Impact Toughness and Dynamic Compression of Ultrafine-Grained Titanium Grade 4

Ivan V Smirnov¹,² and Alexander Y Konstantinov¹

¹Research Institute for Mechanics, Lobachevsky State University of Nizhni Novgorod, 23 Prosp Gagarina, Nizhny Novgorod, 603950, Russia
²Laboratory for Mechanics of Advanced Bulk Nanomaterials for Innovative Engineering Applications, Saint Petersburg University, 7/9 Universitetskaya nab., Saint Petersburg, 199034, Russia

Abstract. This work presents the study of an influence of ultrafine-grained (UFG) structure on impact toughness and dynamic compression of commercially pure titanium Grade 4. The UFG structure was provided by means of equal-channel angular pressing according to the Conform scheme (ECAP-Conform, Ufa) with subsequent heat treatment. Impact toughness tests on samples with U-shaped notch were carried out using a drop weight impact machine. Dynamic compression test of cylindrical samples were carried out on a setup with the Split-Hopkinson pressure bar (SHPB-20) by the Kolsky method. It was found that the impact toughness of the UFG titanium is 15% greater than that of its coarse-grained (CG) counterpart. However, fracture process of the CG material requires 1.5 times more energy. The ECAP treatment significantly increased yield strength of the material. At the same time, an increase of compression strain rate by 6 orders resulted in an increase of yield strength of the CG material by 20%, while yield strength of the UFG titanium remained at the level of a quasi-static load. The UFG material showed a well-expressed strain hardening behaviour for all strain rates.

1. Introduction
Ultrafine-grained (UFG) titanium have attracted significant research attention in the last few decades due to its superior mechanical properties, such as high strength and yield stress, as well as good endurance limit [1,2]. However, one lack that prevents its engineering application is a decreased ductility.

A lot of research is being done to increase the ductility of UFG and nanocrystalline titanium. For example, in Ufa State Aviation Technical University a new approach to enhance the strength and ductility of metallic materials was developed by controlling the grain boundary structure that can be considered as grain boundary engineering of UFG metallic materials [3]. The effective approach in ductility enhancement of UFG titanium is formation of a high fraction of high-angle boundaries in the UFG structure with providing recovery processes of non-equilibrium boundaries [4, 5]. Such approach is possible via combined techniques that include equal channel angular pressing (ECAP), subsequent thermomechanical treatment and annealing [5].

Most studies on the mechanical behaviour of UFG titanium are focused on the phenomena occurring under quasi-static loading, whereas research on dynamic response and fracture of the material under high strain rates and pulse loads are rare. Nevertheless, dynamic characteristics are critically important for evaluating the potential application of ultrafine-grained materials.
The purpose of this work is to begin developing data on dynamic response and dynamic fracture parameters of ultrafine-grained titanium Grade 4, which has not only high static strength, but also good ductility.

2. Materials and experimental procedures

2.1. Materials
In this work, the states of commercially pure titanium Grade 4 with a coarse-grained (CG) and ultrafine-grained (UFG) structure are considered. The chemical composition of the material, in wt.%, was 0.05C, 0.15Fe, 0.05N, 0.007H, 0.36O, residuals<0.3, and Ti as the balance.

The initial material with a coarse-grained structure was produced by Dynamet Company (USA) in the form of hot-rolled rods with Ø12 mm.

The ultrafine-grained structure was produced in Ufa State Aviation Technical University using equal-channel angular pressing (ECAP) combined with the Conform process. The installation was with an angle of 120° in the ECAP abutment. The rod was processed through a total of six passes using route BC at a temperature of 200 °C. Before the ECAP, the rod was annealed at 680 °C for 1 hour. After the ECAP, the rod was annealed at 250 °C for 1 hour and then drawn with a die diameter of 12 mm. This combination of processing regimes makes it possible to produce UFG titanium with high strength and the greatest ductility, and also to ensure uniformity of mechanical properties along the volume of a rod. A more detailed description of the processing regimes and the corresponding properties of the produced titanium, as well as its structure, can be found in [6, 7].

The characteristic parameters of the materials are given in Table 1.

| Structure/ Parameter      | CG   | UFG  |
|---------------------------|------|------|
| Average grain size d (μm)| 11   | 0.7  |
| Microhardness HV          | 243  | 317  |
| Tensile strength σ_t (MPa)| 685  | 1004 |
| Tensile yield strength σ_y (MPa) | 514 | 991  |

2.2. Impact toughness tests
The impact toughness tests were carried out using a drop weight impact test system (Instron CEAST 9350). The Charpy scheme was applied in according to the standard GOST 9454 [8]. The impactor speed at the time of impact was 5 m/s that corresponded to the impact energy ~68 J. Load and energy absorption diagrams were determined by the system of sensors and computer control.

Samples had a length of 55 mm, a height of 8 (working 6) mm and a width of 2 mm and also a U-shaped notch with a curvature radius of 1 mm. The samples were cut from the rod using an electro erosion machine, so that the length of the sample was located along the longitudinal direction of the rod.

2.3. Dynamic compression tests
Tests on dynamic compression of cylindrical samples were performed on a setup with the Split-Hopkinson pressure bar (SHPB) by the Kolsky method [9]. A mathematical model of SHPB is a system of three bars: two infinitely strong and infinitely long thin bars and a "soft" short bar (sample) between them (Figure 1).

