Technological aspects of preparation of nanostructured titanium wire using a CONFORM machine

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Abstract. One of available ways to enhancing the efficiency of production of sections is combining the CONFORM continuous extrusion process and the ECAP method. This paper describes the initial experience with such a combined process deployed in the CONFORM 315i machine, which is equipped with a specially-designed die chamber. Process trials were performed to explore the effects of the set-up of the CONFORM equipment on the resulting microstructure of Ti wire. The feedstock was in the form of CP Ti grade 2 bars of 10 mm diameter. Crucial parameters of the process include the temperature of the die chamber, which was purposefully varied from the initial 500 °C to the final 200 °C, and the cooling stage, which was realized immediately upon the exit from the die chamber. The mean grain size, mechanical properties and thermal stability were measured and studied in the specimens upon forming. The smallest grain size was achieved by two passes at 200 °C.

1. Introduction
In recent fifteen years, numerous SPD processes (Severe Plastic Deformation) were developed. They are used to achieve grain refinement to the nano-scale, with the resulting grain size between 100 and 400 nm. In terms of processing large volumes of material, however, their efficiency is still insufficient for industrial-scale applications. This drawback is eliminated by the CONFORM method which has been known for a long time, being used for continuous industrial-scale production of sections, mostly from aluminium. Through integration with ECAP (Equal Channel Angular Pressing), a continuous process was obtained where feedstock is repeatedly forced through a die by a friction force. This ECAP–CONFORM process was described for the first time in [1]. In those experiments, commercial-purity aluminium wire was extruded using one to four passes. Preparation of ultrafine-grained titanium using this method was reported in [2]. In the study, a 7.2×7.2 mm square wire was made with an accumulated strain of e=6 and subsequently redrawn into a 3 mm-diameter wire (resulting in the total strain of 7.9). Related works [3–5] shows the mechanical properties of Ti wires obtained with the inclusion of additional steps such as cold drawing and special thermomechanical processing.

In the present paper, a description is given of the production of commercial-purity titanium round bars using multiple passes in ECAP-CONFORM equipment. The shape of the die chamber was pre-optimized using computer simulations, the results of which were published in papers [6,7].

2. Experimental
The feedstock consisted of CP Ti grade 2 bars with a diameter of 10 mm. Their chemical composition was measured using Bruker Q4 Tasman optical emission spectrometer and Bruker G8 Galileo gas analyzer as is shown in Tab. 1.
Titanium bars were converted into an “endless” bar of the same diameter as the feedstock using the CONFORM 315i machine. Detailed descriptions of the individual experiments are given in sub-section 2.1. The material’s mechanical properties at the ambient temperature were measured on cylindrical tension test specimens with a gauge length of 25 mm and diameter of 5 mm. In addition, impact toughness tests were conducted using 3×4 mm-section...
specimens. Microhardness was measured using a fully automatic Struers DuraScan 50 microhardness tester and a test load of 9.807 N.

Microstructures of the specimens were observed in JEOL JSM 7400 scanning electron microscope with a field-emission gun (FEG) and a Nordlys EBSD detector (Oxford Instruments). Specimens for EBSD analysis were prepared by ion polishing in JEOL SM-09010 Cross Section Polisher. The conditions of EBSD observation were as follows: 25 kV voltage, working distance of 15.5 mm, 500×500 point lattice and a step size of 0.1 µm. EBSD maps were visualized and edited using HKL Channel 5 software. The grain size was found by the intercept method (by measuring lengths between points of intersection between grain boundaries and a square grid) according to the Czech Standard SN EN ISO 643.

For the purpose of observation in a transmission electron microscope (TEM), thin foils were prepared by final electrolytic thinning in Tenupol 5 device, using the solution of 300 ml CH₃OH + 175 ml 2-butanol + 30 ml HClO₄ at -10 °C and a voltage of 40 V. TEM analysis was performed in the JEOL 200CX instrument with an acceleration voltage of 200 kV. Selective electron diffraction (ED) was employed for qualitative phase analysis.

