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DEVELOPMENT OF THE PRODUCTION OF ULTRAFINE-GRAINED TITANIUM WITH THE CONFORM EQUIPMENT

RAZVOJ PROIZVODNJE ULTRADROBNOZRNATEGA TITANA Z OPREMO CONFORM

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Ultrafine-grained titanium belongs to the materials that possess a high potential for medical applications and, thanks to its mechanical properties, it is already frequently employed in this field. The know-how for manufacturing ultrafine-grained titanium represents one of the most complex issues in the titanium processing. The CONFORM process is one of the techniques available for this purpose. The principle of the CONFORM technique is the continuous ECAP (Equal Channel Angular Pressing) process. At present, it is primarily used for processing the materials with a low flow stress and a good formability. This paper gives a description of this forming technique and its applications in the production of titanium semi-products. Grade-4 titanium was used for the experimental programme. The factors explored in the process trials included the impacts of the shape of the exit chamber and of the temperatures at the key locations within the CONFORM process on the resulting microstructure of the titanium product. Numerical simulations of the process were performed in the DEFORM 3D software using various friction conditions. The actual flow of the material was compared with the results of the simulation.

Keywords: titanium, CONFORM, ECAP, ultrafine grain

Ultradrobnozrnat titan spada k materialom, ki imajo velike možnosti za uporabo v medicini in se že uporablja na tem področju po zaslugi svojih mehanskih lastnosti. Tehnologija izdelave ultradrobnozrnatega titana je najbolj kompleksen izziv pri njegovi proizvodnji. Postopek CONFORM je ena od razpoložljivih tehnik za ta namen. Osnova tehnike CONFORM je kontinuiren postopek ECAP (stiskanje skozi pravokoten kanal). Sedaj se uporablja za predelavo materialov z nizko napetostjo tečenja in dobro preoblikovalnostjo. Članek opisuje to tehniko preoblikovanja in njeno uporabo pri proizvodnji titanovih polproizvodov. Titan čistosti grade-4 je bil uporabljen za preizkuse. V preizkusnih procesih so bili uporabljeni nekateri dejavniki na ključnih mestih pri CONFORM-u, vključno z učinkom oblike izhodnega kanala in temperature, na mikrostrukturo proizvoda iz titana. Numerične simulacije procesa so bile izvršene s programsko opremo DEFORM 3D z uporabo različnih razmer pri trenju. Resnični tok materiala je bil primerjan z rezultati simulacije.

Ključne besede: titan, CONFORM, ECAP, ultradrobna zrna

1 INTRODUCTION

Numerous SPD processes (Severe Plastic Deformation) were developed in the recent 15 years. These processes are used for achieving a grain refinement in materials, typically to a grain size between 100 nm and 400 nm. Their efficiency in processing a large volume of a material is, however, still insufficient for industrial-scale applications. This drawback was eliminated with the CONFORM method. The method has been known for a long time. Today, it is used for the continuous industrial-scale production of sections, mostly from aluminium. Essentially, it is an upgraded ECAP (Equal Channel Angular Pressing) method, where the feedstock is forced through a die by a friction force exerted by a roll that turns ECAP into a continuous process.

This ECAP–CONFORM process was reported for the first time in[1]. In this study, a commercial-grade aluminium wire was extruded using one to four passes. The preparation of ultrafine-grained titanium using this method was reported in[2]. In this study, a 7.2 mm × 7.2 mm square wire with an accumulated strain of $e = 6$ was subsequently redrawn into a diameter wire 3 mm (resulting in the total strain of 7.9).

In the present paper, the production of commercial-grade-titanium round bars using a single pass through the ECAP-CONFORM equipment is described.

2 EXPERIMENTAL WORK

The feedstock consisted of the CP Ti grade 2 bar with a diameter of 10 mm. Their chemical composition is listed in **Table 1**. It was measured using a Bruker Q4 Tasman optical emission spectrometer and a Bruker G8 Galileo gas analyzer.

Table 1: Chemical composition of the feedstock in mass fractions (w/%)
Tabela 1: Kemijska sestava surovca v masnih deležih (w/%)

Fe	O	C	H	N	Ti
0.046	0.12	0.023	0.0026	0.0076	99.822

Titanium bars were converted into the bars of the same diameter as that of the feedstock using a CONFORM 315i machine.

The resulting microstructure was examined using a Nikon Eclipse MA 200 optical microscope. The specimens for the observation in the optical microscope were prepared by metallographic grinding and polishing and then etched using a Kroll agent. The specimens for an EBSD analysis were prepared using a JEOL SM-09010 ion polisher. The EBSD analysis was conducted using a JEOL 7400 electron microscope with a HKL-NORDLYSS EBSD camera. The data were processed using the Chanel5 software.

