Effect of hydrostatic extrusion at 600–700 °C on the structure and properties of Ti–6Al–4V alloy

S. Zherebtsov , A. Mazur , G. Salishchev , W. Lojkowski

Abstract

The effect of hydrostatic extrusion in the range 600–700 °C on the structure and mechanical properties of Ti–6Al–4V titanium alloy with a lamellar microstructure is reported in the paper. Extrusion at 600 °C resulted in the formation of a heterogeneous microstructure consisting of submicrocrystalline grains of size about 0.2 μm and thick lamellae aligned with the extrusion axis. An increase of the extrusion temperature increased the volume fraction of the globularized structure and the grain size. The hydrostatic extrusion leads to a remarkable increase in the strength of the alloy. The ultimate tensile strength of a sample extruded at 600 °C to a true deformation of $e = 1.4$ was 1530 MPa.

Keywords: Hydrostatic extrusion; Ti–6Al–4V titanium alloy; Structure refinement; Strength; Ductility

1. Introduction

Refinement of microstructure is a well-known method used to improve the mechanical properties of metals and alloys [1,2]. A reduced grain size leads to an increase in strength as predicted by the Hall-Petch relationship [3]. In addition, an increase in hardness, fatigue resistance and ductility are observed for most fine-grained materials when compared to their coarse-grained counterparts [1,2,4].

In bulk samples considerable microstructural refinement to the submicrocrystalline (SMC) grain size between 0.1 and 1 μm, can be attained by severe plastic deformation (SPD) [5,6]. However, production of an SMC structure requires lower deformation temperatures than those commonly used in the conventional fabrication of semi-finished products [5]. SPD of some engineering materials is complicated by several problems associated with their relatively low ductility and high strength at low temperatures.

One of the methods which can be used to avoid mechanical failure when carrying out severe straining is to carry out the deformation under high hydrostatic pressure [7]. High-pressure torsion and equal channel angular extrusion are well-known methods which employ high pressure to produce a SMC structure. However, both methods are severely limited by the size of specimens that can be processed and/or the yield strength of the materials [2].

One method that can be used for processing hard-to-deform materials at low temperatures is hydrostatic extrusion (HE) [8,9]. In this process the billet is forced through a die by means of the pressure of a liquid. As it is possible to attain a large strain (e.g. [8]) this method is suitable for the refinement of a material's microstructure. Indeed, the microstructure of a number of metals and alloys (nickel, copper, steel, aluminium alloys, titanium, etc.) can be refined to the nanocrystalline or submicrocrystalline level by means of HE [8–12].

However, the previous investigations have been focused mainly on single-phase materials. In two phase or multiphase alloys the microstructural refinement may be considerably enhanced by the presence of interphase boundaries in the material. This is considered to be of great interest with respect to the $\alpha + \beta$ titanium alloys. These alloys, such as well-known and widely used Ti–6Al–4V alloy, possess a lamellar microstructure where the interphase boundaries divide the initial grains into relatively thin plates.

In the present work the influence of HE carried out in the temperature range between 600 and 700 °C on the structure and properties of Ti–6Al–4V titanium alloy was studied.

2. Experimental

The material used in this study was an α/β titanium Ti–6Al–4V alloy with a chemical composition (in wt%) of 6.3 Al, 4.1 V, 0.18 Fe, 0.182 O, 0.03 Si with the balance being titanium. The phase transition temperature of the alloy is 995 °C. At room temperature, about 85–90% of the material exists as the α-phase.

Cylindrical specimens of diameter 30 mm and length 60 mm were air cooled from the β-region (1010 °C) to obtain a lamellar microstructure. Titanium alloys are well known to deform heterogeneously [13], especially if there is a non-uniform distribution of temperature across the billet. To avoid chilling during extrusion and to increase the homogeneity of the deformation the specimens were encased in low carbon steel containers of 50 mm diameter and 250 mm length which had a chemical composition (in wt%) of 0.35 C, 0.25 Si, 0.65 Mn, 0.2 Cr, 0.035 S, 0.03 P, 0.2 Cu, 0.02 Ni and balance Fe. The containers with the specimens were heated to the deformation temperature (600, 650 or 750 °C) in a furnace, in air, and the specimens were then located into the working chamber of a hydrostatic extruder (Fig. 1). The delay before beginning the extrusion was 15–20 s.