The effect of deformation on thermal expansion of titanium and the temperature range for recovery were studied using Linseis L75 Platinum horizontal dilatometer with an Al₂O₃ specimen chamber and a pull bar. Changes in temperature were detected using an S-type thermocouple. The specimens were 5 mm-diameter cylinders with the length of approximately 20 mm. Nitrogen (N₂) was used as the protective gas. Heating to 950 °C at the rate of 3 K/min was followed by cooling at 20 K/min to 600 °C. Then the specimen was left to cool to the ambient temperature in air. Recovery processes were monitored in Linseis PT-1600 heat-flux calorimeter equipped with an S-type thermocouple. The measurement was conducted in Ar protective gas at a flow rate of 600 ml/min. The specimens were placed in crucibles of Al₂O₃ with lids. The specimens, having the weight of approximately 41 mg, were cut off from 5 mm-diameter bars.

Table 1 Chemical composition of feedstock

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

2.1 Detailed Description of Process Experiments

In the present paper, results of three different experiments relating to preparation of titanium wire in CONFORM machine are discussed. The key difference between these experiments lies in the temperature of the die chamber during continuous extrusion and in the number of repeated passes through the CONFORM machine. The feedstock had the form of titanium grade 2 bars of 10 mm diameter and 1.5 m length. During the experiment, the output wire was sheared to identical lengths.

In the experiment no. 1, an inductor was used at the inlet in the CONFORM machine for preheating the feedstock. The inductor power was set at 14 kW and gradually reduced to 6, 4 and 2 kW during the experiment. Once the wire was introduced into the machine, the speed of the wheel was reduced from 2 revolutions per minute to 1 rpm. At this speed, the extrusion process continued until completion. At the start of the experiment, the temperature of the die chamber was 500 °C. Immediately after the bite, the temperature was reduced to 350 °C and then to 320 °C. Another process variable monitored was the temperature of the wheel. The total duration of the experiment was 30 minutes.

The experiment no. 2 focused on maximizing the number of repeated passes of the titanium wire through the die chamber of the CONFORM machine. During the experiment, the wheel speed ranged from the initial 2 rpm to 0.3 rpm. The power of the inductor for preheating the wire was set at 14 kW. The variable which was varied in this trial was the
temperature of the die chamber. The process commenced at 500 °C. Once the wire was introduced into the machine, the temperature was reduced to 320 °C. In the subsequent steps, extrusion took place at 300 °C, 280 °C and 250 °C. During the last passes, the die chamber was cooled in a controlled manner at the rate of 2 °C/min. The controlled cooling was accompanied by reducing the induction coil power to 3 kW and by decreasing the speed of the wheel. The decrease in temperature led to higher requirement for the machine's power input and force. In contrast to the first experiment, the temperature of the abutment was found to be elevated over a long time. Although it is not heated directly, its temperature during the experiment was higher than that of the die chamber by up to 50 °C. Specimens of the wire for metallographic observation and mechanical testing were taken after the tenth pass, at the temperature of about 180 °C.

The experiment no. 3 was aimed at achieving the lowest possible forming temperature in an appropriate number of passes of the Ti wire through the CONFORM machine. The friction in the die chamber was reduced by applying industrial lubricant. In this experiment, pre-heating by the induction coil was not used. The speed of the wheel was constant and identical in all passes: 0.5 rpm. The effects of the number of passes at the die chamber temperature of 200 °C upon the resulting mechanical properties and final microstructure were explored. The repeated extrusion process took approximately 100 minutes due to the low circumferential speed. A total of three passes were completed. Upon each of them, samples for testing were taken.

