Strengthening of a Ti–6Al–4V titanium alloy by means of hydrostatic extrusion and other methods

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ABSTRACT

The mechanical properties of Ti–6Al–4V titanium alloy with four different microstructures were studied: a submicrocrystalline structure of grain size about 0.4 μm, produced by multiaxial forging in stages at temperatures decreasing from 700 to 550 °C; a cold-worked submicrocrystalline structure produced by cold rolling after multiaxial forging; a mixed lamellar and submicrocrystalline globular structure produced by hydrostatic extrusion at 600 °C and a microcrystalline structure produced by conventional heat treatment after multiaxial forging at 930 °C. The highest strength, about 1500 MPa, was found for both the cold-worked submicrocrystalline and the hydrostatically extruded materials, but the extruded material had a higher ductility, i.e., higher total elongation and elongation before necking. The submicrocrystalline and the microcrystalline materials exhibited moderate strength of 1300 and 1050 MPa, respectively, with an elongation to failure of about 9%. The results were interpreted in terms of the microstructural features and the strengthening mechanisms resulting from the production processes.

1. Introduction

Titanium alloys are extensively used in the aerospace industry because they possess high specific strength and excellent corrosion resistance. The two-phase Ti–6Al–4V alloy displays an excellent combination of mechanical properties and workability which makes it one of the most widely used titanium alloys. The applications of the Ti–6Al–4V alloy could be broadened by obtaining increased strength whilst maintaining a satisfactory degree of ductility. The conventional method of strengthening the alloy is water quenching and ageing, which achieves a 20–50% increase in tensile strength, up to 1200 MPa, compared to the annealed condition [1].

According to the well-established Hall–Patch relationship, decreasing the grain size is a practical method for improving the strength of a material. Recently, major research efforts have been directed towards obtaining nanocrystalline materials that combine high strength and ductility [2]. Similarly, much effort is directed towards refining the structure of titanium and its alloys. Warm deformation to a strain above $e = 3$ permits to obtain titanium alloys with submicrocrystalline (SMC) structure and grain size of 0.1–1 μm [3]. Titanium alloys with a SMC structure and grain size of 0.4 μm, produced by large strain at 550 °C possess a tensile strength of 1300 MPa, which is about 25% higher than that achieved after strengthening the material by conventional heat treatment [4].

Another standard method used to increase the strength of metallic materials is cold working. However cold working of coarse-grained two-phase titanium alloys is difficult because of the material's relatively low ductility and its high strength at low temperatures. Microstructure refinement increases the ductility of titanium alloys [5] so that they can be subjected to additional cold working. Cold working of a SMC material leads to an additional strength increase of 40–50% [6]. Although the procedure needed for the preliminary formation of the SMC structure followed by cold working is effective, it is a quite laborious method compared to a simple cold working process.

Heavy straining at low temperature can be carried by means of hydrostatic extrusion. Application of high pressure lowers the brittle/ductile transition temperature of metals, prevents formation of cracks and enables very large degrees of plastic deformation to be achieved at a high rate of deformation [7]. Recently the interest in hydrostatic extrusion (HE) as a method for processing 'hard-to-deform materials' has increased considerably as it has been shown that the method produces a submicro- or nanostructure [8–10] and that hydrostatically extruded metals and alloys exhibit significantly enhanced strength [8–12]. The aim of the work reported in this paper was to compare the microstrucutres, strength and ductility of Ti–6Al–4V alloy after four different strengthening treatments so that the optimum processing method can be selected for a specific application.