The extrusion temperatures 600, 650 and 700 °C were chosen in accordance with the extrusion machine capacity, however this temperature was low enough to attain the SMC structure in Ti–6Al–4V alloy [14].

The deformation was carried out in a horizontal hydrostatic extruder operating at pressures of up to 1.5 GPa. Castor oil was used as the working fluid. The strain rate during HE was about 40 mm/s ($\sim 10^{-1}\,s^{-1}$). A conical die with an angle of 30° was used.

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

The microstructure of the extruded samples was examined in the longitudinal and the transverse sections using a JEM-2000 EX transmission electron microscope (TEM) at an accelerating voltage of 200 kV, a LEO-1530 scanning electron microscope (SEM) and optical microscopy.

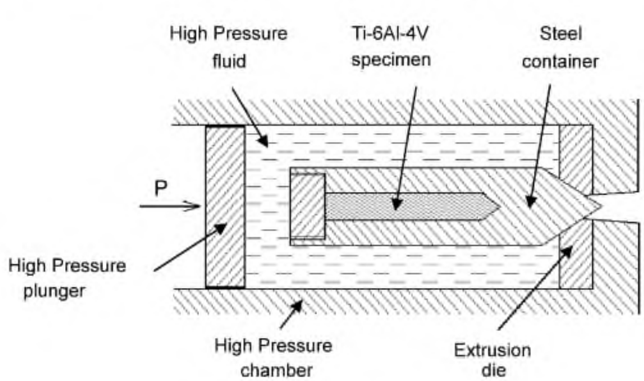

Fig. 1. Scheme of HE set-up.

X-ray diffraction was used to determine the crystallographic texture of samples extruded at 600 and 700 °C. Inverse pole figures for the α-phase were measured from the longitudinal and transverse sections using Cu Kα radiation.

The microhardness of the extruded samples was measured on the longitudinal section using a PMT-3 machine with load of 50 g. The tensile properties of the material extruded at 600 °C were determined by a tensile test at room temperature. Cylindrical samples with a gauge diameter of 3 mm and length of 18 mm were used for the tensile test.

The microstructure, texture and mechanical properties were determined on the central portion of the extruded samples where steady extrusion occurred.

3. Results

The 'as-received' material had a colony structure of lamellar/basketweave α-precipitations in the β-grains of average grain size 150 μm. The boundaries of the initial β-grains were decorated by a 1.5 μm thick layer of α-phase (Fig. 2a). The fine microstructure of the alloy consists of α-lamellae of thickness 0.5 μm and β-phase in the form of very thin (about 50 nm) laths separating the α-lamellae (Fig. 2b).