3. Microstructure upon Experimental Processing

The feedstock microstructure contained equiaxed grains with scarce twins (Fig. 1). The microstructure upon deformation consisted of two types of structure: recrystallized equiaxed grains and a small proportion (less than 15 %) of distorted grains divided into subgrains by low-angle boundaries. The feedstock and processed microstructures exhibited identical notable texture. The (1000) planes were aligned with the specimen axis. The experiment in extruding Ti wire by means of the CONFORM equipment was based on varying the key process parameters. They included the speed of the wheel, the die chamber temperature, the application of cooling downstream of the die chamber, and others. The decisive parameter for the entire process is the die chamber temperature. It was varied in a controlled manner from the initial 500 °C to the final value of 320 °C. Specimens were taken to explore the effect of passes at various die chamber temperatures. The microstructure of the specimen processed at the die chamber temperature of 500 °C contained undeformed equiaxed grains. Its grain

![Fig. 1 – Feedstock microstructure (length of scale is 20 µm)](image1)

![Fig. 2 – Microstructure of CP-Ti upon a single CONFORM pass (length of scale is 5 µm)](image2)
size pattern was bimodal. The average grain size was 1.9 µm. Neither the small nor the large grains exhibited distorted sub-structure. The die chamber temperature of 450 °C was sufficient for the microstructure to recover/recrystallize. No effects of cooling were detected. The recovery/recrystallization and potential grain growth finished before the specimen was cooled. However, the workpiece extruded at 400 °C showed some initial changes, as it contained a small amount (10 – 15%) of deformed unrecrystallized grains. Workpieces exiting the die chamber were cooled with water. In them, the deformed grains with a size of no more than 5×10 µm were divided into subgrains by low-angle boundaries. The average grain size was 1.9 µm.

Figure 2 shows the workpiece microstructure after extrusion at 350 °C and water cooling. It consists of slightly elongated deformed grains which, however, lack the above-mentioned substructure. EBSD analysis focused on the centre of the circular cross-section of the extruded product. The analysed surface lies in a 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.

In the second process experiment, the microstructure after ten passes was heavily distorted and at the limit of the imaging capability of the EBSD method. The crystallographic indices could not be identified. The estimated grain size was 0.75 µm.

In the third process experiment, the pre-heating of Ti bars prior to their feeding to the CONFORM machine was eliminated. The chamber temperature was set to 200 °C and the wheel speed was 0.5 ms⁻¹. In this experiment, three passes of Ti wire through the CONFORM machine were used. As the amount of strain introduced into the material was substantial, the EBSD method could not be used. All three specimens were examined using TEM.

3.1 TEM Analysis of Microstructure
Samples were taken in longitudinal and transverse directions from the Ti wire upon each pass and used for making foils for transmission electron microscopic observation. The longitudinal cross sections are documented. In this direction, the change in grain size with the introduced strain should be critically assessed.

Fig. 3 Region with grains with deformation texture within Ti wire upon the first pass at 200 °C.

Fig. 4 General view of Ti wire substructure upon the second pass at 200 °C.

After the first CONFORM pass at the chamber temperature of 200 °C, the microstructure on the longitudinal section consisted of polyhedral grains with increased dislocation density, distorted grains and even disc-shaped grains. The deformation microstructure is shown in Fig. 3.
The nature of the substructure after the second pass is very similar to the condition seen on the transverse cross-section. The Figure 4 shows very fine-grained polyhedral microstructure. The mean grain size found by measurement was $d \sim 310 \pm 30\,\text{nm}$. In contrast to the previous specimen, whose longitudinal cross section showed areas with grains containing deformation texture or even disc-shaped grains, no such patterns were found here.

Figure 5 characterizes the substructure on the longitudinal section of the specimen upon three CONFORM passes. The nature of the substructure is very similar to the condition seen on the transverse cross-section. Polyhedral grains can be seen, the mean size of which is $d \sim 420 \pm 30\,\text{nm}$. Again, there were areas with low dislocation density in the substructure, as well as regions with higher dislocation densities.

4 Mechanical Properties of Extruded Titanium Wires

Mechanical properties of the feedstock and of the Ti wire extruded in a single pass at 350 °C (experiment no. 1) are given 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, the reduction of area and elongation in the products, as well as the impact toughness upon the first pass are lower than those of the feedstock. Peculiar results were found after the third pass at 200 °C (experiment no. 3). The ultimate strength was equal to the level upon a single pass. Yield strength, however, was higher by 17 MPa. Elongation declined, whereas reduction of area remained to that of the feedstock.