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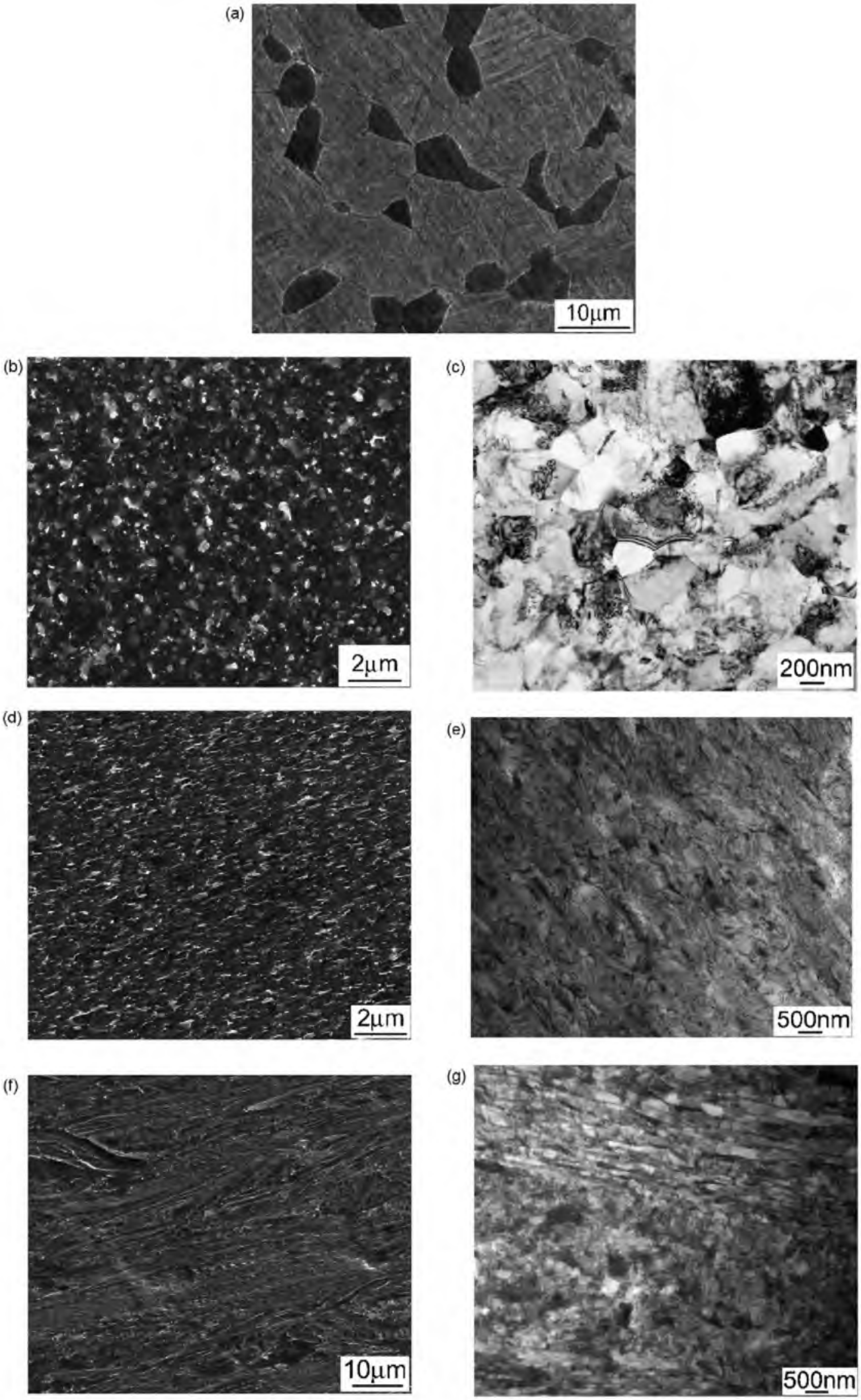

Fig. 1. Microstructure of the Ti–6Al–4V alloy: (a) MC heat strengthened; (b and c) SMC; (d and e) SMC cold rolled to $\lambda = 2.5$; (f and g) hydrostatically extruded; (a, b, d and f)—SEM images, (c, e and g)—TEM images.

The processing methods were:

1. Multiaxial forging, which resulted in a SMC structure with a grain size of 0.4 μm.
2. Conventional solution treatment and ageing as a strengthening procedure after high temperature multiaxial forging, which resulted in a microcrystalline (MC) structure. This was the reference material in this paper.
3. Cold rolling of the SMC alloy.
4. Hydrostatic extrusion.