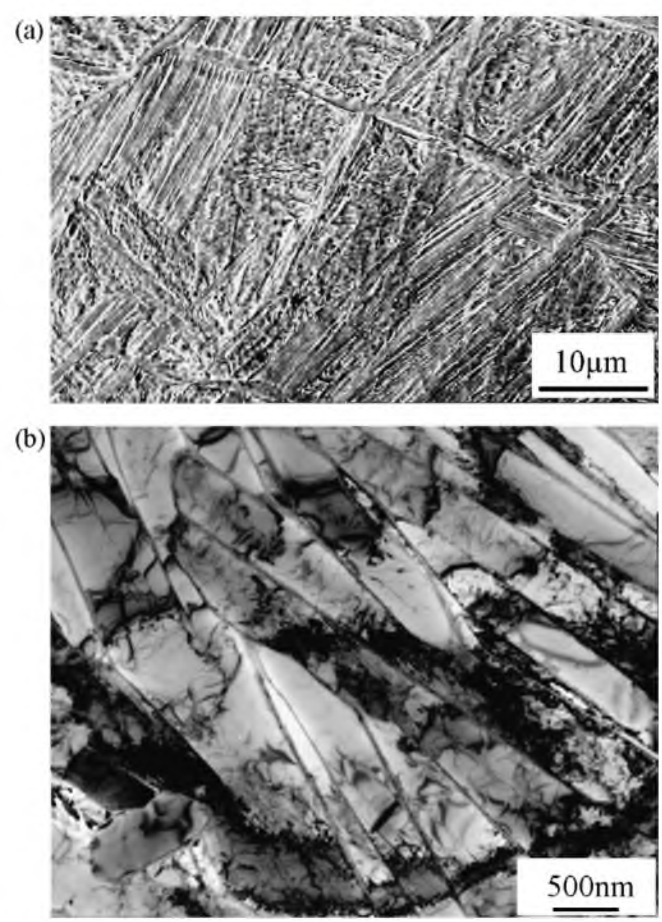

Fig. 2. Initial microstructure of Ti–6Al–4V alloy after air cooling from β-region: optical (a) and TEM (b) images.

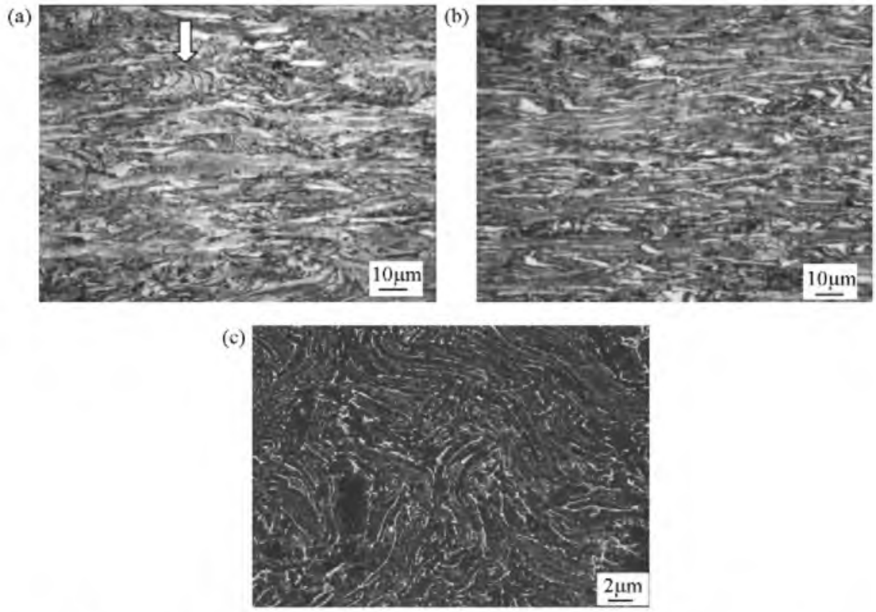

Fig. 3. SEM images of Ti–6Al–4V alloy microstructure after HE at 600 °C (a and c) and 700 °C (b); (a and b) longitudinal section (extrusion axis is horizontal); (c) transversal section.

