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Hydrostatic extrusion of Al coated titanium obtained by the magnetron technique

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ABSTRACT

Purpose: The paper synthetically presents the ideas of hydrostatic extrusion of metals in order to obtain strong refinement of their structure. It seems that modification of extruded material and the surfaces of dies may be one of the methods for limiting these adverse phenomena. The paper describes the role of lubricating the Al layer during the titanium extrusion process.

Design/methodology/approach: Studies concerning this technique are conducted at The Polish Academy of Sciences Institute of High Pressure Physics. Due to strong plastic deformations (SPD), the cumulative hydrostatic extrusion (HE) process must be used, i.e. the process of step-by-step extrusion with low deformation ratio for each step, the deformations created in each step accumulate and result in strong cumulative deformation.

Findings: This is because the available working pressures of extrusion presses are limited, thus limiting the maximum deformation ratio available in a single extrusion pass. This limitation is additionally sharpened by higher strength of material deformed in cold state or at low temperatures.

Research limitations/implications: Due to tribological conditions existing between the extruding tool (a die) and the flowing material, the die becomes worn quickly and the finishing quality (roughness) of the extruded material surface deteriorates.

Practical implications: As a result, the extruding pressure increases which has a negative impact, the machine and tooling load becomes higher and the phenomenon of abrupt outflow of the material being extruded from the extruding tool occurs, causing poor tolerances of lateral dimensions of the product.

Originality/value: The process of depositing Al coatings on cylindrical surfaces of titanium material being extruded using the PA PVD technique was presented in detail. Also, the paper describes examinations of Al coating properties and structure of Grade 3 titanium in subsequent steps of hydrostatic extrusion process.

Keywords: Hydrostatic extrusion; Al coating; PVD; Titanium; Grade 3; Calotest; Spherical microsection; Vickers hardness; DSI method

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MANUFACTURING AND PROCESSING

1. Introduction

Pure titanium is a good material for medical applications, such as dental and orthopaedic implants. It has a thermal conductivity several times lower than conventional prosthetic materials, a high degree of hardness, mechanical strength and durability. In addition, it does not cause allergic reactions and is resistant to corrosion. In prosthetics, titanium alloys with Al, Nb and Ta or alloys of Ti-Al-V are used. The disadvantage of these is the toxic alloying additives used in order to increase strength and implant capabilities.

The use of pure titanium eliminates this problem, provided that the material will have adequate durability. Achievement of such is possible by the use of severe plastic deformations generated by means of hydrostatic extrusion [1-9].

Fragmentation of titanium grains to a nanometric or ultra fine level requires the generation in its microstructure of severe plastic deformations at low temperature or at ambient temperature [2].

Severe plastic deformations (SPD) entail cumulative hydrostatic extrusion (HE), i.e. sequential extrusion with minor deformation steps, which added up lead to a cumulative severe deformation. This necessity is due to limitations in the available operating pressure for extrusion presses, which limit the available degree of deformation per unit of extrusion operation. This is further compounded by the higher strength of the material deformed cold or at low temperature. Due to the tribological conditions between the shaping tool (mould) and the flowing material there is accelerated wear on the tool and an increase in the surface roughness of the extruded product. As the result, there is an unfavourable increase in extrusion pressure, meaning an increased load on the installation and tooling, and the phenomenon of an uneven flow of extruded material from the mould, resulting in the product failing to maintain high tolerance in lateral dimensions.

Reduction or elimination of the above mentioned phenomena during hydrostatic extrusion with severe deformations makes it possible to stabilize the process and produce titanium products/semi-finished products in a fully controlled manner within constant process parameters.

One of the ways to reduce adverse phenomena may be the use of additional materials obtained by means of PVD technologies. Coatings are applied to the surface of the extruded titanium and the mould's openings [12].

2. Research methodology and discussion

The process of hydrostatic extrusion is carried out on high-pressure presses made and designed in IWC PAN and operating at pressures of up to 2500 MPa.

Al coatings on titanium batches are produced at the Koszalin University of Technology, using the Pulsed Magnetron Sputtering technique.

Metallographic tests and Vickers micro hardness and Martens HM indentation hardness H_{IT} measurements were carried out at the University's Institute of Precision Mechanics using a Haneman micro hardness meter (Neopfot2 attachment), and the CSEM company's MHT using the DSI (Depth Sensing Indentation) method. Metallographic tests were carried out using a Kulotester (Calotest) test bench with a 30 mm ball [10,11,13].

