consistent with an earlier report in which a high solution heat treatment temperature (>1173 K) led to a lower vanadium enrichment in the β phase and thus to a transformation into α' instead of α'' upon quenching [35]. However, the microstructure of the lamellar alloy in Fig. 6(b) shows α -phase peaks with a main peak of $(101)_{\alpha/\alpha'}$ and a peak corresponding to the $(110)_{\beta}$ plane at $2\theta \approx 39.6^\circ$ confirming the existence of the β -phase in the microstructure. The volume fraction of β -phase at the latter heat-treated condition was only $\sim 6\%$ calculated using standard procedures [36].

The XRD patterns of the martensitic alloy show clearly the appearance of new peaks after HPT processing (Fig. 6(a)) at $2\theta = 44.5^\circ$ and $2\theta = 64.4^\circ$ marked with open inverted triangles and this corresponds to the (200) and (220) planes of the *fcc* structure. The volume fraction of the *fcc* phase was estimated as $\sim 40\%$ after HPT through 10 turns. Nevertheless, for the $\alpha+\beta$ alloy there was no evidence for *fcc* or β phases in the X-ray diffraction patterns. Therefore, since the initial condition of this alloy consisted of α and β phases, these results confirm the dissolution of the β -phase during HPT which is consistent with earlier results [37].

Comprehensive evaluations of the nanostructured Ti-6Al-4V alloys during PDA showed that the microstructures exhibited no significant change after annealing at 773 K except for a reduction in the peak broadening. Nevertheless, the peak corresponding to the $(110)_{\beta}$ plane at $2\theta \approx 39.6^\circ$ appeared after annealing at temperatures of 873 and 973 K. Close inspection of Fig. 6(a) reveals that the *fcc*-phase is stable after PDA up to 973 K but the intensity of this phase decreases significantly.

1 A set of representative SEM images is presented in Fig. 7 for the α' and $\alpha+\beta$ alloys after
2 PDA at 923, 973 and 1023 K for 60 min. These images show that the microstructures of the
3 samples consist of equiaxed fine grains after PDA and, as marked, there are some grains with
4 lamellar $\alpha+\beta$ microstructures surrounded by α grains. The results demonstrate clearly that the
5 grain size increases with increasing PDA temperature in both alloys and the average grain sizes
6 in the $\alpha+\beta$ alloy are larger than in the α' alloy. Measurements gave average grain sizes in the α'
7 alloy of ~ 0.6 , ~ 0.8 and ~ 1.5 μm after PDA at 923, 973 and 1023 K, respectively. The
8 corresponding values for the $\alpha+\beta$ alloy were ~ 1.0 , ~ 1.2 and ~ 3.0 μm after PDA at the same
9 three temperatures.
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20 **4. Discussion**

21 *4.1. Microstructural evolution during HPT processing*

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23 Using appropriate heat treatments, two different initial microstructures of the Ti-6Al-4V
24 alloy were subjected to HPT for 10 turns and the results show the development of different
25 hardnesses and significant different microstructures after processing. The hardness of the α'
26 alloy is higher ($H_v \approx 390$) and the grain size is smaller (~ 30 nm) than for the $\alpha+\beta$ alloy ($H_v \approx$
27 350 and grain size of ~ 40 nm). The initial martensitic phase with a high level of residual stress
28 has a substructure containing predominately dislocations and stacking faults with a few
29 platelets containing twins due to the shear transformation and a supersaturated chemical
30 composition [38] and this explains the microstructural difference between the two alloys after
31 HPT. In general, an investigation of the initial conditions shows that the volume fractions of
32 boundaries in the α' and $\alpha+\beta$ structures are high but not as high as in the α' alloy. These
33 boundaries act as nucleation sites for grain fragmentation together with subgrain formation
34 during the initial stages of deformation.
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54 It is important to note that the initial crystal structure of the martensitic alloy is *hcp* and,
55 based on the low volume fraction of the β (*bcc*) phase in the $\alpha+\beta$ alloy (only 6%), it is
56 reasonable to assume that the *hcp- α* phase controls the deformation mechanism in both alloys.
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1 Twinning plays an important role in the deformation mechanism of coarse-grained *hcp*-Ti due
2 to the lack of a sufficient number of slip systems and twinning undoubtedly plays a significant
3 role in grain refinement at least in the early stages of deformation [39-42]. In fact, the
4 introduction of extra interfaces within the grains during deformation twinning leads to
5 significant grain refinement and extraordinary increments in hardness during HPT processing at
6 room temperature including during the further straining following the saturation of twinning. It
7 was reported that, after large amounts of plastic deformation, the twins break up into subcells
8 and also the twinning propensity decreases with decreasing grain size in Ti during SPD
9 processing [40]. Therefore, a new deformation mechanism is needed to accommodate this flow.
10 The present results suggest that the *hcp* to *fcc* phase transformation plays an important role in
11 the accommodation of constrained deformation in the highly-defect martensitic alloy. The α'
12 (*hcp*) alloy consists of a high volume fraction of boundaries and the substructure contains
13 predominately dislocations and stacking faults with a few platelets containing twins: therefore,
14 this microstructure leads to significant grain refinement and these defects promote the
15 formation of an *fcc* phase during HPT processing [43].
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34 4.2. Microstructural evolution during PDA