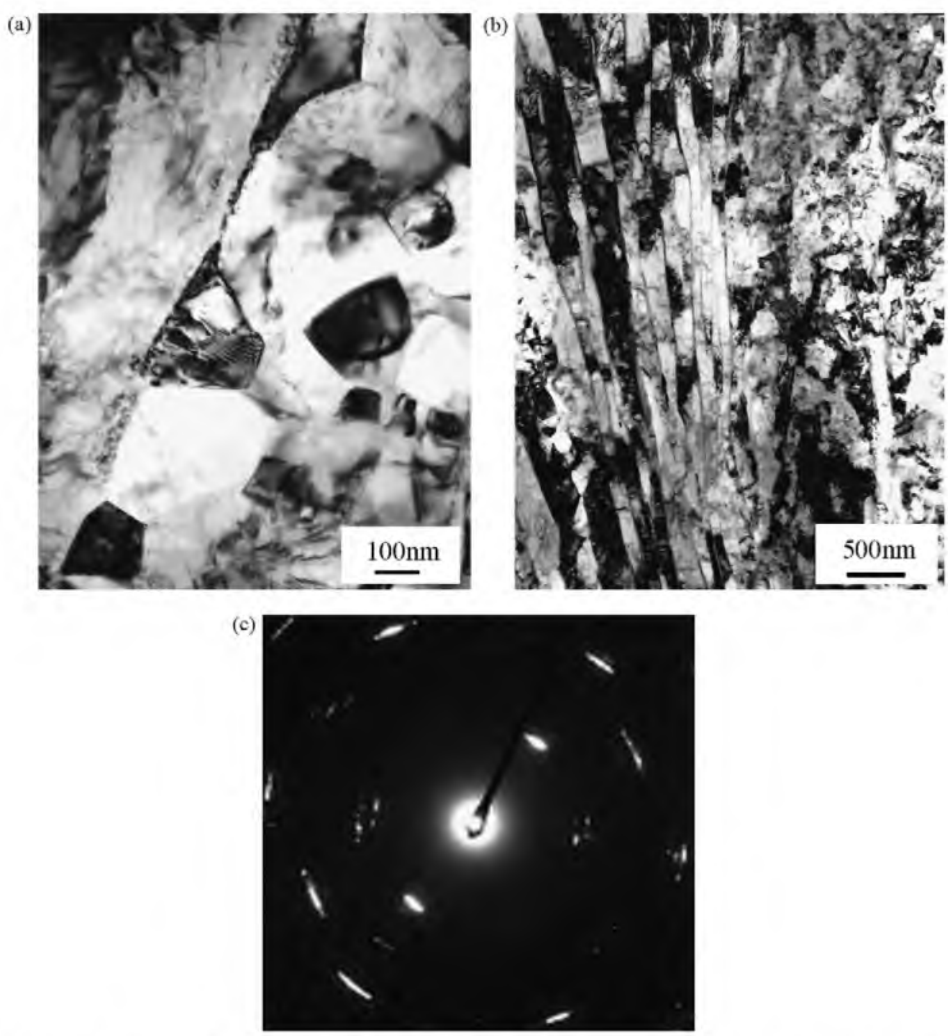

Fig. 4. TEM images of Ti–6Al–4V alloy extruded at 600 °C (a and b); diffraction pattern (c) obtained from the lamellar microstructure (image (b)) from the area of 12.6 μm^2. The axis of HE is vertical.

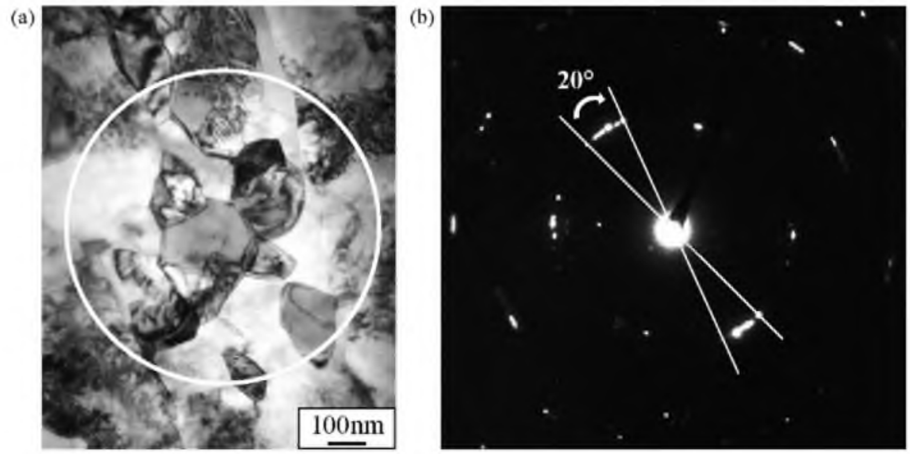

Fig. 5. TEM images of Ti–6Al–4V alloy extruded at 600 °C (a); diffraction pattern (b) obtained from the grain microstructure (image (a)) from the area of 0.5 μm^2.