3. Depositing an Al coating on Titanium - MS PVD technology

Prior to production of the coating, the titanium batches were washed in an alkaline solution assisted by ultrasonics, and then rinsed in distilled and demineralised water and dried in a stream of warm air. The thus prepared titanium batches were placed on the satellites of a planetary system for movement of substrate in the vacuum chamber of an apparatus for producing coatings. After pumping out the vacuum chamber to a pressure below 2×10^{-5} hPa the titanium batches were heated using a radiation heater to a temperature of 150°C, and then ion purified in an argon glow discharge plasma with a discharge power density of less than 0.5 W/cm^2. In the next step, a coating of Al was produced, moving the titanium batches between two rectangular magnetrons aligned opposite each with an active target area of 90 × 180 mm. The targets were made of A0 (99.7%) class aluminium and the operating gas used was argon with a purity of 99.995%. Impulse power supplies were used both to power the magnetrons and for polarization of the substrate. The sputtering power of each magnetron was 3 kW and the substrate polarization voltage was 100 V. The Al coatings prepared had thickness of 10-15 microns, depending on requirements.

4. Hydrostatic extrusion

Table 1 shows the experimental data of the CP Ti grade 3 titanium hydrostatic extrusion process. A cumulative titanium extrusion process was carried out using an applied Al coating and without it, using conventional lubrication based on Cu-based lubricants. In both cases the material was extruded twice, with a total real deformation of ε ~ 1.

For the experiments conducted using an Al coating on titanium batches, significantly lower extrusion pressures were observed, namely ~ 15% in the first extrusion, and ~ 25% for the second extrusion respectively. Figure 1 shows the pressure characteristics of the hydrostatic titanium extrusion process with an Al layer (waveforms indicated by a dashed line) and without an Al layer (continuous line).

Apart from the marked difference in the extrusion pressures, the presented waveforms also show much better stability of the hydrostatic extrusion process when using Al layers. The extrusion processes take place at a constant pressure and no oscillations are observed in the course of the deformation. In the samples lubricated conventionally, pressure undergoes a marked change. In the first extrusion process, observed was its steady increase associated with titanium wear on the mould, and in the second, fluctuations due to heterogeneous lubricating conditions along the length of the extruded sample. In the second Al coated sample extrusion process, a slight increase in extrusion pressure was observed (Fig. 1) associated with consumption of the Al covering. The change in the thickness of the Al coating g is shown in the diagram (Fig. 4).

Figure 2 shows the dependence of extrusion pressure as a function of actual deformation for a single extrusion of CP Ti grade 3 titanium using an Al layer and for conventional lubrication. Regardless of the value of the applied deformation in the range ε ~ 2-4, a marked impact on the application of an Al layer in the form of a reduction of extrusion pressure is observed. The differences are about 200 MPa, which for example for a sample extruded with a deformation of ε = 4 constitutes ~ 20%.

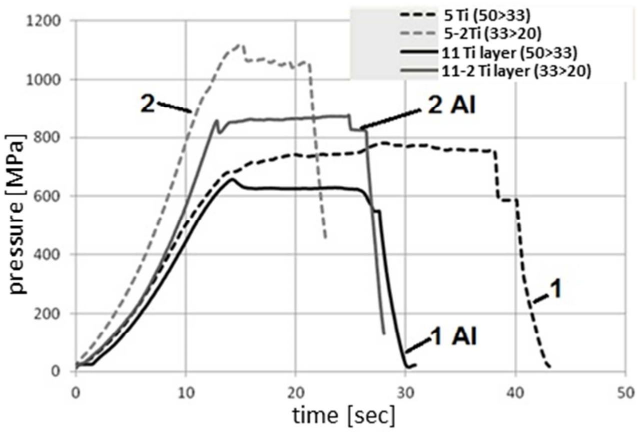

Fig. 1. Pressure characteristics of the hydrostatic CP Ti Grade 3 titanium extrusion process

