ARCHIVES OF METALLURGY AND MATERIALS
Volume 57 2012 Issue 3
DOI: 10.2478/v10172-012-0095-3

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MECHANICAL PROPERTIES OF TITANIUM PROCESSED BY HYDROSTATIC EXTRUSION

WŁAŚCIWOŚCI MECHANICZNE TYTANU WYCISKANEGO HYDROSTATYCZNIE

The mechanical properties of titanium Grade 2 subjected to the hydrostatic extrusion technique (HE) were investigated. The hydrostatic extrusion technique is a method which refines the titanium grains to a nano-metric size.

Compared with coarse grained titanium (CG-Ti), nanocrystalline titanium (NC-Ti) is characterized by a much higher yield stress, tensile strength and microhardness. The yield stress of NC-Ti determined in tensile tests is higher than that measured in compression test. The Young modulus of NC-Ti is slightly lower than that of CG-Ti.

Keywords: severe plastic deformation (SPD), nanocrystalline titanium, hydrostatic extrusion (HE), mechanical properties, nanocrystalline structure

Przedmiot badań stanowiły właściwości mechaniczne tytanu Grade 2 poddanego wyciskaniu hydrostatycznemu. Technika wyciskania hydrostatycznego jest metodą, która umożliwia rozdrobnienie ziarna tytanu do wielkości nano-metrycznej.

W porównaniu z tytanem gruboziarnistym (CG-Ti), tytan nanokrystaliczny (NC-Ti) charakteryzuje się o wiele wyższą granicą plastyczności, wytrzymałością na rozciąganie oraz mikrotwardością. Granica plastyczności NC-Ti wyznaczona podczas testów rozciągania jest wyższa niż granica plastyczności wyznaczona w testach ściskania. Natomiast moduł Young'a nanokrystalicznego tytanu jest nieznacznie mniejszy niż moduł tytanu gruboziarnistego.

1. Introduction

The present study is concerned with a new group of plastic forming processes that are based on heavy plastic deformation (known as the Severe Plastic Deformation methods - SPD). These methods are used for refining structure in metals and alloys to the nano-metric grain size [1-2]. One of them is hydrostatic extrusion (HE), which was used in this study for refining the microstructure of coarse-grained Ti (CG-Ti) to the nano-grained Ti (NC-Ti). Titanium Grade 2 in the form of rods was subjected to multi-pass extrusions during which the diameter of the rods was gradually reduced. The HE process was continued until the value of plastic deformation exceeded 3. It has been shown in [3-4] that the plastic forming by HE changes the average grain size of Ti, determined on transverse sections using an image analysis, below 100 nm (Tab.1).

The aim of this paper is to show in detail how such a change in grain size influences the mechanical properties of the material which is intended to work under the conditions of heavy loadings and force transmission. The examinations included the measurements of the yield stress, tensile strength, microhardness and Young modulus. The Young modulus is important form the constructional point of view, for example when selecting an appropriate material for implants into the human body. The mechanical properties of NC-Ti were compared with those of the Ti-6Al-4V titanium alloy. The results indicate that NC-titanium can successfully replace this alloy in a number of applications.

2. Material, processing and investigation methods

The material examined was commercially pure titanium (Ti⩾99.4% - Grade 2) in the form of rods. The rods of Ø20 and Ø33 mm were subjected to the multi-pass extrusion resulting in the final diameter Ø3 and Ø5 mm (see Table 1).

The hydroextrusion experiments consisted of three multi-pass processes in which the sample diameter was gradually reduced: (I) Ø20mm → Ø3mm, (II) Ø33mm

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TABLE 1

Process parameters of the titanium rods employed in this work

Sample – diameter [mm]	Material	State	Grain size [μm]	Accumulated strain (ε_T)	Number of HE passes
(I) Ø20	pure Ti	CG – initial – as received	5,8	–	–
(II) Ø33	pure Ti	CG – initial – as received	12,4	–	–
(III) Ø20	pure Ti	CG – initial – as received	160,0	–	–
(I) Ø3	pure Ti	NC – after HE – nano Ti	0,063	3.79	10
(II) Ø5	pure Ti	NC – after HE – nano Ti	0,055	3.77	12
(III) Ø3	pure Ti	NC – after HE – nano Ti	0,056	3.79	12
Ø5mm	Ti-6Al-4V	CG – initial – as received	–	–	–

→ Ø5mm and (III) Ø20mm → Ø3mm (Tab. 1). All the extrusions were carried out at room temperature. The HE technique employed in the present experiments was described in detail in our earlier publications [5,6]. The processes were carried out at the Institute of High Pressure Physics, Polish Academy of Sciences (Warsaw).

The tensile and compression tests were carried out at room temperature with the same initial straining rate = $3.3\times10^{-4}s^{-1}$. The Young modulus was determined by Berkovich indenter in nano-indentation tests as the tangent of the line to the unloading curve. The microhardness was measured on cross-sections using the Vickers method under a load of 200 g.