The samples extruded at temperatures 600, 650 and 700 °C under a pressure of 1 GPa at an extrusion ratio of 4, 4 and 6.2, respectively, or to the true strain of 1.4, 1.4 and 1.8, respectively, had no any visible surface cracks. The macrostructure in the longitudinal sections of all extruded samples was homogeneous and free of pores and cracks.

The microstructure of the alloy extruded at 600 °C was found to be much finer than that of the initial condition (Fig. 3a). The boundaries of the original β-transformed grains could not be detected. The microstructure consisted of fine-grained areas with the remainders of colonies of α-lamellae. Usually the α-lamellae and β-laths were aligned along the extrusion axis, creating a pronounced longitudinal metallographic texture. However, in some places, the lamellae were kinked and some parts of them were oriented across the direction of metal flow as denoted by an arrow in Fig. 3a. The volume fractions of the fine-grained and lamellar structures were found to be about 20% and 60%, correspondingly. Coarse α-particles of size of about 5 μm were also observed in the microstructure and comprised a volume fraction of about 20%.

In the transverse section the extruded alloy also had a lamellar-type microstructure (Fig. 3c). However the lamellae were found to be folded and kinked in different directions forming a specific 'curly' type of microstructure [15]. Again, the boundaries of the original β-transformed grains could not be detected. The absence of the coarse α-particles in a cross-section implies that their appearance in a longitudinal section is associated with the coincidence of the section surface and the planes of some α-laths aligned along the extrusion axis.

TEM examination of the sample extruded at 600 °C revealed a heterogeneous microstructure where lamellae, aligned along the extrusion axis, alternate with areas consisting of very fine (about 0.2 μm in diameter) globular grains or subgrains (Fig. 4). The thickness of α-lamellae was found to be quite variable: in some areas the thickness had decreased from 500 nm in the initial state to about 200 nm, however some α-lamellae with