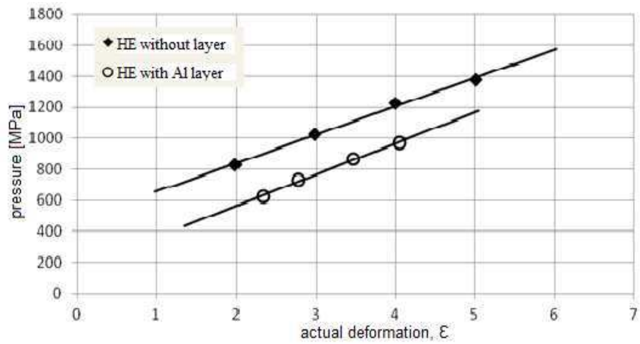

Fig. 2. Pressure dependence as a function of actual deformation for single CP Ti Grade 3 titanium extrusion processes using an Al layer and conventional lubricants

Table 1.
Process parameters for the hydrostatic extrusion of CP Ti grade 3 titanium

	Initial diameter d_o [mm]	Final diameter d_f [mm]	Reduction R (d_0^2/d_f^2)	Cumulative reduction a R_{sum}	Actual deformation ε=lnR	Cumulative deformation ε_{cum}	Extrusion pressure p_{HE} [MPa]
				Ti without layer			
5 Ti	50	32.8	2.32	2.32	0.84	0.84	723
5-2 Ti	32.8	19.78	2.75	6.39	1.01	1.85	1066
				Ti layer			
11 Ti	50.35	32.9	2.34	2.34	0.85	0.85	627
11-2 Ti	32.9	19.96	2.72	6.36	1.00	1.85	853

5. Microstructural tests of Al coating

The research materials were Al-coated titanium cylinders. In the analyzed hydrostatic extrusion process a reduction of the original cylinder diameter from 50 mm to 10 mm was obtained. The extruded original 50 mm Al coated titanium cylinder, and a diagram of the diameter reduction in successive extrusion stages are shown in Fig. 3 and Fig. 4.

Fig. 3. The original Al coated Grade 3 titanium cylinder with a diameter of 50 mm

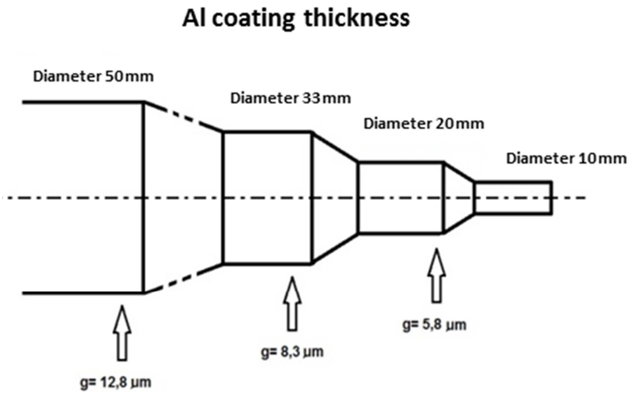

Fig. 4. Diagram of the titanium cylinder after undergoing the hydrostatic extrusion process, titanium with an Al coating having a thickness g; first pressing φ(phi) – 33 mm, second pressing φ(phi) -– 20 mm, and third φ(phi) – 10 mm

6. Structure of the Al coating

The structure and thickness of the Al coating following successive stages of extrusion was disclosed and analyzed on spherical metallographic sections obtained on the lateral surface of cylinders. Figures 5 and 6 show the structure of the Al coating after the second and third stage of extrusion - the diameters 33 mm and 20 mm. The Al coating thickness in this case decreased by o ~ 3.5 microns. Reduction in the thickness of the Al coating in the extrusion process is also accompanied by a change in surface morphology – Figures 5b and 6b. The occurring micro-pores formed by crimping of the initial highly developed surface structure (a surface formed in the cutting rolling process), undergo considerable elongation. The pores constitute a natural convenient way of storing the lubricants used in the extrusion process.

7. HV 0.01 and HV 0.5 hardness tests

The Vickers HV 0.01 coating surface micro-hardness tests following successive stages of extrusion do not show any significant difference. The surface hardness of the aerologic system of the Al/Ti-Grade 3 coating is 165 HV0.01. The lack of any change in the surface hardness of the system following successive stages of extrusion can be attributed to the formation of a high extrusion surface temperature exceeding the re-crystallization temperature for the Al coating. For aluminium (Al) of a high purity, the re-crystallization temperature is very low and amounts to 0.1-0.2 T_{topn} ($T_{Al\text{-}topn}$= 660°C). On this basis, surface temperature of extruded titanium can be estimated and it can be concluded that it exceeded the limit of 132°C. Tests of micro-hardness of the coating on titanium conducted on a spherical cross-section make it possible to define the nature of their change as a function of depth from the surface.