2. Material and methods employed

The material used was α/β titanium alloy Ti–6Al–4V of composition of 6.3 Al, 4.1 V, 0.18 Fe, 0.182 O, 0.03 Si weight percent, with the balance titanium, with an $\alpha+\beta \rightarrow \beta$ transition temperature of 995 °C. The volume fraction of the β-phase in the alloy at room temperature was 8–10%.

The multiaxial forging (MF) procedure was as follows. Sequential deformation of a sample in three orthogonal directions was used to produce large samples of material with a submicrocrystalline structure. Each stage of the deformation was carried out under isothermal conditions at a strain rate of about $10^{-3}\,\mathrm{s}^{-1}$. The deformation temperature was gradually reduced in order to ensure that the desired microstructure was obtained. The initial deformation temperature was 700 °C and the final temperature was 550 °C, which produced a grain size of approximately 0.4 μm. After the MF procedure the samples were annealed at 650 °C for an hour to reduce residual stresses. The procedure has been described in detail elsewhere [3,4].

The microcrystalline (MC) structure was produced according to the conventional procedure including solution treatment carried out after MF at about 930 °C [1]. Heat treatment involved heating to 945 °C, water quenching and ageing for 3 h at 500 °C [1].

The cold rolling procedure was as follows. Square-sectioned bars with the SMC microstructure were rolled at room temperature in several stages to a total reduction $\lambda = 2.5$ with a tetra-axial rolling mill. The reduction after each step was about 15%. The total reduction was calculated as $\lambda = \ln(F_0/F)$, where F_0 and F are respectively the initial and final cross-sectional area of the bar.

The material for hydrostatic extrusion (HE) was prepared as follows. Initially a lamellar microstructure was obtained by heating the alloy to 1010 °C, in the β-region, followed by air cooling. Since titanium alloys are well known to deform heterogeneously, especially if there is a non-uniform distribution of temperature across the billet, the specimens were encased in low carbon steel containers to avoid chilling during extrusion and to increase the homogeneity of the deformation. Use of a container is desirable but may not be necessary for practical application. However additional experiments should be made to clarify this problem.

The containers with the specimens were heated in air to 600 °C in a furnace, and afterwards were located in the high pressure vessel of a hydrostatic extruder operating at pressures of up to 1.5 GPa. A conical die with an angle of 30° was used and castor oil was employed as the working fluid. There was a delay of 15–20 s before beginning the extrusion process, which was carried out at a ram speed of about 40 mm/s (strain rate $\sim 10^{-1}\,\mathrm{s}^{-1}$).

The extruded samples were air cooled and the titanium alloy cores were obtained by machining away the steel containers. The true strain of the extruded samples was calculated as $e = \ln(F_0/F)$, where F_0 and F are the respective initial and final cross-sectional