The mechanical properties at room temperature were measured using the cylindrical tension-test specimens with a gauge length of 25 mm and a diameter of 5 mm. In addition, impact-toughness tests were conducted using 3 mm × 4 mm section specimens.

The coefficient of the friction between CP-Ti and Inconel (the material of the die chamber) was measured by means of a tribological pin-on-disc test.

The process of continuous extrusion of titanium was modelled using the DEFORM 3D software based on the FEM method. The strain and temperature fields that varied with time were analysed with mathematical tools. The heat-transfer coefficient between the titanium feedstock and the wheel was measured to be 1000 W/(m^2 K). The input material data were obtained from the material-data calculation in the JMatPRO software.

3 RESULTS AND DISCUSSION

In the trials of extruding a Ti wire with the CONFORM equipment, the key parameters varied: the speed of the wheel, die-chamber temperatures, the cooling downstream of the die chamber and others. The temperature vs. the time record from the experiment is shown in **Figure 1**. The decisive parameter for the entire process is the die-chamber temperature that varied between the initial 500 °C and the final value of 350 °C. Numbers 1–4 in the figure denote the time instants, at which the samples were taken for an EBSD analysis. The point, at which the Ti wire was cooled right after exiting the die chamber, is identified as well.

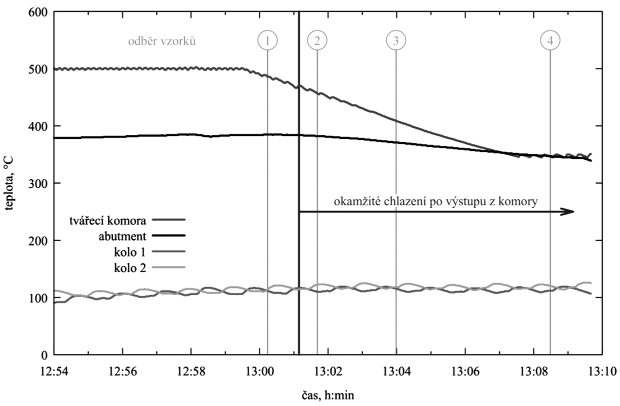

Figure 1: Temperatures at the key locations during the ECAP-CONFORM processing of a titanium bar

Slika 1: Temperature na ključnih mestih med ECAP-CONFORM stiskanjem titana

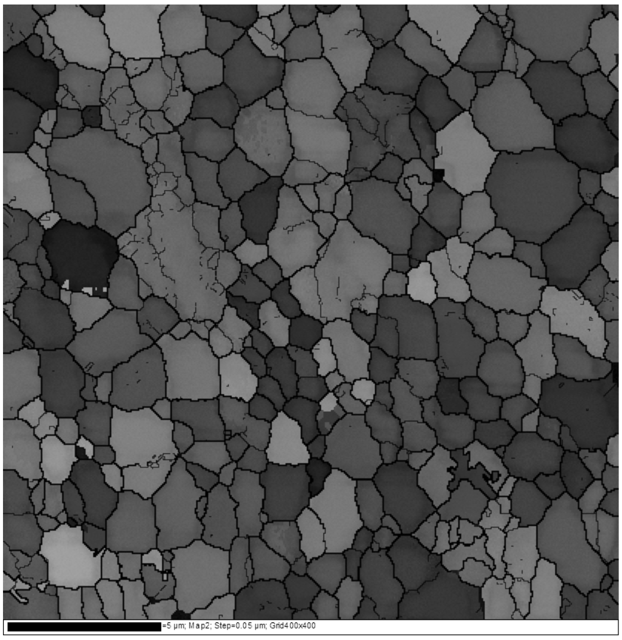

Figure 2: EBSD micrograph of specimen no. 4

Slika 2: EBSD-posnetek vzorca št. 4

The microstructure of specimen 1 consists of undeformed equiaxed grains. The grain-size pattern is bimodal and the average grain size is 1.9 µm. Neither the small nor the large grains show signs of deformation.

The microstructure of specimen 2 is identical to that of specimen 1. The chamber temperature of 450 °C is sufficient for the microstructure to recover/recrystallize. No effects of cooling were observed. The recovery/recrystallization and potential grain growth were finished before the specimen was cooled.