Figure 7 shows a series of micro-hardness measuring imprints and the structure of the Al coating on the surface of a spherical metallographic section The maintained symmetry of hardness of imprints proves that the distortion of measurement data associated with the curve of a spherical metallographic section is insignificant. The profile of change in hardness as a function of distance from the surface (Fig. 8) in a given measurement point was obtained using the geometric dependencies of an elliptical spherical metallographic section on the surface of the cylinder. The distance of the tested point (hardness imprint) from the cylindrical surface and the cylinder lying on the major axis of the ellipse on the spherical metallographic section describes the relationship (1).

$$x = \sqrt{\mathrm{R}^2 - \mathrm{d}_1^2} + \sqrt{R^2 - d_2^2} \qquad (1)$$

Where R – is the radius of the Kulotester ball, d_1 – is half the length of major axis of the ellipse, and d_2 – the distance of the point on the major axis to the centre of the ellipse.

Figure 8 shows the profile of changes in hardness in the cross-section of the Al coating and titanium after the first and second extrusion.

a) b)

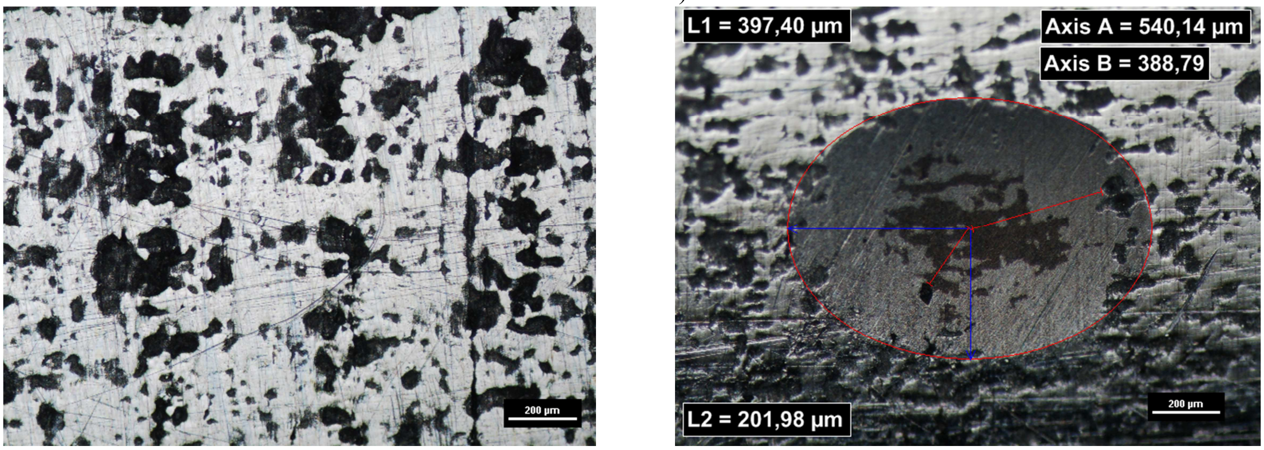

Fig. 5. Cylinder 33 mm, Al coating thickness 8.3 microns, depth 9.7 microns 33 mm

a) b)