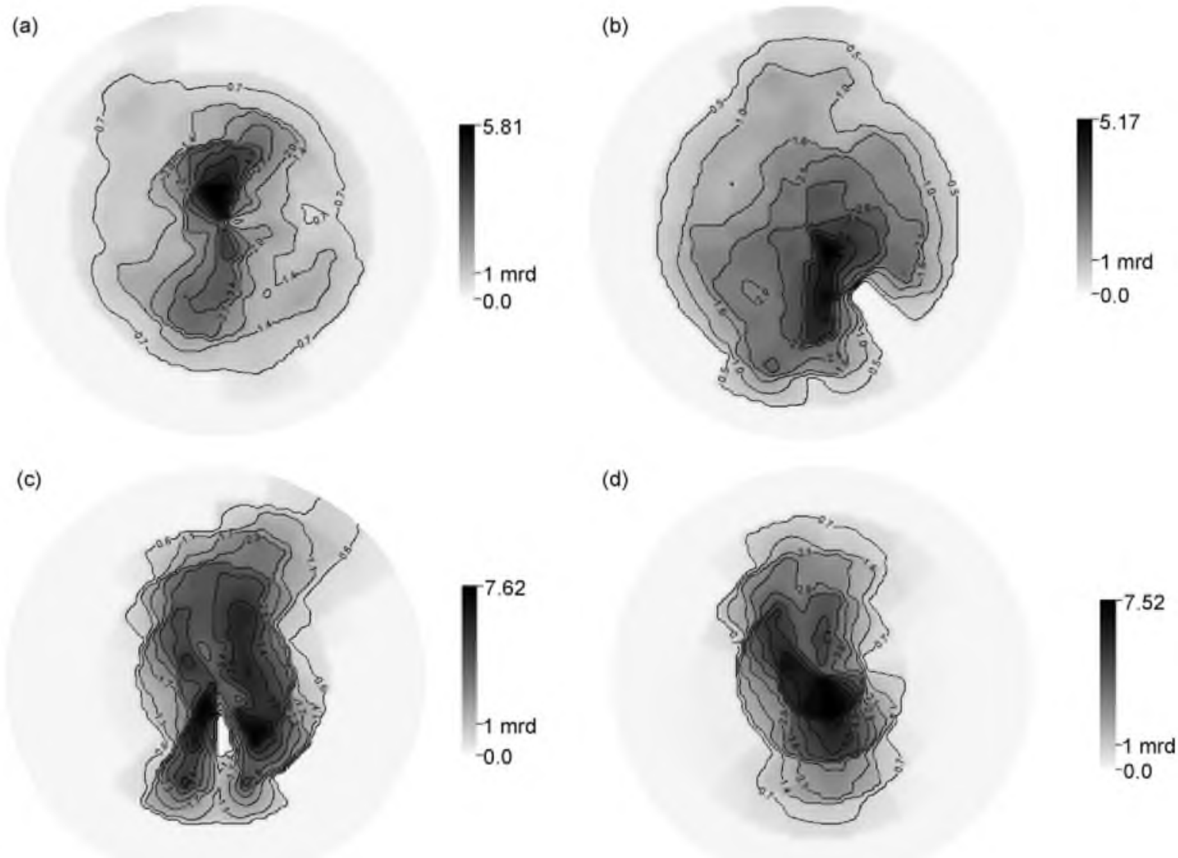

Fig. 2. Pole figures (0 0 0 2) of the α-phase of Ti–6Al–4V alloy in the following conditions: (a) MC; (b) SMC; (c) SMC cold rolling and (d) hydrostatically extruded at 600 °C.

Table 1
Mechanical properties of the various conditions of Ti–6Al–4V alloy.

Production method/microstructure	YS, MPa	UTS, MPa	AR, %	TE, %
Hydrostatic extrusion at 600 °C Mixed lamellar and SMC	1439/1432	1532/1528	20/20	12.5/12
MF + cold rolling SMC	1432/1422	1472/1468	51.5/48	8/8
MF SMC	1184/1174	1300/1300	61/58	7.5/7
MF + heat treatment MC	967/950	1055/1045	33/30	9/9

YS: yield stress; UTS: ultimate tensile strength; AR: area reduction; TE: total elongation.

areas. The hydrostatic extrusion method has been described in detail in [11].

Tensile tests using cylindrical samples of 3 mm diameter and 18 mm gauge length were carried out on a "Schenk" machine at room temperature and a crosshead speed of 1 mm/min. The mechanical properties: yield stress (YS); ultimate tensile stress (UTS); reduction of cross-section (AR) and total elongation (TE) were determined in accordance with the standard of International Organization for Standardization ISO 6892:1998. Each quoted result is based on the average of two tests.

The microstructures were investigated using a JEM-840 scanning electron microscope and a JEM-2000 EX transmission electron microscope. Measuring the thickness of lamellae was carried out by means of the intercept method. X-ray investigations were carried out on an ARL X'TRA (Thermo) diffractometer with Cu-K_α irradiation.

3. Results

The microstructures of the alloy after the four processing methods are shown in Fig. 1.

Conventional strengthening by heat treatment after high temperature MF produced a bi-modal microcrystalline structure composed of primary α-grains with a mean size of 5 μm and colonies of disperse α- and β-lamellae with a thickness of less than 0.2 μm, within the transformed β-grains (Fig. 1a). These samples will be referred to as being in the MC condition.