Specimen 3 (the die-chamber temperature of 400 °C) contains a small amount (10–15 %) of distorted unrecrystallized grains. The specimen was cooled with water upon exiting the chamber. The deformed grains with a size of no more than 5 µm × 10 µm are divided by low-angle boundaries into subgrains. Their average size is 1.9 µm.

Specimen 4 was formed at the die-chamber temperature of 350 °C and then cooled with water upon exiting the chamber. Its microstructure consists of slightly elongated, deformed grains that, however, lack the above-mentioned substructure (**Figure 2**). The EBSD analysis focused on the centre of the circular cross-section of the extruded product. The analysed surface is on the plane that is parallel to the bending plane/flow plane in the CONFORM chamber. On the EBSD maps shown, the axis of the extruded section is vertical. The grain size in this specimen was 1.4 µm.

The textures in all the specimens are aligned on the basal plane. The texture of specimen 4 is documented in **Figure 3**.

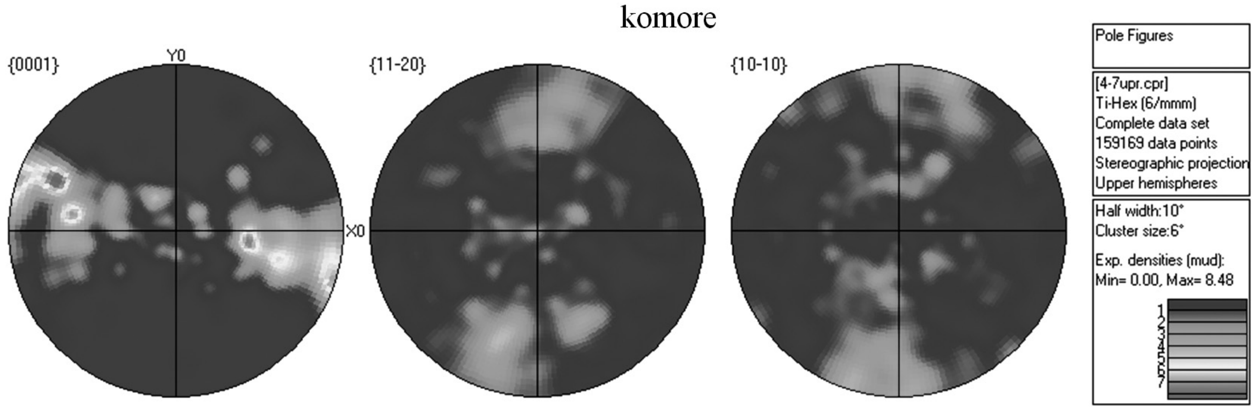

Figure 3: Areal densities of the points
Slika 3: Področja gostote točk

Table 2: Mechanical properties and the average grain size in various states of CP-Ti
Tabela 2: Mehanske lastnosti in povprečna velikost zrn pri različnih stopnjah CP-Ti

Condition	$R_{p0.2}$	R_m	A_g	A_5	Z	KCV	d
	MPa	MPa	%	%	%	J cm^{-2}	μm
Feedstock	354.3	470.4	9.3	32.3	64.2	64.2	23.5
Upon pass 1	620.1	693.5	12.0	26.3	55.7	27.5	1.4

The mechanical properties of the feedstock and of the Ti wire upon a single pass at the die-chamber temperature of 350 °C are listed in **Table 2**. As expected, the yield stress and ultimate strength of the product are higher than those of the feedstock. On the other hand, its contraction and elongation as well as the impact toughness upon the first pass are lower than those of the feedstock.

Figure 4 shows the macrostructure of the product in the die chamber after the processing was interrupted. The dead zones in the transitional region are clearly visible. The traces of deformation can be seen at some points.

Figure 5a shows a micrograph of the undeformed material. The average grain size in this case is 25 μm. The microstructure above the abutment is shown in **Fig-**

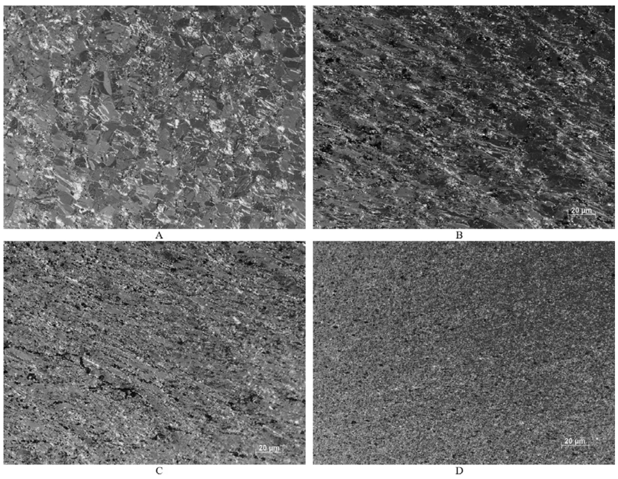

Figure 5: Microstructures in the selected locations within the die chamber. The notation corresponds to that in **Figure 4**.
Slika 5: Mikrostrukture na izbranih lokacijah v komori. Oznake ustrezajo tistim na **sliki 4**.