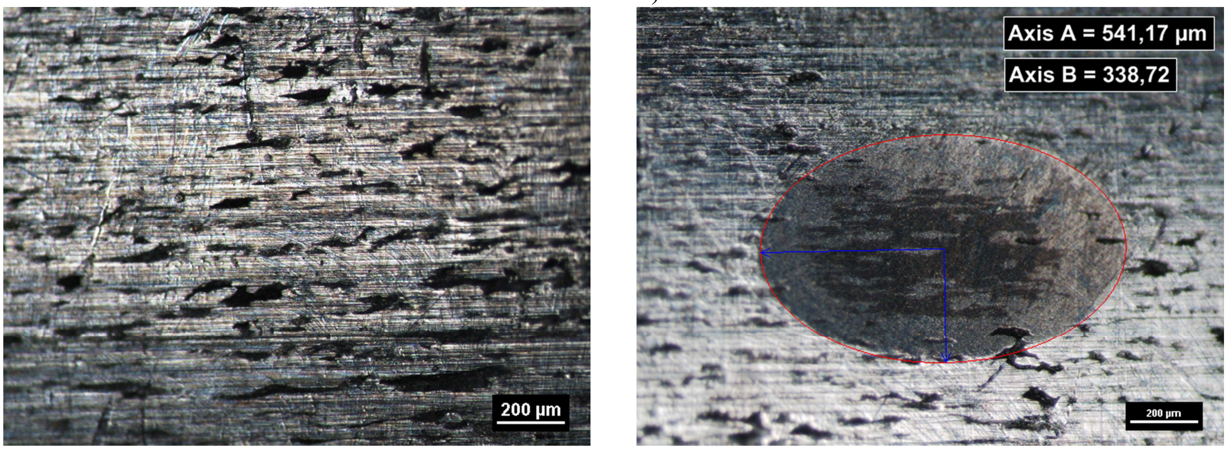

Fig. 6. Cylinder 20 mm, Al coating thickness 5.8, depth of metallographic section 9.7 microns 20 m

a) b)

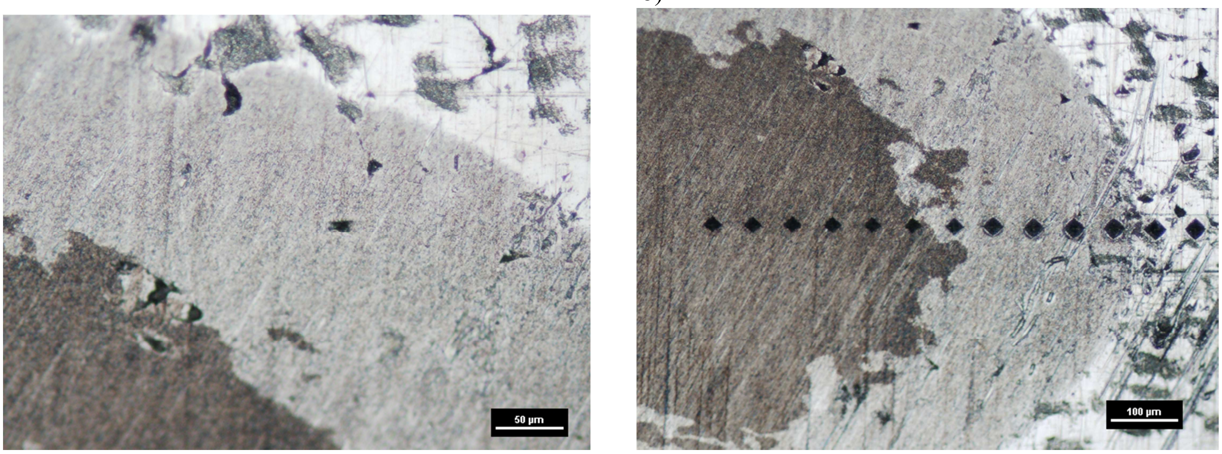

Fig. 7. Al coating structure disclosed on a spherical metallographic section, cylinder 33 mm

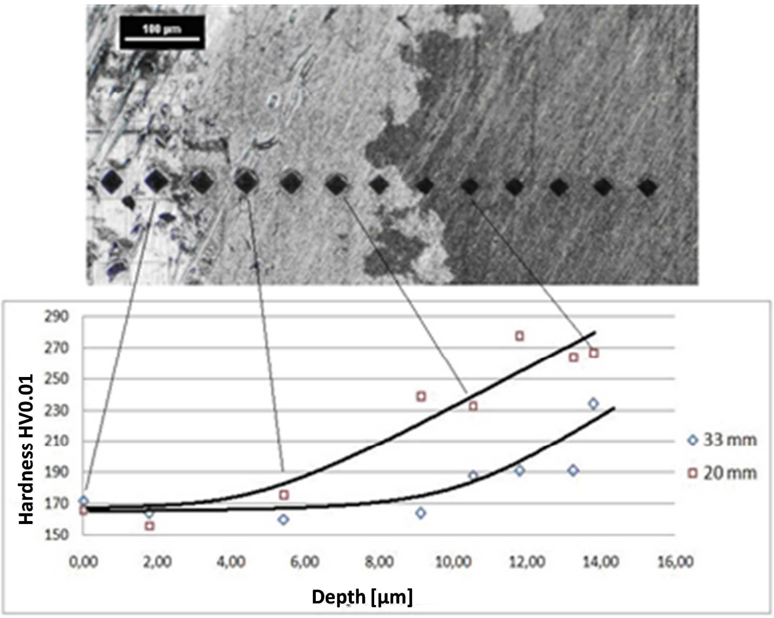

Fig. 8. Profile of the changes in hardness in the surface layer of Grade 3 Titanium covered with an Al coating after the first and second stage of hydrostatic extrusion

Analysis of hardness profile data reveals the existence of an area on the deformed titanium surface with a lower hardness relative to the core. Its thickness in both cases is ~ 14 microns. The presence of this area is associated with the presence of the Al coating and re-crystallization processes caused by the heat of the extrusion process.

