MATERIALS.

TECHNOLOGIES.

DESIGN

УДК 669.721:539, P.A.C.S. 62.25.-g

Strength and fracture mechanism of nanomaterials and medical products

Прочность и механизм разрушения наноматериалов и изделий медицинского назначения

G. V. Klevtsov ¹, R. Z. Valiev ², N. A. Klevtsova ³, I. V. Alexandrov ⁴, A. A. Matchin 5, M. N. Tyurkov 6, M. V. Fesenyuk 7 Г. В. Клевцов ¹, Р. 3. Валиев ², Н. А. Клевцова ³, И. В. Александров ⁴, А. А. Матчин ⁵, М. Н. Тюрьков ⁶, М. В. Фесенюк ⁷

- ^{1,3,6} Togliatti State University, Belorusskaya Str. 14, Togliatti, 445020, Russia
- ^{2,4}Ufa State Aviation Technical University, K. Marx Str. 12, Ufa, 450008, Russia
- ⁵ Orenburg State Medical University, Sovetskaya Str. 6, Orenburg, 460000, Russia
- ⁷ JSC PA «Strela», Shevchenko Str. 26, Orenburg, 460005, Russia
- ¹Klevtsov11948@mail.ru
- ^{1,3,6} Тольяттинский государственный университет, Россия, 445020, Тольятти, ул. Белорусская, 14
- 2.4 Уфимский государственный авиационный технический университет, Россия, 450008, Уфа, ул. К. Маркса, 12
- Оренбургский государственный медицинский университет, Россия, 460000, Оренбург, ул. Советская, 6
- ⁷ АО «ПО «Стрела»», Россия, 460005, Оренбург, ул. Шевченко, 26

ABSTRACT

The strength and fracture mechanism of Grade 4 titanium, austenitic corrosion-resistant steel, and magnesium alloy Mg-Zn-Ca under tension, torsion, shock loading and fatigue were studied. The materials were investigated in the initial coarse-grained (CG) state and after equal-channel angular pressing (ECAP) in the ultrafine-grained (UFG) state. The studies carried out have shown that the strength properties of the materials under study in the UFG state for all types of static and fatigue tests are higher or comparable in comparison with materials in the CG state. The only material characteristic that has decreased during ECAP is toughness. Therefore, these UFG materials, along with CG materials, are promising for the manufacture of medical devices for various purposes, experiencing various static and cyclic loads during operation.

KEYWORDS

Medical devices; titanium; austenitic steel; magnesium alloy; structure; strength; fracture.

КИДАТОННА

Исследована прочность и механизм разрушения широко применяемого в медицине титана Grade 4, аустенитной коррозионностойкой стали и магниевого сплава Mg-Zn-Ca при растяжении, кручении, ударном нагружении и усталости. Материалы исследованы в исходном крупнозернистом (КЗ) состоянии и после равноканального углового прессования (РКУП) в ультрамелкозернистом (УМЗ) состоянии. Проведенные исследования показали, что прочностные свойства исследуемых материалов в УМЗ состоянии при всех видах статических и усталостного испытаний выше или соизмеримы по сравнению с материалами в КЗ состоянии. Единственная характеристика материалов, которая снизилась в процессе РКУП – это ударная вязкость. Поэтому данные УМЗ материалы наряду с КЗ материалами являются перспективными для изготовления медицинских изделий различного назначения, испытывающих в процессе эксплуатации разнообразные статические и циклические нагрузки.

КЛЮЧЕВЫЕ СЛОВА

Медицинские изделия; титан; аустенитная сталь; магниевый сплав; структура; прочность; разрушение.

¹ Klevtsov11948@mail.ru

Introduction

The use of "sparing" operating technologies in maxillofacial surgery, traumatology and other areas of medicine involves the miniaturization of medical devices: a variety of implants, instruments and devices designed for attaching implants and bone fragments. During operation, such products can experience various stresses, both in magnitude and in the type of loading: static, shock, cyclic [1]. Therefore, the problem of miniaturization of medical devices cannot be solved without the use of materials that, in addition to good biocompatibility, have a high set of mechanical properties under various types of loading. These requirements are fully met by a new class of bulk nanostructured metallic materials with an ultrafine-grained (UFG) structure obtained by severe plastic deformation (SPD) methods, in particular, by equal channel angular pressing (ECAP) [2]. Therefore, the issue of the influence of the UFG structure on the strength and fracture mechanism of medical materials under various types of loading is

The purpose of this work is to assess the strength and fracture mechanisms under various types of loading of medical materials after ECAP in comparison with the original coarse-grained (CG) materials.