An impactor 1 generates a one-dimensional elastic compression wave in an incident bar 2. This wave propagates along the bar with a speed of c. In view of a difference in an acoustic stiffness of the materials of the bar 2 and sample 4, the wave is split so that part of it is reflected back, and the other part passes through the sample into the transmitter bar 5. In this process, the sample can undergo elastic-plastic deformation, while the bars deform only elastically.
The pulses of deformation of the incident bar (incident pulse and reflected pulse) and the transmitter bar (transmitted pulse) were measured using strain gauges 3 and 6 glued to the lateral surfaces of the bars. The signals from the strain gauges were captured on a multichannel computer measuring system. Formulae for the calculation of the mechanical characteristics by the pulses of deformation of the measuring bars in case of the dynamic compression were proposed by Kolsky. A more detailed description of the wave theory and computational formulas can be found in [9, 10].

A set of steel measuring bars with a diameter of 20 mm was used for titanium testing. The system was loaded by a steel impactor with a length of 300 mm and a diameter of 20 mm. Dynamic compression was performed for two impactor speeds (and, correspondingly, strain rates) 13 and 22 m/s.

The samples had a length of 4 mm and a diameter of 8 mm. The samples were cut from the rod using an electro erosion machine, so that the cylinder axis was located along the longitudinal direction of the rod.

Quasi-static tests were carried out using standard test machine (Shimadzu AG-50kNX) with a capturing speed of 1 mm/min. A sample deformation was measured by a traverse displacement.

3. Results and discussion

3.1. Impact toughness tests
It is well known that a grain reduction can lead to an increase in metal strength. On the other hand, this leads to a decrease in ductility of the material. However, as shown in [5, 7], the correctly selected sequence of processing regimes of severe plastic deformation and heat treatment allow obtaining the UFG material with good ductility.

Figure 2 shows the results of the impact toughness tests of the titanium with the ultrafine-grained and coarse-grained states. The impact toughness of the UFG titanium is 15% greater than the impact toughness of its original coarse-grained analogue. At the same time, the fracture of coarse-grained material requires 1.5 times more energy.

Fracture surfaces have a dimpled character for both material states, Figure 3. The similar dimpled fractography was observed in impact toughness tests of Ti Grade 5 in [11]. Such a fracture surface indicates a localized plastic deformation, which suggests good perspective for increasing the plastic properties of ultrafine-grained titanium.

3.2. Dynamic compression tests
Figure 4 shows the "engineering stress-engineering strain" curves for the compression of the titanium at different strain rates. The curves for the initial grain state of the material are significantly different for its ultrafine-grained state. The yield point (for 0.2% deformation) at slow deformation is 660 MPa for the CG material and 1020 MPa for the UFG material. An increase in the strain rate by 6 orders led to an increase in the yield stress of the CG material to 810 MPa. However, the yield stress for the UFG material remained at the level corresponding to the quasi-static loading.
**Figure 2.** Characteristic diagrams in impact toughness tests of titanium. a) coarse-grained state; b) ultrafine-grained state; A is the displacement work; S is the area of a sample cross section in the point of impact; t* is the start time of macro fracture; KCU is the impact toughness; KCU* is the full specific work.

**Figure 3.** Fracture surfaces of titanium Grade 4 after impact toughness tests. a) coarse-grained state; b) ultrafine-grained state.

**Figure 4.** Characteristic diagrams in compression tests of titanium for different strain rates. a) coarse-grained state; b) ultrafine-grained state.

Deformation hardening is observed for both states and for all strain rates that is unusual for materials with ultrafine-grained structure. Thus, due to combination modes of ECAP and heat treatment, it was possible to achieve such a structure with large-angle grain boundaries, which promote the mechanisms of grain boundary sliding and contribute to strain hardening during plastic
deformation [5], that results in ductility enhancement. However, the ultrafine-grained titanium still showed less ductility than the coarse-grained material at a dynamic load.

4. Conclusions
Impact toughness and response on dynamic compression of pure titanium Grade 4 processed by equal-channel angular pressing (ECAP-Conform) were investigated. ECAP processing resulted in an ultrafine-grained (UFG) structure of the material. The impact toughness tests were carried out according to the Charpy scheme for samples with U-notch. The dynamic compression tests were carried out on a setup with the Split-Hopkinson pressure bar at strain rates of up to $10^3$ 1/s. The conclusions are as follows:

1. Impact toughness of UFG titanium processed by optimal regimes and combination of ECAP and heat treatment can be no worse than that of the material prior to processing. In this study the impact toughness of the UFG titanium is 15% greater than that of its coarse-grained (CG) counterpart. Both CG and UFG samples fractured in a ductile manner. However, fracture process of the CG material requires 1.5 times more energy.

2. The UFG titanium showed significantly enhanced yield strength compared to the CG titanium at quasi-static loading. However, an increase in a compression strain rate by 6 orders resulted in an increase in yield strength of the CG material by 20%, while yield strength of the UFG titanium remained at the level of a quasi-static load. Both CG and UFG samples showed strain hardening behavior for all strain rates.

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