35 The results from these experiments show clearly that the single α' -phase changes to a dual
36 $\alpha'+fcc$ -phase and the dual $\alpha+\beta$ -phase changes to a single α -phase after HPT processing through
37 10 turns. Nevertheless, the results show further that the heavily deformed α' and α transform to
38 equilibrium α and β during PDA so that $\alpha'+fcc \rightarrow \alpha+\beta+fcc$ and $\alpha \rightarrow \alpha+\beta$ phase transformations
39 occur in the former and latter conditions, respectively. Close inspection of the hardness data
40 shows that the hardness increases slightly up to 773 K due to the formation of the new phase
41 and thereafter decreases to 1023 K due to a combination of recrystallization and grain growth.
42 The hardness evolution with annealing temperatures in Fig. 4 suggests that the onset of
43 decomposition occurs at 573 K but the β -phase is detectable after annealing at 873 K according
44 to the XRD results in Fig. 6. The SEM images show clearly some equiaxed grains containing
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1 lamellar $\alpha+\beta$ structure after PDA at 923 K and this confirms the occurrence of recrystallization
2 at this temperature in both alloys. The results also demonstrate that the grains remain very fine
3 even after annealing at 1023 K for 60 min.
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6 It was suggested earlier that the fine precipitation of α and β phases from the fully
7 martensitic structure occurs by a nucleation and growth process controlled by atomic diffusion
8 at the martensite plate boundaries or at internal structures such as twins [44]. The volume
9 fraction of the β -phase in both alloys increases by increasing the annealing temperature, but the
10 present investigation shows there is a very low volume fraction of the β -phase in the HPT-
11 processed Ti-6Al-4V alloy for the two initial conditions after PDA (less than 5%). These
12 results demonstrate that the *fcc*-phase decreases by increasing the annealing temperature in the
13 α' alloy and this phase is stable even after annealing at 973 K. It was reported earlier that the
14 *fcc*-phase, which was detected after a solid solution treatment and subsequent water quenching
15 in a Ti-20Zr-6.5Al-4V alloy, completely decomposed into an α -phase after annealing at 973 K
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32 *4.3. Mechanical properties after PDA*