1. Materials and research methods

Titanium Grade 4, austenitic corrosionresistant steel (0.023% C, 17.95% Cr, 7.95% Ni, 1.85% Mn, 0.6% Cu, 0.35% Mo, 0.38% Si, 0.15% Co,) and magnesium alloy Mg-Zn-Ca (1.0% Zn, 0.2% Ca), widely used in medicine, were chosen as the investigated materials in CG and UFG states.

Titanium Grade 4 in the CG state was investigated after homogenization annealing at a temperature of 680 °C for 1 hour. The UFG state was obtained according to two regimes. The first regime: annealing + ECAP-conform (ECAP-C) at 250 °C (route Bc, n = 6). The second regime: annealing + ECAP-conform at 200 °C + drawing (ECAP-C + D) at 200 °C. CG austenitic steel was investigated in the initial (hot-rolled) state. The UFG steel structure was obtained by quenching from 1050 °C + ECAP at a temperature of 350 °C (route Bc, n = 4, $\varphi = 120$ °). Magnesium alloy Mg-Zn-Ca in the CG state was investigated after homogenization annealing at a temperature of 450 °C, 24 hours, cooling in water. ECAP was carried out according to the regime: route Bc, $\varphi = 120^{\circ}$, n = 2 at a temperature of 430 °C + n = 1at a temperature of $400 \, ^{\circ}\text{C} + \text{n} = 1$ at $350 \, ^{\circ}\text{C}$.

The fine structure of UFG materials was investigated using a JEM-6390 scanning electron microscope and a JEM-2100 transmission electron microscope. Mechanical tests included: hardness tests (hardness tester TN 300), tensile tests (N50KT machine), impact strength (pendulum impact devices JB-W300 and TSKM-50), torsion tests (KTS 403-20-0.5 and MK-50), fatigue strength (Instron machine 8802).

2. Results of the study

It is known that the structure of materials after ECAP is characterized by grain refinement, a significant increase in crystal structure defects, and a number of other features [2]. It can be seen from Table 1 that after ECAP the hardness and strength properties of materials in tension increase, and the elongation decreases, with the exception of the magnesium alloy, in which after ECAP the elongation increases by a factor of 2.5.

Analysis of operational damage of medical devices showed that a large percentage of the destruction of screws for fixing plates and bone fragments in maxillofacial surgery and traumatology occurs by twisting the smooth area of the screw between the threaded part and the head during the process of unscrewing the screws that have grown together with the bone, for example, after the patient recovers. Therefore, it is important that the material of the screw resists deformation and torsional fracture well. The test results showed (Fig. 1, a, b, c) that UFG titanium and austenitic steel have higher torsional strength properties in comparison with the corresponding CG materials (Fig. 1, a, b).

Average grain size and tensile properties of materials

Средний размер зерна и механические свойства материалов при растяжении

Material	State	$d_{av.,}\mu m$	НВ	UTS, MPa	YS, MPa	El., %
	CG (annealing)	25	255	750±10	650±30	20±0.5
Grade4	UFG (ECAP-C)	0.4	293	1050±15	900±25	14±0.7
	UFG (ECAP-C+D)	0.2	_	1250±10	1100±30	11±0.5
	CG (annealing)	415	_	119±9	65±5	9±0.3
Mg-Zn-Ca	After ECAP	5÷40	_	210±10	97±7	23±0.5
	CG (initial)	30	159	624±6	283±2	65±0.7
Stainless steel	UFG (ECAP)	0.55	363	1112±15	1065±15	20±0.5