3. Results and discussion

The results of the tensile and compression tests showed that, hydrostatic extrusion causes a significant increase of the yield point and tensile strength (Tab.2). This can be attributed to the grain boundary strengthening associated with the grain refinement to the nano-metric size. The yield point of NC-Ti increases over 100% in both tension and compression (Tab.2 and 3). The tensile strength increased by 150% on average (Tab.2).

The results of the compression and tensile tests indicate that the yield point ($\sigma_{0.2}$) increases with increasing of strain accumulated (ε_T) in the extruded billets. The dependence of ε_T on the flow strain is nonlinear. The highest rate $d\sigma/d\varepsilon_T$ is observed during the initial extrusion passes.

Examples of ε_T dependence on compression strain are shown in Fig.1. The non-linear behavior revealed by the data points in Fig. 1 can be explained in terms of the strain localization and the weakening of the strengthening effect as the grain refinement proceeds. An additional factor is that the subsequent extrusion passes usually induce decreasing strain increment.

TABLE 2

Mechanical properties measured in tensile and compression tests ($\sigma_{0.2}$ – yield stress, σ_m – tensile strength, A – elongaiton)

process and sample		tensile test				compression test
		$\sigma_{0.2}$ [MPa]	σ_m [MPa]	A [%]	increase σ_m [%]	$\sigma_{0.2}$ [MPa]
(I) Ø20→ Ø3	Ø20-CG Ø3-NC	375 936	472 1058	16.4 7.8	– 122	360 770
(II) Ø33→ Ø5	Ø33-CG Ø5-NC	357 1040	482 1141	21.1 5.6	– 137	320 750
(III) Ø20→ Ø3	Ø20-CG Ø3-NC	265 966	375 1113	40 6.3	– 197	260 755
Ti-6Al-4V	Ø5	926	1140	7.9	–	880

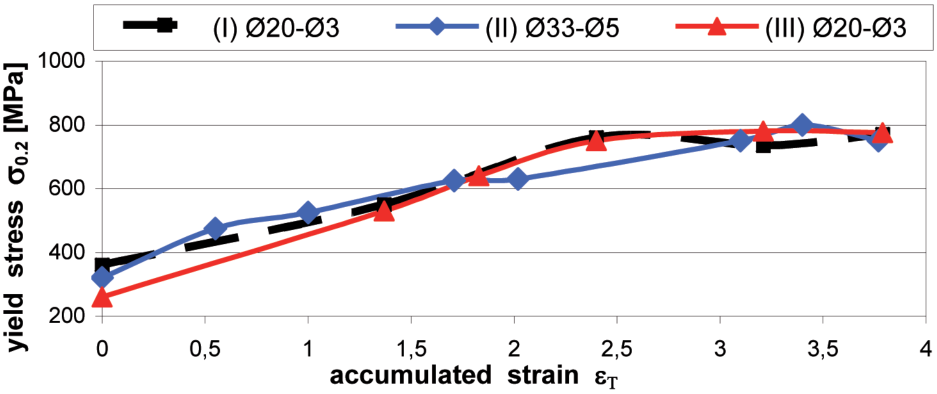

Fig. 1. Compression stress as a function of accumulated strain ε_T. Results for conducted number of extrusions

It should be noted that because of the friction and increasing of cross section of samples the results of the upsetting are increased and the force is not much higher. However, this effect refers identically the NC and CG materials. In this study, the results obtained for samples of micro-crystalline materials indicate that the yield stress in tension and compression are usually similar.

This is not the case for nanocrystalline (NC) and ultra-fine-grained (UFG) materials, which exhibit a substantial difference between the yield stress in tension and compression. This effect called the tension-compression asymmetry (T/C asymmetry) [7-15,22], has been observed in nanocrystalline titanium [11-13].

In the present study the values of the yield stress in tension and compression were similar, i.e. the T/C asymmetry was not observed for CG-Ti (Tab.2). In HE-processed NC-Ti the yield point in compression was lower than that in tension (Tab.2 and 3 – the values of the coefficient given in Table 3 are the $\sigma_{0.2}$ NC-Ti/$\sigma_{0.2}$ CG-Ti ratio). The T/C asymmetry took place in NC-Ti. Therefore this effect is a feature of the nano-metric structure of HE-processed titanium and is very likely related to the elongated shape of grains. Additionally the tensile straining increases elongation of grains.

Moreover, in NC-Ti the grain boundary regions, which are more liable to compression than the grain interiors, occupy a relatively great volume [14,15]. Another factor responsible for the T/C asymmetry could be a high level of residual stresses observed in the materials subjected to HE.

It should be noted that in the literature reports, concerning the UFC and NC materials, the value of $\sigma_{0.2}$ in compression is higher than in tension. This was observed in e.g. aluminum, copper and nickel in which the yield point in compression was higher than that in tension by 20-30% [22]. It is also supposed that the compressibility of NC materials may increase with decreasing grain size, which was confirmed by the dynamic molecular simulation performed for NC-Ni [15].

The results obtained here shown that the strength of the NC-Ti rods processed by HE are near those of the Ti-6Al-4V (Tab.2). This permits to conclude that NC-Ti can be considered an alternative to the Ti alloys in structural applications.