| Table 2 Mechanical properties and average grain sizes in various states of CP-Ti |
|-------------------------------------|----------------|---------|-----|---------|--------|
|                                     | YS $0.2$ & YS Rp0.2 | UTS      | $A_5$ | RA      | KCV    | $d$    |
|                                     | MPa             | MPa     | %    | %       | Jcm$^{-2}$ | μm    |
| Feedstock                           | 354             | 470     | 32.3 | 64.2    | 5.39    |
| Single pass (320°C)                 | 620             | 694     | 26.3 | 55.7    | 27.5    | 1.4    |
| Three passes (200 °C)              | 637             | 698     | 17.8 | 66.2    | -       | 0.42   |

As part exploration of the resulting mechanical properties, microhardness was measured using an automatic microhardness tester. In every specimen, a total of 197 indentations were made using the nominal load of 9.807 N. Microhardness testing was employed to map the uniformity of strain within specimens from the experiment no. 3. The variation in hardness across the feedstock’s cross section was up to 71 HV. By contrast, the distribution of hardness in specimens after three passes was substantially more uniform. The variation in hardness on the cross-section was a mere 33 HV. Hardness distributions within the feedstock and within specimens taken upon each pass are shown in Fig. 6.
5 Results of Thermal Analysis

Results of dilatometer measurements were processed by means of the data evaluation module of Linseis software (Fig. 7). The specimen upon one pass through the CONFORM machine at 350 °C showed reduced expansion, which can be explained by the annihilation of dislocations and elimination of the lattice stress. This was not observed in the annealed feedstock, which received no deformation. Effects of phase transformations can thus be ruled out. The calculated difference between the changes in lengths of the feedstock and the processed specimen suggests that the temperature range, in which substantial change in length is due to recovery, is 432 °C – 576 °C.
The same method was used for measuring the variation in thermal expansion for the specimen upon three passes at 200 °C. The lower limit of the interval, in which the deformed Ti wire begins to recover, is shifted towards notably lower temperatures. Figure 8 reveals that the interval is 300-560 °C.

Results of the measurement in a differential scanning calorimeter (DSC) conducted on a specimen upon a single pass at 350 °C are shown in Fig. 9. Beyond 300 °C, the temperature rise in both specimens is in accordance with the thermal schedule (constant heating rate of 10 K/min). Despite that, the curves for both specimens ("conformed" and annealed in the DSC calorimeter) show differences. As the calculation shows, there is a steeper increase in stress in the processed specimen at temperatures above 440 °C (see Fig. 9). Apparently, an exothermic reaction takes place, generating heat. This region matches the recovery zone identified by the dilatometer measurement. The higher onset temperature found in the DSC may be due to the higher heating rate, the specimen size, and the resulting response. It may be attributed to the release of heat during stress relaxation in the recovery process. In the extruded specimens, the recrystallization peak was detected at lower temperatures, which corresponds to the theoretical and empirical knowledge of the dependence of recrystallization on temperature and strain. In the specimen extruded in three passes in the CONFORM machine at 200 °C, the DSC method showed a more notable decrease in the temperature for the onset of the exothermic reaction, which was approx. 320 °C, as shown in Fig. 10.
The recrystallization temperature in the undeformed specimen was $T_{rx} = 615 \, ^\circ C$. In the “conformed” specimen upon a single pass, it was 594 °C. A decrease in the recrystallization temperature down to 527 °C in a CP-Ti specimen upon eight ECAP passes has been reported in literature [8].

6 Conclusion
Process trials were performed to explore the impact of the CONFORM set-up on the microstructure of the resulting Ti wire. The strongest influence is that of the die chamber temperature. The smallest mean grain size of 310 nm, as measured by means of EBSD and TEM, was achieved by the second pass at the die chamber temperature of 200 °C. The strength of the Ti wire after the third pass was as high as 697 MPa. However, the mean grain size in this product rose to 420 nm due to recrystallization. Yet, its strength is equal to that of the wire obtained upon a single pass at 320 °C in the experiment no. 1, where the mean grain size was as large as 1.4 µm.

Thermal stability of the CONFORM-processed CP-Ti was explored using thermal analysis and was found at 450 °C. This temperature limit, however, is reduced by the amount of energy introduced.

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