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35 Despite the success in achieving a highly-refined microstructure in the Ti-6Al-4V alloy, it
36 is reasonable to anticipate that the tensile properties of the nanostructured Ti-6Al-4V specimens
37 processed by HPT are not satisfactory when testing at room temperature. It is readily apparent
38 from Fig. 5(a) that the tensile properties after HPT and PDA at 773 K show a very limited
39 elongation to failure due directly to the HPT processing. Accordingly, an optimization of these
40 properties requires PDA of the nanostructured specimens under appropriate conditions in order
41 to improve the ductility. The present investigation shows that it is possible to achieve a very
42 high yield strength (~1120 MPa) and UTS (~1200 MPa) in addition to good ductility
43 (elongation to failure of ~26%) through a combination of HPT processing of the initial
44 martensitic state and PDA at 873 K for 60 min. Specifically, PDA below 973 K is beneficial
45 because it improves the ductility without significantly decreasing the strength whereas PDA at
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1 973 K and higher leads to an acceleration in recovery and grain growth and thereby leads to a
2 strength reduction.
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4 The results show clearly the effect of the initial microstructure on the mechanical
5 properties of samples after HPT processing followed by PDA. Thus, the initial martensitic
6 microstructure leads to greater grain refinement during HPT processing and the average of
7 grain sizes of samples are smaller after PDA. This additional grain refinement is directly
8 responsible for the improved mechanical properties of the martensitic alloy.
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10 There are also other important difference in the two alloys relating to the existence of the
11 *fcc*-phase in the martensitic alloy after PDA. It can be deduced from these experiments that the
12 *fcc*-phase improves the material strength and this is consistent with results on the Ti-20Zr-
13 6.5Al-4V alloy [45]. Thus, it appears that the easy gliding basal $\langle a \rangle$ slip in the matrix is
14 effectively blocked at the phase interface due to the discontinuity of slip across the interface
15 and this is confirmed by a recent report in which the room-temperature yield strength of
16 polycrystalline Ti was observed to increase from ~381 to ~731 MPa after formation of the *fcc*-
17 phase [46].
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36 All of these results show that the β -phase may affect the measured elongations to failure.
37 The initial lamellar microstructure shows lower elongations to failure by comparison with the
38 martensitic alloy after PDA at 873 to 1023 K and the lamellar alloy after PDA at 973 and 1023
39 K. The XRD results show clearly that the volume fraction of the β -phase in the initial lamellar
40 microstructure is a maximum at ~6%. In addition, the results demonstrate that the volume
41 fraction of this phase is increased by increasing the annealing temperature and this explains the
42 lower elongation to failure of samples after PDA at 1023 K by comparison with PDA at 973 K.
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53 *4.4 Significance of using miniature configurations in the testing of HPT samples*

54 It is important to note that the results obtained in tensile testing are dependent not only
55 upon the properties of the material and the testing conditions, including the strain rate and
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1 temperature, but also upon the specimen size and geometry. For example, experiments have
2 shown that the measured elongations to failure in tensile testing tend to increase with
3 decreasing gauge length [47,48] and this is because the area of necking constitutes a major
4 fraction of the gauge length in samples with very short gauge lengths so that much of the
5 measured elongation in simple tensile testing is related to flow in the necked region. This
6 suggests, therefore, that it may be unreasonable to make a direct comparison between the total
7 elongations achieved in conventional tensile testing with longer gauge lengths with the results
8 obtained in tensile testing using HPT samples with gauge lengths only of the order of ~1 mm.
9 Instead, it appears initially that more realistic data may be obtained by measuring the uniform
10 strain that occurs prior to the formation of the neck.
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23 Despite these apparent difficulties associated specifically with miniature samples, the use
24 of miniature tensile specimens cut from the 10 mm disks that are prevalent in conventional
25 HPT processing is now becoming a standard testing procedure. Therefore, the present
26 experiments provide important data on the total elongations to failure and these results may be
27 compared directly with the data obtained in many other similar investigations. Furthermore, it
28 is important to note that the gauge thicknesses in the HPT tensile samples are very small,
29 typically of the order of ~0.6 mm, and it is found in practice that this leads to premature failure
30 because there are insufficient grains in the cross-sectional areas to maintain deformation to very
31 high elongations. As a result, experiments have shown that the tensile elongations recorded
32 with HPT samples are generally smaller, not larger, than the elongations attained with
33 conventional tensile samples. For example, this was demonstrated in a very recent report
34 comparing the elongations to failure achieved in samples prepared by different processing
35 procedures, including HPT, in various Al-Mg-Sc alloys [49]. It should be noted also that,
36 whereas an increase in the measured total elongation was reported for a Ti-6Al-4V alloy sheet
37 using conventional cross-sections but gauge lengths smaller than 10 mm, there was no
38 significant effect of gauge length on the strength characteristics including both the YS and the
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1 UTS [50]. Based on this information, it is therefore concluded that the present results, with a
2 very high yield strength of ~1120 MPa and an elongation to failure of ~26%, provide a
3 reasonable description of the Ti-6Al-4V alloy under the specific conditions used in these
4 experiments.
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7 **5. Summary and conclusions**