The microstructure of the alloy after the multiaxial forging treatment consisted of grains of the α- and β-phases with a mean grain size of about 0.4 μm (Fig. 1b and c), which will be referred to as the SMC condition. The non-homogeneous diffraction contrast and the high dislocation density inside some grains in the microstructure of the alloy indicated a high level of residual stresses and elastic distortion of the crystal lattice.

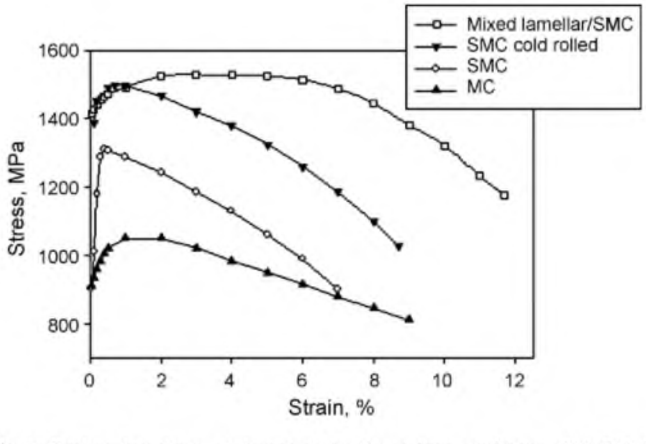

Fig. 3. Engineering stress–strain curves for the various conditions of Ti–6Al–4V alloy.

The microstructure of the SMC alloy after cold rolling at room temperature following MF comprised elongated α- and β-grains with diffuse boundaries and a very high dislocation density. The size of the grains was 100–200 nm in the transverse direction and 300–400 nm in the longitudinal direction (Fig. 1d and e).

TEM observations of the sample hydrostatically extruded at 600 °C reveal a heterogeneous microstructure with lamellae, aligned along the extrusion axis, alternating with areas consisting of very fine globular grains or subgrains about 0.2 μm in diameter (Fig. 1f and g). The volume fractions of the fine grained and lamellar structures were found to be about 35 and 65% correspondingly. The diffraction patterns from the grain/subgrain areas usually showed azimuthal scattering of reflections within 15–20° which indicated a medium degree of misorientation [11]. The structure of the hydroextruded alloy will be referred to as mixed lamellar and SMC.

Analysis of the (0 0 0 2) α-phase pole figures revealed a quite diffuse texture for both the SMC and MC conditions of the alloy (Fig. 2a and b). As the microstructures contained only a small percentage of the β-phase the effect of texture on the mechanical properties can be neglected for both conditions. After cold rolling and hydrostatic extrusion a typical axial texture was formed in the α-phase alloy as the basal $(0\,0\,0\,1)_\alpha$ plane normal direction was aligned with the transverse (radial) directions of the deformed specimens (Fig. 2c and d). Estimates of the phase composition for each conditions of the alloy did not reveal any detectable change in the percentage of each phase.

The mechanical properties of the alloy in the various conditions are shown in Table 1 and Fig. 3. The tensile tests results show that the grain refinement to produce the SMC structure significantly enhanced the strength compared to the MC alloy strengthened by heat treatment. The yield stress and ultimate tensile strength of the SMC alloy were 22–24% higher than that of MC alloy. The total elongation of the SMC and MC alloys are rather similar, but the reduction of cross-sectional area of the SMC alloy is twice that of the MC alloy.

Cold rolling of the SMC alloy caused an additional increase in the ultimate tensile stress by 20–25%, an enhancement of 30–40% compared to the MC heat treated alloy. The ductility of the SMC alloy was hardly changed as a result of the rolling having a total elongation to failure of 8%.

As with the cold rolling, the hydrostatic extrusion resulted in a significant enhancement of the mechanical characteristics compared to the SMC and MC structured alloy samples. Both the strength and total elongation of the extruded alloy are considerably higher than those of the SMC and MC alloys. It should be noted that the extruded material did not exhibit the early necking which occurred with the alloy samples in the other conditions.