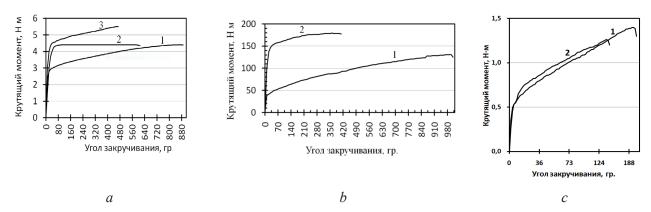


Fig. 1. Diagram "Torque – twist angle" during torsion testing of specimens with a diameter of 3 mm from CG (1) and UFG titanium Grade 4 (a): ECAP-C (2), ECAP-C + D (3), specimens with a diameter of 10 mm from CG (1) and UFG (2) stainless steel (b) and specimens with a diameter of 3 mm from magnesium alloy Mg-Zn-Ca (c) in the initial state (1) and after ECAP (2)

Рис. 1. Диаграмма «Крутящий момент – угол закручивания» при испытании на кручение образцов диаметром 3 мм из КЗ (1) и УМЗ титана Grade 4 (a): РКУП-К (2), РКУП-К+в (3), образцов диаметром 10 мм из КЗ (1) и УМЗ (2) стали 08Х18Н9 (b) и образцов диаметром 3 мм из магниевого сплава Mg-Zn-Ca (c) в исходном состоянии (1) и после РКУП (2)

The torsional strength characteristics of a magnesium alloy after ECAP differ little from those for the alloy in the initial state (Fig. 1, c). Consequently, titanium and stainless steel with the UFG structure are more promising for the manufacture of medical screws in comparison with CG materials.

The results of testing materials for impact strength (KCV) showed (Table 2) that for all investigated materials ECAP leads to a decrease in the value of KCV at room temperature. Therefore, when using medical devices made of UFG materials, one-time dynamic loads should be avoided.

Average value of impact strength (KCV) of materials at 20 °C Среднее значение ударной вязкости (КСV) материалов при 20 °C

Material	Titanium Grade 4		Stainless steel		Mg-Zn-Ca alloy	
State	CG (annealing)	UFG (ECAP-C)	CG (initial)	UFG (ECAP)	CG (annealing)	After ECAP
KCV, MJ / m ²	0.27	0.07	2.9	0.7	6.0 · 10 ⁻²	2.7 · 10-2

It is known that ECAP increases the fatigue limit of most structural materials and has an ambiguous effect on the fatigue strength in the region of low-cycle fatigue, the region where most of the destruction of medical implants occurs. Testing of samples in the region of lowcycle fatigue showed that for the UFG materials studied, the coefficient n in the Paris equation is less than for CG materials, therefore, they are less sensitive to cyclic overloads. As an example, Fig. 2 shows the results of fatigue testing of the austenitic stainless steel under study.

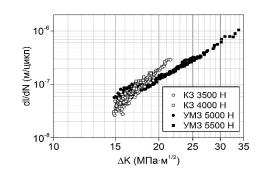


Fig. 2. Straight line section of kinetic diagrams of fatigue fracture of the stainless steel in the initial state (light points) and after ECAP (dark points)

Рис. 2. Прямолинейный участок кинетических диаграмм усталостного разрушения стали в исходном состоянии (светлые точки) и после РКУП (темные точки)

Conclusion

The studies have shown that the strength of UFG titanium Grade austenitic stainless steel and magnesium alloy Mg-Zn-Ca in all types of static tests (tension, torsion) and fatigue tests are higher or comparable in comparison with CG materials. The only material characteristic that decreased during ECAP is impact strength. Therefore, these materials in the UFG state, along with CG materials, are promising for the manufacture of medical devices for various purposes, experiencing various static and cyclic loads during operation.

Acknowledgments

This work was supported by a grant from the Russian Science Foundation (Project no. 20-63-47027).

References

- 1. Nanostructured Titanium for Maxillofacial Mini-Implants / I. P. Semenova et al. // Advanced Engineering Materials. 2016. V. 18, No. 7. P. 1216-1224.
- 2. Valiev R. Z., Zhilyaev A. P., Langdon T. G. Bulk Nanostructured Materials: Fundamentals and Applications: Hoboken. New Jersey: John Wiley & Sons, 2014. 440 p.