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11 1- Two different heat treatments were used to achieve martensitic α' and lamellar $\alpha+\beta$ initial
12 microstructures in a Ti-6Al-4V alloy and these materials were processed by high-pressure
13 torsion (HPT) through 10 turns at room temperature. The results show that after HPT the
14 hardness of the α' alloy is higher than the $\alpha+\beta$ alloy ($H_v \approx 390$ and ~ 350 , respectively) and
15 the grain size is smaller (~ 30 and ~ 40 nm, respectively). There are phase transformations of
16 $\alpha' \rightarrow \alpha' + fcc$ and $\alpha + \beta \rightarrow \alpha$ during HPT in the martensitic and lamellar alloys, respectively
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25 2- Following HPT, the samples were subjected to post-deformation annealing (PDA) for 60
26 min at temperatures in the range of 473 to 1023 K. The results show there are phase
27 transformations of $\alpha' + fcc \rightarrow \alpha + \beta + fcc$ and $\alpha \rightarrow \alpha + \beta$ due to PDA up to 1023 K in the
28 martensitic and lamellar alloys, respectively.
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35 3- The initial martensitic microstructure leads to greater grain refinement during HPT with
36 average grain sizes that are smaller after PDA. There is an *fcc* phase in the martensitic alloy
37 after PDA and this appears to improve the strength.
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42 4- An optimum very high yield strength (~1120 MPa) and ultimate tensile strength (~1200
43 MPa), combined with significant ductility (elongation to failure of ~26%), was achieved in
44 the alloy processed by a combination of HPT in the martensitic state and PDA at 873 K for
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68 267464-SPDMETALS.
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Table caption

Table 1 Microhardness, YS, UTS and elongation (δ) of α' and $\alpha+\beta$ -Ti-6Al-4V alloys in the initial condition and after PDA at 873-1023 K for 60 min.

Figures captions

Fig. 1 Optical micrographs of the Ti-6Al-4V microstructures after (a) annealing at 1273 K for 30 min followed by water quenching (α' alloy) and (b) annealing at 1023 K for 45 min followed by air quenching and then annealing at 873 K for 3 h followed by furnace quenching ($\alpha+\beta$ alloy).

Fig. 2 TEM images and corresponding SAED patterns of (a) α' and (b) $\alpha+\beta$ alloys after HPT processing through 10 turns.

Fig. 3 (a) High magnification TEM image and corresponding SAED pattern of nanocrystalline α' alloy: the diffraction pattern shows a series of *hcp* and *fcc* phases; (b) dark-field image relating to the diffraction spot (*fcc* phase) surrounded by a white circle in Fig. 3(a).

Fig. 4 Dependence of Vickers microhardness of the HPT-processed samples for the α' and $\alpha+\beta$ alloys on the annealing temperature using annealing times of 60 min: the lower dashed lines denote the Vickers microhardness for the initial α' and $\alpha+\beta$ alloys.

Fig. 5 Stress-strain curves at an initial strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$ after HPT followed by PDA at different temperatures for (a) α' and (b) $\alpha+\beta$ alloys: the stress-strain curves for the α' and $\alpha+\beta$ alloys in the initial conditions are also shown by the dashed curves.

Fig. 6 X-ray patterns of (a) α' and (b) $\alpha+\beta$ alloys before and after HPT processing through 10 turns and after PDA at 773, 873 and 973 K for 60 min.

Fig. 7 SEM images of (a-c, left column) α' and (d-f, right column) $\alpha+\beta$ alloys after PDA at temperatures from 923 to 1023 K for 60 min.