4. Discussion

The changes in strength of metallic materials as a function of grain size are described by the Hall–Patch relationship [2,13]. In the case of pure metals (including titanium) refinement of the grain size to below 100 nm or to a submicron range may lead to an increase of strength by a factor of two, or even more [2,14]. In titanium alloys this effect is considerably lower because solid solution strengthening and hardening by precipitation of α-phase in the β-matrix contribute to the strength of the alloy in a manner that does not depend on the grain size. Multiaxial forging of the Ti–6Al–4V alloy leads to additional grain boundary and dislocation strengthening which was manifested by a higher strength than the hardening induced by the presence of thin α-lamellae caused by conventional heat treatment. The difference in strength between the SMC and the MC heat-strengthened conditions of the alloy is 20–25%.

Cold rolling of the specimens with the SMC structure to $\lambda = 2.5$ increased the strength of the material by another 13%. This increase can be attributed, not only to grain/interphase boundary strengthening and dislocation strengthening, but also to strengthening induced by the texturing of the microstructure. Rolling resulted in a typical axial texture (Fig. 2) where the basal plane of the hcp lattice is oriented in the rolling direction. Subsequent tensile deformation forces slip systems with a high Taylor factor to operate, which further enhances the strength of the alloy.

The results of tensile tests revealed several interesting features related to the ductility of the SMC and cold-worked SMC alloys. The tensile elongation at fracture of these two samples is lower than that of the conventionally heat treated MC condition. However, the reduction of area at failure of the SMC and cold rolled SMC alloy is higher than that of the MC alloy by 2 and 3.4 times, respectively (see Table 1 and Fig. 3). Such behaviour is a common feature of nanocrysytalline and SMC materials irrespective of the method by which the structure is produced. Earlier necking and reduced tensile elongation, relative to their microcrystalline counterparts, has been observed in fine-grained materials produced by severe plastic deformation [15], by consolidation of nanosize particles [14] and by electrodeposition [16].

In literature such behaviour is usually attributed to plastic instability originating from the lack of an effective hardening mechanism in heavily worked materials [14,15]. The plastic instability in such materials manifests itself either as shear bands or by "early" necking.

The samples of Ti–6Al–4V alloy extruded to $e = 1.4$ at 600 °C show the highest strength and ductility in spite of the microstructural heterogeneity (Fig. 1d). The ultimate tensile strength was 1530 MPa, which was greater than that exhibited by the alloys subjected to the other procedures reported in this paper (Table 1, Fig. 3). It should be noted that the hydrostatically extruded alloy has an appreciably higher ductility in terms of elongation to failure and elongation before necking than those exhibited by the alloy in the other conditions.

One of the factors that may contribute to the relatively high properties of the extruded alloy is the pronounced axial crystallographic texture (Fig. 2). Since the tensile axis is perpendicular to the c-axis of the hcp lattices in the α-phase, prismatic slip operates quite easily during the tensile test, leading to some increase in ductility [17]. A similar texture was observed in the SMC cold rolled condition, however in this case, a favourable crystallographic texture did not manifest itself, neither through an increase in strength, nor through delocalization of deformation. Therefore it is unlikely that this factor plays a major role in the high strength and ductility of the extruded alloy.