TABLE 3

Tension / compression asymmetry on terms of the percentage increase and yield stress ratio

	process	tensile test	compression test
$\sigma_{0.2}$ increase [%]	(I)	150	114
	(II)	191	134
	(III)	264	190
ratio: $\sigma_{0.2}$ NC-Ti / $\sigma_{0.2}$ CG-Ti	(I)	2.50	2.14
	(II)	2.91	2.34
	(III)	3.65	2.90

The Young modulus of titanium and its alloys widely varies depending on the temperature and chemical composition. It also depends on the crystallographic orientation so the Young modulus of microcrystalline titanium can range from 100 to 148 GPa.

The values of E measured in the HE-processed samples ranged from 107 to 129 GPa (Tab.4). Thus, they appear to be smaller than those of coarse-grained Ti (148 GPa.)

TABLE 4

Values of the Young modulus for the HE-processed, coarse-grained titanium and Ti6Al4V alloy

Specimen	E [GPa]
CG-Ti Ø33 – before HE	148
NC-Ti Ø3 (I) – after HE	108
NC-Ti Ø5 (II) – after HE	107
NC-Ti Ø3 (III) – after HE	129
Ti-6Al-4V	130

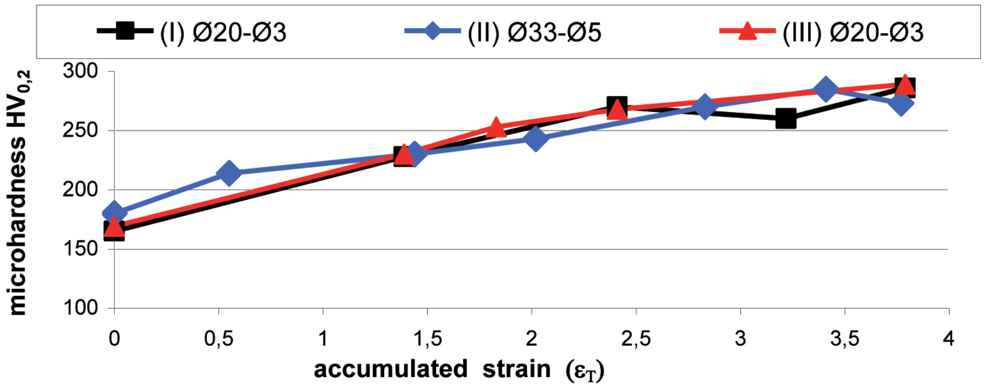

Fig. 2. Microhardness as a function of strain

A lower value of E for nano-materials is attributed to a significant number of the atoms located near the grain boundaries [19]. It is also probable that the results obtained in the present experiments are associated with the increased number of grain boundaries in the material volume and their elastic deformation. It may also be due to the technological defects, such as micro-cracks and pores [20, 21]. However in our experiments this factor did not influence the value of E since the three-axial compressive stresses that act during HE eliminated the possibility of cracking.

Materials with the structure refined by the SPD methods exhibit a higher hardness [16-18]. The results of microhardness measurement show that comparing with the starting material (CG-Ti) the NC-Ti obtained in the present experiments exhibits the microhardness increasing more than 50%. In fact microhardness of NC-Ti approaches to the microhardness of the Ti-6Al-4V alloy (Tab.5).

TABLE 5
Microhardness of NC-Ti and its percent increase with respect to the microhardness of the starting material

Sample	$HV_{0.2}$	Increase [%]
(I) Ø20-CG	165	–
(I) Ø3-NC	286	73
(II) Ø33-CG	177	–
(II) Ø5-NC	273	54
(III) Ø20-CG	170	–
(III) Ø3-NC	290	70
Ti-6Al-4V	315	-

The results further demonstrate that microhardness increases gradually with the increasing of ε_T value. The fastest increase was observed during the first extrusions as shown in Fig. 2. For example, at the strain ε =1.4 the hardness increases by 34% (on average), whereas with the further increase of the strain up to ε =3.8 it only increases by 66%. In HE, the smaller increase of the hardness during the final extrusion stages may also be attributed to the smaller increase of the strain during these stages. The microhardness varies as a function of ε_T in the same manner as the yield point, however the changes are smaller.

4. Conclusions

Titanium Grade 2 rods were subjected to hydroextrusion and their mechanical properties before and after were examined. The HE yielded nanocrystalline titanium with a grain size of about 60nm. The refinement of the grains to the nano-metric scale resulted in a significant improvement of the mechanical properties, such as the yield stress, tensile strength, and the microhardness.

The values of $\sigma_{0.2}$ and σ_m of NC-Ti increased by 100 to 200% and the microhardness increased by more than 50%. The yield stress is different in compression and in tension. The effect which is known as the tension/compression asymmetry (T/C asymmetry) has appeared. In compression the yield stress is smaller by about 23%. The value of the Young modulus in NC-Ti was below that of CG-Ti, which may be attributed to the significant contribution of the grain boundaries in the volume of the NC material.

Acknowledgements

Financial support of Structural Funds in the Operational Programme – Innovative Economy (IE OP) financed from the European Regional Development Fund – Project Nr POIG.01.01.02-00-015/08-00.

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Received: 20 April 2012.