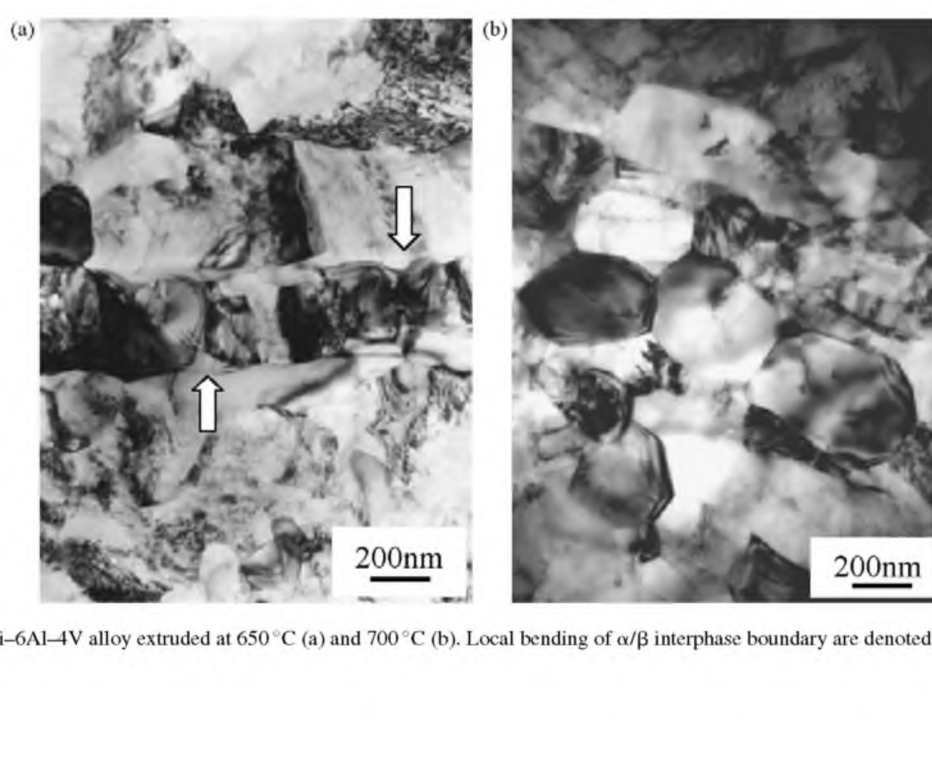

Fig. 6. TEM images of Ti–6Al–4V alloy extruded at 650 °C (a) and 700 °C (b). Local bending of α/β interphase boundary are denoted with arrows. Extrusion axis is horizontal.

Table 1
Mechanical properties of Ti–6Al–4V alloy

Condition	Yield stress (MPa)	Ultimate tensile stress (MPa)	Tensile elongation (%)	Reduction of area (%)
MC heat-strengthened [15]	960	1050	9	32
SMC (grain size 0.4 μm) [15]	1180	1300	7	60
Extruded at 600 °C to $e = 1.4$	1435	1530	12	20

a thickness of few micrometers were also observed. Thin α-lamellae contain diffused dislocation pile-ups, which slightly bend the lamellae in the plane of the foil by less than 1° (Fig. 4b). A selected area diffraction pattern obtained from the lamellar microstructure showed a small azimuthal scattering that also indicates a slight misorientation within the lamellae. However, in some thick lamellae grains with a medium-angle misorientation of 10–15° were revealed. The diffraction patterns from SMC areas usually show azimuthal scattering of reflections within 15–20° (Fig. 5) which also indicates that the misorientation is within the low to medium range. Because of very small grains, there is often more than one grain in the section of the foil (grain size is about 200 nm, while the thickness of the transparent part of the foil is about 300 nm). It was, for this reason, that the misorientation could not be examined systematically by the available equipment.

The microstructure hardly changed when the temperature of HE was increased to 650 and 700 °C, respectively (Fig. 3b).

However, the division of the lamellae was found to be more pronounced at the higher extrusion temperatures than at 600 °C (Fig. 6). Many transverse sub-boundaries were observed within the α-lamellae after extrusion at 650 and 700 °C. Local bending of the α/β interphase boundaries had appeared in the intersections of sub-boundaries and interphase boundaries, as indicated by arrows in Fig. 6a. Chains of grains were observed instead of some lamellae (Fig. 6b). The grain size produced by the extrusion was found to be about 0.25 μm at 650 °C and to 0.35 μm at 700 °C.

Inverse pole figures from transverse and longitudinal sections of the specimens extruded at 600 and 700 °C are shown in Fig. 7. Both specimens have a fibrous texture with the basal $(0\,0\,0\,1)_{\alpha}$ plane normal direction aligned with the transverse (radial) direction of the extruded specimens.

The microhardness of the samples was reduced with an increase of the HE temperature. The microhardness (Hv) was found to be 347, 335 and 323 for the samples extruded at 600, 650 and 700 °C, respectively. To evaluate the mechanical properties a tensile test was carried out on a sample extruded at 600 °C. The properties of the extruded sample and those of two reference samples of the alloy [4] having a SMC grain size around 0.4 μm, and the MC heat-strengthened condition at room temperature are given in Fig. 8 and Table 1. HE resulted in a noticeable enhancement of the mechanical characteristics compared to the SMC and typical MC alloy. 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 does not exhibit early necking unlike the alloy in the SMC and MC conditions.