On the other hand, the excellent mechanical properties of the hydrostatically extruded alloy may result from its microstructure. Because of the alignment of the lamellae along the tensile axis the free path for dislocation glide within the α-lamellae of thickness 0.2 μm is expected to be $a/\sin 45^\circ \approx 0.3\ \mu\text{m}$. Consequently a mixture of the SMC grains and thin lamellae should behave like the usual fine-grained material. Moreover, the mechanical behaviour of the hydrostatically extruded sample implies an ability to strain harden without early necking. It has been shown in [18] that uniformly 'mixing' micron-sized grains into an ultrafine-grained matrix imparts some strain hardening capacity to the material. In the hydrostatically extruded alloy the remains of the colony structure may play a similar role. However, for this mechanism to be effective, the dislocations should be able to cross the interface of the α/β boundaries. Initially a mutual orientation of the α- and β-phases satisfies the Burgers orientation relationship ($(0\,0\,0\,1)_\alpha//(1\,1\,0)_\beta$ and $[1\,1\,\bar{2}\,0]_\alpha//[1\,1\,1]_\beta$ [19]), which has been assumed as permitting easy slip transmission across the α/β interfaces [19,20]. The deformation obviously results in deterioration of coherency and an increase in the stress needed for the dislocations to cross the interfaces. However, as long as the lamellar microstructure exists, a partial "transparency" of α/β boundaries for dislocations may remain [20,21]. The strengthening effect of such boundaries may be described, by an analogy with low-angle boundaries, or by a relationship similar to the Hall–Patch equation: $\sigma \approx d^m$, where d is a distance between the sub-boundaries or interfaces and m is between 1/2 and 1 [22], depending on the "transparency" of the boundary. Consequently, parts of the material with the lamellar microstructure simultaneously exhibited increased ductility and contributed, along with the SMC grains, to the high strength.

HE is accompanied by adiabatic heating of the material in the die area by up to 150 °C [8,23,24]. This may lead to dynamic recovery, particularly in the areas with the highest accumulation of local strain. Such a short thermal treatment may have a beneficial effect on ductility, without leading to grain growth. The ductility enhancement mechanisms may be connected with equilibration of the interfaces and grain boundary structures. In particular it may lead to the annihilation of statistically stored dislocations [25] accumulated at grain boundaries and interfaces and partial relaxation of the stresses resulting from the different plastic and elastic properties of the precipitates and the matrix [26,27]. Valiev et al. [28,29] described that process as a transition from grain boundary nonequilibrium of first order to nonequilibrium of second order. The first equilibration process involves annihilation of grain boundary dislocations, whilst the second one grain growth and triple junction migration. In the short time the material is extruded, most likely only the first equilibration stage takes place and therefore ductility is increased without decrease of strength. In Ref. [30] it was shown that a short annealing at 150 °C of nickel after severe plastic deformation increases ductility whilst preserving high strength. It was also shown that grain growth has not taken place, whilst the structure of grain boundaries was equilibrated because of annihilation of grain boundary dislocations. Although the subject needs further studies, it is plausible that in the present case the adiabatic effects may have contributed to the increased transparency of the interfaces to dislocation movement in the similar way as during the above-mentioned experiment with Ni produced by severe plastic deformation.

The results obtained show the possibility of increasing the strength of Ti–6Al–4V alloy by more than 50% compared to the conventionally heat treated condition, whilst, at the same time, maintaining good ductility. The simplest way to attain high strength is HE of the alloy pre-heated to 600 °C. The cold rolled material with the SMC structure also has a very high strength, which is comparable to that obtained by means of hydrostatic extrusion. The choice of the particular method to obtain the desired properties will depend on the intended application of the material.

5. Conclusions

The mechanical properties and microstructure of Ti–6Al–4V alloy processed by four different methods have been compared: a submicrocrystalline structure with a grain size of about 0.4 μm, which was produced by multiaxial deformation in stages at decreasing temperatures from 700 to 550 °C; a cold-worked submicrocrystalline structure, which was produced by cold rolling following the multiaxial deformation; a mixed lamellar/submicrocrystalline structure, which was formed by hydrostatic extrusion at 600 °C and a microcrystalline structure produced by multiaxial forging followed by conventional solution heat treatment and ageing.

The Ti–6Al–4V alloys processed by hydrostatic extrusion and that formed by multiaxial forging and subsequent cold rolling possessed a very high tensile strength of about 1500 MPa. The submicrocrystalline material produced by the multiaxial forging method and the microcrystalline structured material formed by forging and heat treatment show moderate strengths of 1300 and 1050 MPa, respectively, with an elongation to fracture about 9%.

The hydrostatically extruded alloy possessed the highest ductility in terms of elongation before necking and total elongation to fracture.

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