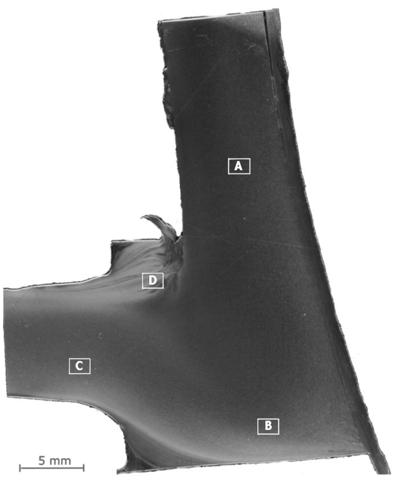

Figure 4: Macrostructure of CP-Ti inside the die chamber
Slika 4: Makrostruktura v notranjosti CP-Ti komore

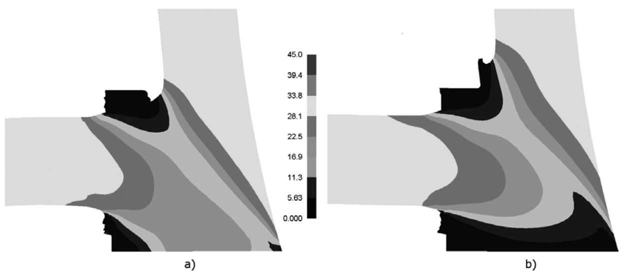

Figure 6: Distribution of the absolute material-flow velocity in mm s^{-1}. Variant: a) with the friction coefficient of 0.2; b) friction coefficient of 0.7.
Slika 6: Razporeditev absolutne hitrosti toka materiala v mm s^{-1}. Varianta: a) koeficient trenja 0,2, b) koeficient trenja 0,7.

ure 5b. In this location, the grains are elongated in the direction at an angle of 45°. The elongation is not uniform; instead, it shows a wave-like pattern. A better alignment of the grains can be seen in **Figure 5c**, which shows a micrograph of the bottom part of the die-exit area. The dead zones exhibit the finest grains without any traces of the flow lines (**Figure 5d**).

3.1 Comparison between mathematical simulation and physical experiment

The coefficient of friction between CP-Ti and the tool material was measured using a non-standard, tribological test arrangement: $\mu = 0.62$. The result is not conclusive, as the conditions of the test cannot perfectly match those of the real-world titanium-forming process.

Two FEM simulations were performed with two different friction-coefficient values (0.2 and 0.7), while keeping the other initial and boundary conditions identical.

A sample result of the calculation of the absolute flow velocity is shown in **Figure 6** for both values of the friction coefficient. Upon comparing the results with an actual specimen (**Figure 4**), it becomes apparent that the value of $\mu = 0.7$ is closer to the actual value. A comparison between the distribution of the zones with the minimum movement of the material calculated by the numerical simulations and that found in the specimen upon interrupting the forming process suggests the same result: the value of $\mu = 0.7$ is more appropriate. The obtained value is very far from the values of the coefficient of friction during the wire drawing with lubrication.[3]

4 CONCLUSIONS

The process trials were performed, whereby the impact of the settings of the CONFORM equipment on the microstructure of the resulting Ti wire was explored. The strongest influence is that of the die-chamber temperature. The smallest average grain size found with the EBSD analysis is 1.4 μm. The limit temperature for achieving this value in an uninterrupted process was 350 °C. The Ti wire produced under these conditions showed a strength of 693 MPa and an impact toughness of 27.5 J cm^{-2}.

The main goal of this study was to describe the continuous extrusion of titanium by means of a mathematical model. The model was constructed in the FEM software DEFORM-3D. The outcome of the simulations is in good agreement with the experimental results. The die chamber of the Conform™ machine was adapted for titanium forming.

The coefficient of friction between titanium and the die-chamber temperature was 0.7. In the further optimisation efforts, the die chamber will be adjusted in order to suppress the dead zones where no movement of the material occurs. This will be first verified with a mathematical simulation and then with additional process trials including multiple passes in order to obtain a nanostructured titanium bar.

Acknowledgments

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