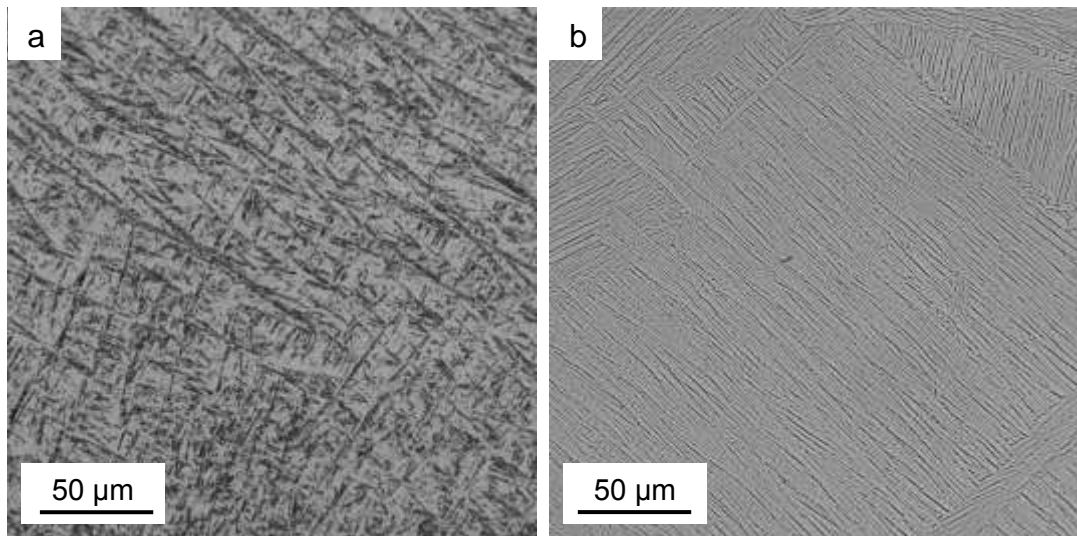


Fig. 1 Optical micrographs of the Ti-6Al-4V microstructures after (a) annealing at 1273 K for 30 min followed by water quenching (α' alloy) and (b) annealing at 1023 K for 45 min followed by air quenching and then annealing at 873 K for 3 h followed by furnace quenching ($\alpha+\beta$ alloy).

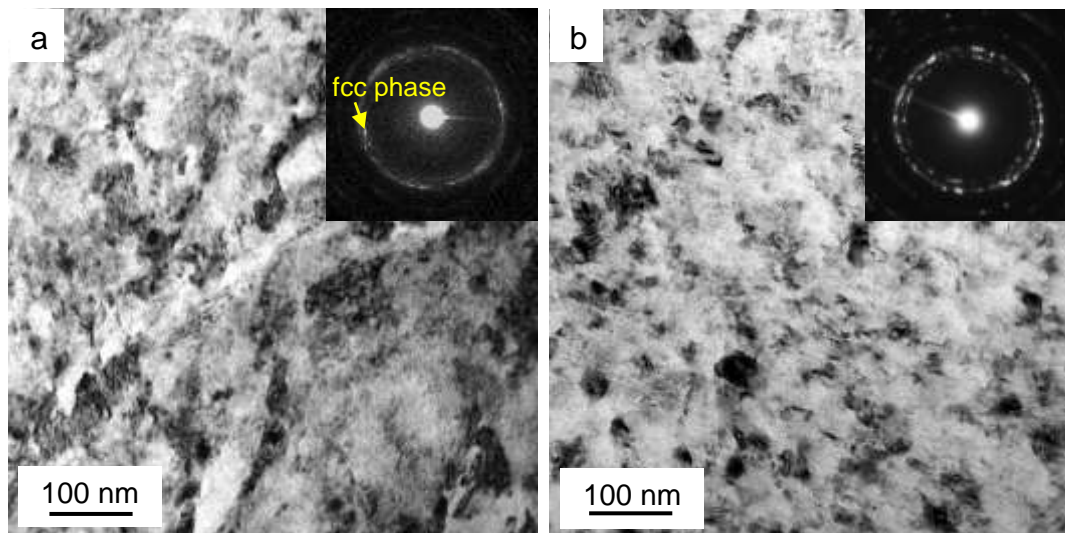


Fig. 2 TEM images and corresponding SAED patterns of (a) α' and (b) $\alpha+\beta$ alloys after HPT processing through 10 turns.