HV 0.5 hardness measurements of the core of the Grade 3 extruded titanium (Fig. 9) show the expected increase in the value from 235 HV0.5 for the strand material to 304 HV0.5 for the 10 mm diameter cylinder, amounting to ~ 30%.

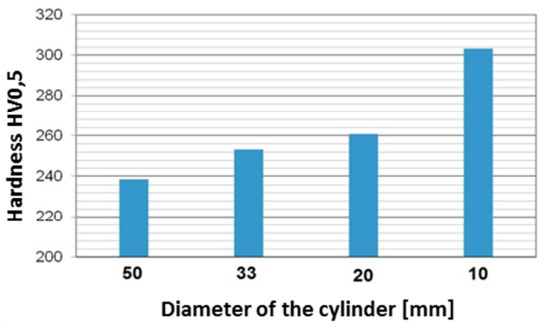

Fig. 9. Core hardness of hydrostatically extruded Grade 3 titanium

8. DSI tests

The measurements carried out on samples of titanium for the maximum loading force were F_{max}=1 N and a load speed of 2000 mN/min. The pause in the operation of maximum force F_{max} was 15s. 10 measurements were taken for each possibility.

Table 2 lists the average values of indentation hardness H_{IT} and Martens hardness HM obtained for Grade 3 titanium following successive stages of hydrostatic extrusion.

The tests related to measurements using DSI methodology contained in this paper [14] for metals show the existence of a correlation between Martens hardness HM and tensile strength Rm at the level r=0.9297 adopting the form of a linear equation 2.

$$Rm= 0.3HM+ 69 \tag{2}$$

On this basis, with the data of Table 2, the tensile strength of titanium Rm following successive stages of extrusion was estimated and the is shown in Figure 10.

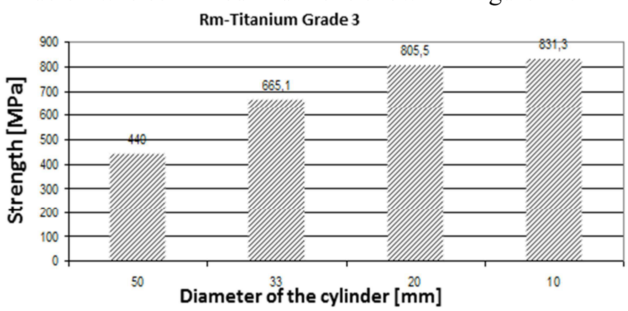

Fig. 10. Tensile strength of hydrostatically extruded Grade 3 titanium

Table 2.
Indentation hardness H_{IT} and Martens hardness HM values obtained for titanium grade 3 after hydrostatic extrusion

	Ti 33	Ti 20	Ti 10
$H_{IT,}$ [MPa]	2674	2855	2973
Standard deflection	44	85	73
HM, [MPa]	1987	2455	2541
Standard deflection	28	63	55

9. Conclusions

Use of an Al coating obtained by means of PVD-Arc technology, with a thickness of 12 microns on the initial surface of an extruded Grade 3 titanium cylinder with a diameter of 50 mm reduces.

In successive stages of extrusion, the Al coating is plastically deformed together with the extruded material and its thickness decreases following successive stages of extrusion.

The surface porosity of the Al coating creates natural lubricant pits reducing the friction coefficients in the centre of the mould's openings.

Hardness tests, performed with DSI method, on the core of extruded titanium show the expected increase in the titanium's values and tensile strength.

Additional information

Selected issues related to this paper are planned to be presented at the 22nd Winter International Scientific Conference on Achievements in Mechanical and Materials Engineering Winter-AMME'2015 in the framework of the Bidisciplinary Occasional Scientific Session BOSS'2015 celebrating the 10th anniversary of the foundation of the Association of Computational Materials Science and Surface Engineering and the World Academy of Materials and Manufacturing Engineering and of the foundation of the Worldwide Journal of Achievements in Materials and Manufacturing Engineering.

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