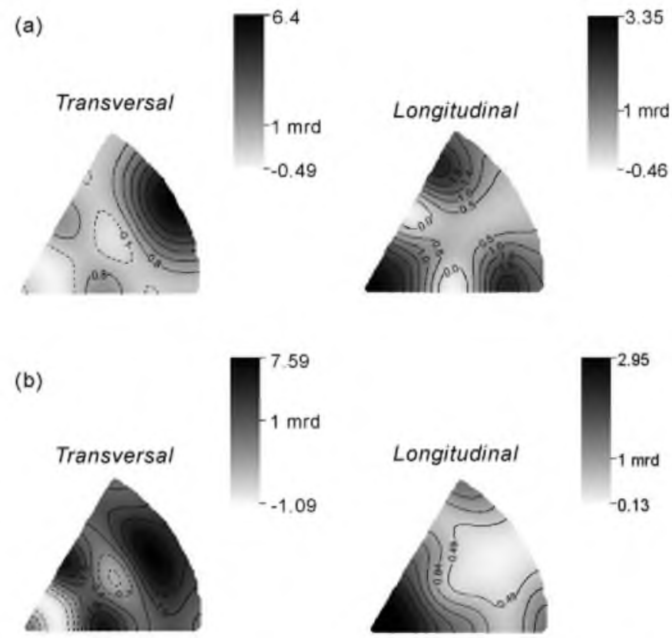

Fig. 7. Inverse pole figures from transversal and longitudinal sections of the specimens extruded at 600 °C (a) and 700 °C (b).

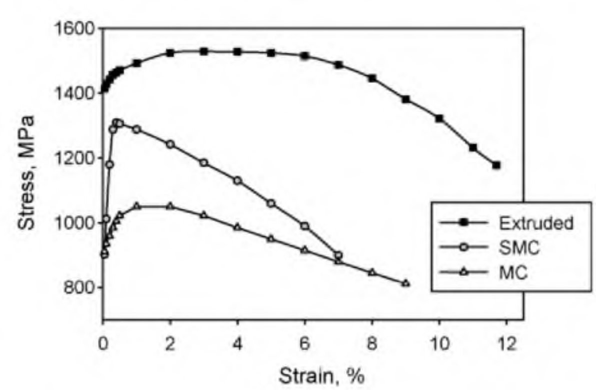

Fig. 8. Engineering stress–strain curves for specimens of Ti–6Al–4V alloy in different conditions.

4. Discussion

In the α/β titanium alloys with a lamellar phase morphology, microstructural refinement during hot/warm working is usually associated with globularization, i.e. conversion of the lamellar microstructure into a globular one (e.g. [13]). The size of the α- and β-grains, forming during globularization, is reduced with a reduction of the deformation temperature [14].

The microstructure of Ti–6Al–4V alloy hydrostatically extruded at 600–700 °C to the strain of 1.4–1.8 consists of grains or sub-grains 200–350 nm in diameter. For comparison, the grain size attained by a simple compression of Ti–6Al–4V at the same temperatures was about twice the size of that obtained by HE [5]. This effect may be attributed to the higher strain rate ($10^{-1}\,s^{-1}$ against $10^{-3}\,s^{-1}$) in the case of the extrusion process [11]. However, the percentage of the globularized microstructure in the hydrostatically extruded specimens was found to be quite low, about 20%, while the remainder of the material consisted of the remains of the lamellae aligned along the extrusion axis.

Titanium alloys are known to deform heterogeneously, especially at low-temperature. Heterogeneous deformation leads, in turn, to non-homogeneous globularization. It has been shown in [13] that colonies of lamellae with a "hard" orientation (a high Taylor factor) survive almost intact even after very large strains in the surrounding regions. The alternating of the lamellar and grain microstructures in the extruded specimen is apparently related to the different rates of globularization in the various α-colonies. During HE, the microstructure of some colonies is almost unchanged while the lamellae in other colonies become divided by the intra-phase dislocation boundaries (Fig. 4). With further straining the misorientation of those boundaries increased to the high-angle range leading to fragmentation of the lamellae and further globularization of the fragments (Fig. 4a) [5,13]. Unfavourably oriented lamellae may also be broken down and turned toward the "soft" orientation with a low Taylor factor by shear bands [13], however some lamellae were still oriented across the direction of metal flow (Fig. 3a).