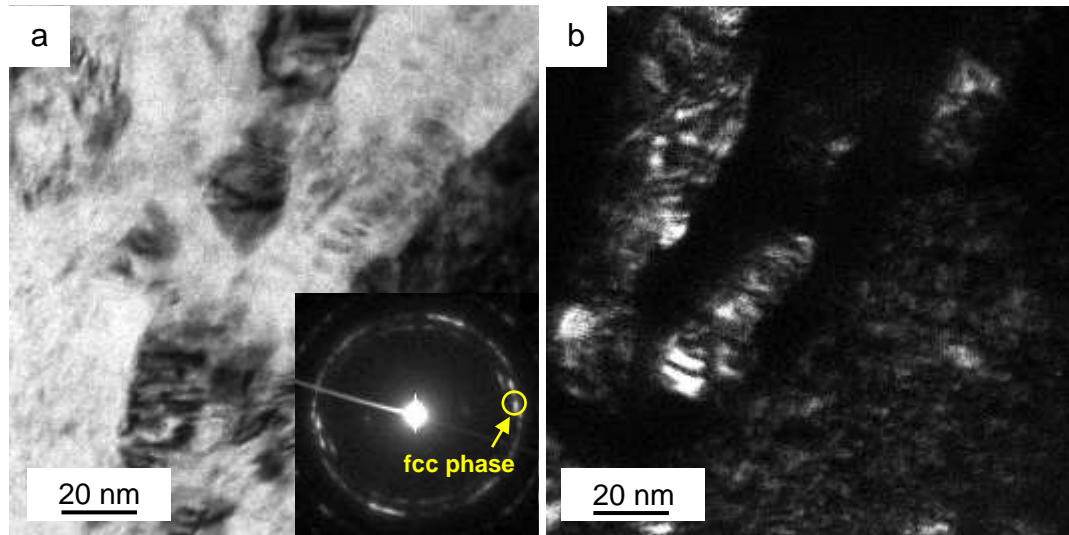


Fig. 3 (a) High magnification TEM image and corresponding SAED pattern of nanocrystalline α' alloy. The diffraction pattern shows a different series of *hcp* and *fcc* phases. (b) The dark-field image relates to the diffraction spot (*fcc* phase) surrounded by a white circle.

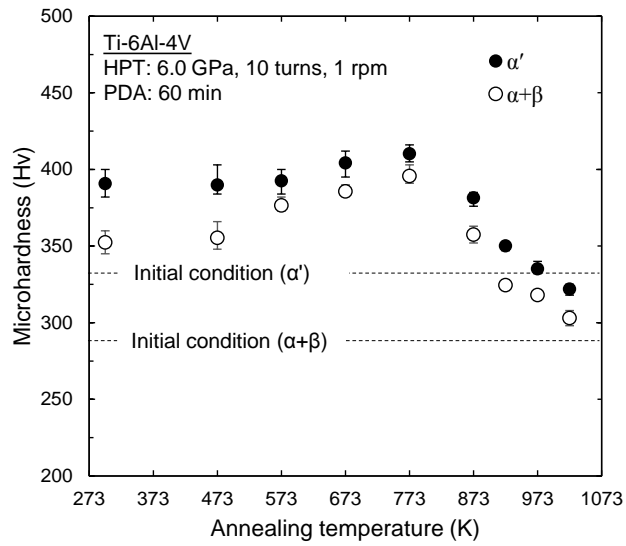


Fig. 4 Dependence of Vickers microhardness of the HPT-processed samples for the α' and $\alpha+\beta$ alloys on the annealing temperature using annealing times of 60 min: the lower dashed lines denote the Vickers microhardness for the initial α' and $\alpha+\beta$ alloys.

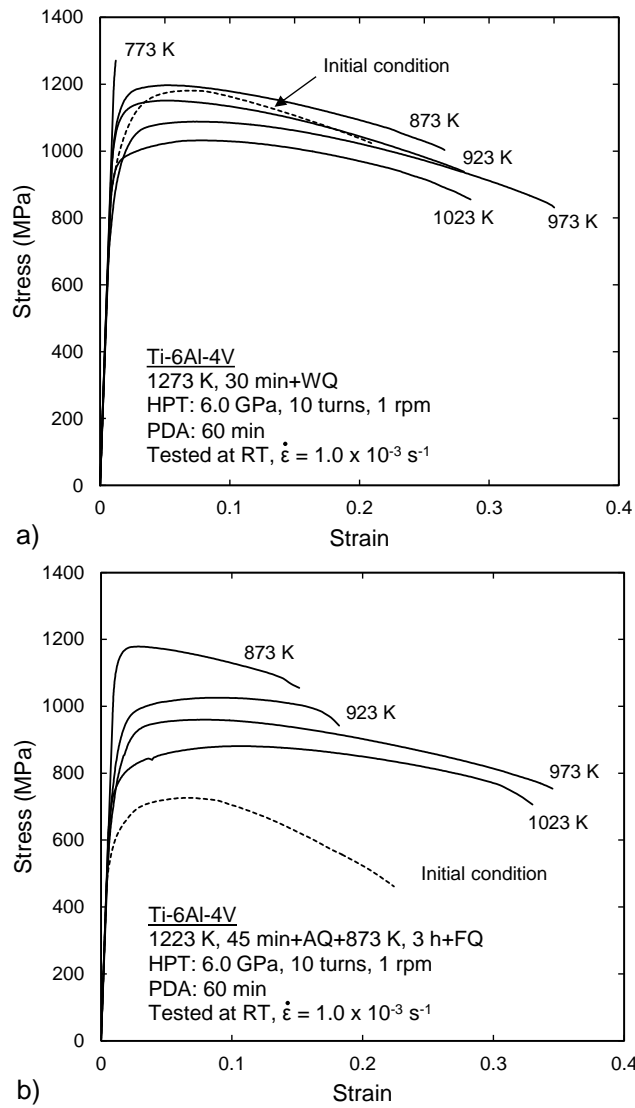


Fig. 5 Stress-strain curves at an initial strain rate of $1.0 \times 10^{-3} \text{ s}^{-1}$ after HPT followed by PDA at different temperatures for (a) α' and (b) $\alpha+\beta$ alloys: the stress-strain curves for the α' and $\alpha+\beta$ alloys in the initial conditions are also shown by the dashed curves.

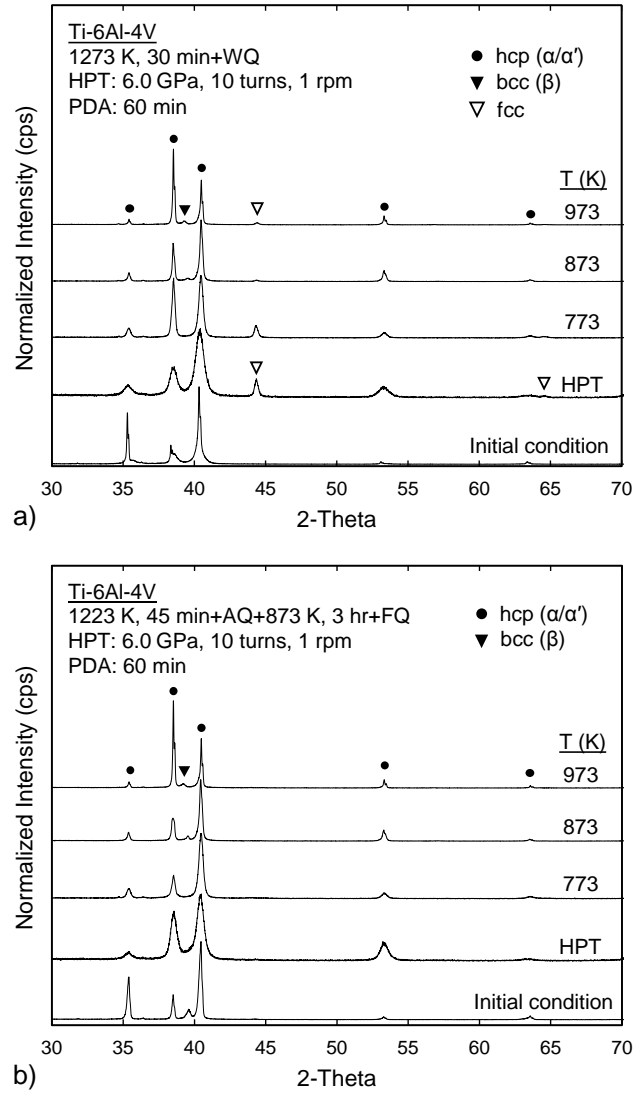


Fig. 6 X-ray patterns of (a) α' and (b) $\alpha+\beta$ alloys before and after HPT processing through 10 turns and after PDA at 773, 873 and 973 K for 60 min.