Being controlled by diffusion [16], globularization can be intensified by raising the deformation temperature [17]. In this work the incremental increase of the extrusion temperature by 50 °C, from 600 °C to 650 and to 700 °C, slightly increased the globularization of the lamellar microstructure (Fig. 6) and increased the grain size.

The specimen of Ti–6Al–4V alloy extruded at 600 °C to $e = 1.4$ shows outstanding mechanical properties in spite of the microstructural heterogeneity. The value of the ultimate tensile strength, σ_U, was found to be 1530 MPa, which exceeds the σ_U of a completely globularized SMC Ti–6Al–4V alloy with grain size of about 0.4 μm by 230 MPa ($\sigma_U = 1300$ MPa) and by about 500 MPa for the MC heat-strengthened alloy ($\sigma_U = 1050$ MPa) [4]. It should be noted that the extruded alloy has also an appreciably higher ductility in terms of elongation to failure and elongation before necking than that of the SMC or MC alloys (Fig. 8).

The combination of a high strength and ductility demonstrated by the extruded alloy is very interesting. An attempt to explain such mechanical behaviour may be made on the basis of the microstructural and texture results obtained. Because of the alignment of the lamellae along the tensile axis the free path of dislocation glide within the α-lamellae of thickness 0.2 μm is expected to be $\boldsymbol{a}/\sin 45° \approx 0.3$ μm, consequently a mixture of the SMC grains and thin lamellae should behave like the usual fine-grained material. However, the mechanical behaviour of the hydrostatically extruded sample implies an ability to strain harden without exhibiting early necking [18].

It has been shown in [18] that uniformly mixing micron-sized grains into an ultrafine-grained matrix impart some strain hardening capacity to the material. In the hydroextruded alloy, the remains of the colony structure may play a similar role. However, in this case the dislocations should be able to percolate through the interphase 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 to allow for easy slip transmission across the α/β interphases [20]. The deformation obviously results in deterioration of coherency and an increase in the stress needed for the dislocations to cross the interphases. However, as long as the lamellar microstructure exists, a partial "transparency" of α/β boundaries for dislocations may remain. The strengthening effect of such boundaries may be described, by an analogy with low-angle boundaries, by a relationship similar to the Hall-Petch equation: $\sigma \approx d^m$, where d is the distance between the sub-boundaries or interphases and m is between 1/2 and 1 [21], depending on the "transparency" of the boundary. Consequently, parts of the material with the lamellar microstructure simultaneously increased the ductility of the extruded material and at the same time contributed, along with the SMC grains, to a high strength. However, further experimental data are needed to verify this hypothesis.

Another factor that may contribute to the relatively high ductility of the extruded alloy is the orientation of the tensile axis perpendicular to the c-axis of the hcp-lattices in the α-phase (Fig. 7). Consequently prismatic slip operates quite easily during the tensile test, which may lead to an increase in ductility [22]. However this factor plays rather a minor role, since a favourable crystallographic texture cannot explain either, an increase in strength, or delocalization of deformation for the extruded alloy.

Hydrostatically extruded Ti–6Al–4V alloy with such a high strength and ductility does have a potential for practical applications, typically for the production of high-strength wire.

5. Conclusions

1. HE of Ti–6Al–4V alloy under a pressure of 1.1 GPa at temperatures of 600–700 °C and strain rate of $10^{-1}\,c^{-1}$ to true strain $1.4 \div 1.8$ results in a considerable refinement of the microstructure. However, the microstructure is quite heterogeneous and consists of very fine grains (0.2–0.4 μm in size depending on the HE temperature) and packs of thin lamellae oriented along the extrusion axis.
2. As the result of HE at 600 °C to a true strain of 1.4, the alloy exhibits a very high strength whilst maintaining good ductility. The yield stress and ultimate tensile strength of the alloy was 1435 and 1530 MPa, respectively, and the elongation to fracture was 12%.

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