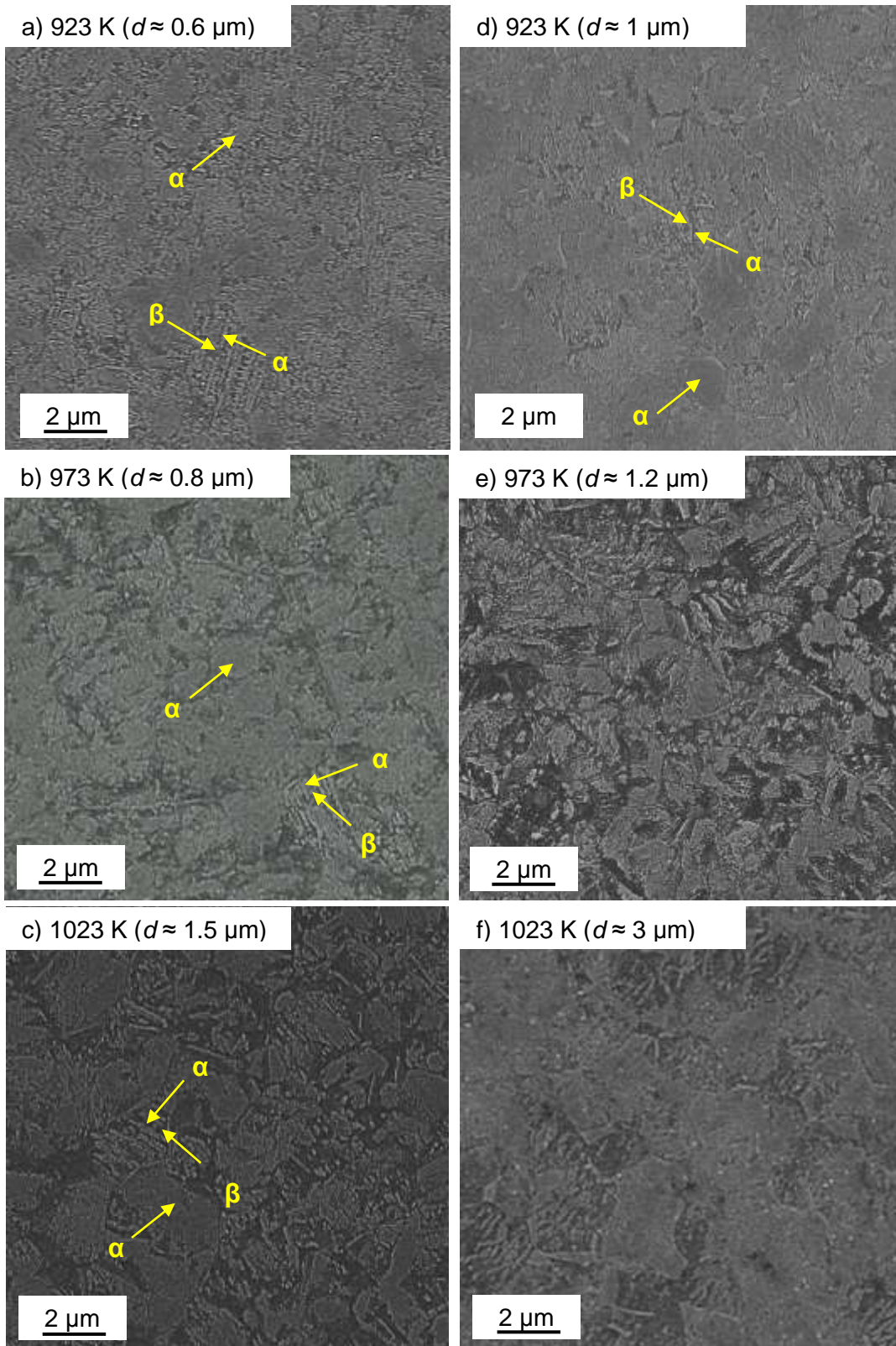


Fig. 7 SEM images of (a-c, left column) α' and (d-f, right column) $\alpha+\beta$ alloys after PDA at temperatures from 923 to 1023 K for 60 min.

Table 1. Microhardness, YS, UTS and elongation (δ) of α' and $\alpha+\beta$ -Ti-6Al-4V alloys in initial condition and after PDA at 873-1023 K for 60 min.

Alloy	Annealing temperature (K)	Hv	YS (MPa)	UTS (MPa)	δ (%)
α'	Initial condition	330 ± 3	930 ± 32	1190 ± 25	20 ± 3
	873	380 ± 4	1120 ± 40	1200 ± 28	26 ± 2
	923	350 ± 2	1050 ± 25	1150 ± 10	28 ± 2
	973	335 ± 3	920 ± 30	1080 ± 22	35 ± 3
	1023	320 ± 3	810 ± 25	1030 ± 15	28 ± 4
$\alpha+\beta$	Initial condition	290 ± 4	250 ± 38	730 ± 30	23 ± 4
	873	360 ± 4	1100 ± 30	1180 ± 20	15 ± 2
	923	325 ± 3	750 ± 25	1025 ± 12	18 ± 5
	973	320 ± 3	740 ± 35	960 ± 24	34 ± 3
	1023	300 ± 4	660 ± 22	880 ± 10	33 ± 2