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Assessment of the properties and structure of porous titanium samples via magnetic pulse compaction

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ABSTRACT

In recent decades, intensive research and effective efforts have been made to apply titanium and titanium alloys in mechanical engineering, the chemical industry, medicine, and other fields. Powdered materials based on titanium play a significant role in this process. Titanium is well suited for various technological applications due to its combination of high strength, low specific weight, and high corrosion resistance. Tubular and ring-shaped products made from titanium powder occupy a certain niche. This study aims to investigate the possibility of producing single-layer and multi-layer tubular samples from titanium powder using the magnetic pulse sintering method. The materials used for the research were titanium powders obtained by grinding titanium sponges with particle sizes ranging from 80 to 250 µm. The microstructure of the obtained samples showed the potential to produce tubular and ring-shaped products with porosity ranging from 30% to 50%. It was found that the outer surface of the single-layer tubular samples is more porous and has lower hardness than the inner surface, which can be explained by the use of a low-melting material applied to the inner surface of the shell during magnetic pulse compaction. When producing a two-layer sample, higher strength can be achieved while maintaining permeability.

1. Introduction

In recent decades, intensive research has been conducted on the effective application of titanium and titanium alloys in the automotive, military, and chemical industries, as well as in medicine [1–7]. Environmental and health protection issues, industrial wastewater treatment, and implant manufacturing are becoming increasingly important. In this context, titanium-based powder materials, including various powder formulations, play a crucial role [4,6].

Titanium powders are well suited for forming and sintering, and the products made from them, which can be given quite complex shapes during manufacturing, retain all the advantages of compact metals while simultaneously acquiring new beneficial properties [6–8].

Compared to other porous substances, titanium powder materials offer several advantages: low specific weight, high specific strength, corrosion resistance, and biological inertness [7]. Furthermore, they can be welded and machined on cutting tools. Due to their unique chemical inertness, they have great potential in medical technology, microbiology, as well as the food and pharmaceutical industries [2,3]. In medicine, porous titanium provides excellent bonding of elements to meet the mechanical requirements of bone substitutes. It is recommended that pore sizes fall within the range of 200–500 µm [6]. Porous filtering products for medical applications, such as aerators and liquid spray devices, are typically made in the form of tubes, rings, or cups [5]. Porous permeable elements made from powder materials are also frequently used in the production of evaporative system filters. For these purposes, titanium powders and titanium powder composites such as Ti-Al and Ti-Al-Mn are especially suitable [3–4]. These materials exhibit high corrosion resistance, ductility, thermal stability, mechanical strength, and good processability.

The properties of products made from titanium powders largely depend on the pressing process. The radial pressing scheme is the most optimal method for a wide range of powder materials, particularly for thin-walled and highly porous products [8]. One of the methods for producing tubular elements is the magnetic pulse compaction (MPC) method [9–13].

MPC methods occupy an intermediate position between static and high-speed methods in terms of impact rate. The relatively slow, increasing, and then decreasing impulse of the external load does not cause the formation of shock waves in the compacting material. At the same time, the relatively high speed of the medium allows for the implementation of an inertial compaction mechanism, resulting in increased pressure on the material [10]. These factors determine the high potential of MPC methods and, consequently, the importance of their theoretical development and applied research. Some studies focus on describing individual aspects of these processes, such as the properties of powder media under load [12], the generation of strong pulse magnetic fields [11], and the technologies of magnetic pulse processing of metals [13]. MPC is particularly useful for manufacturing small batches of products. The method is simple to implement, especially for parts with high porosity.

2. Materials and methods

Titanium powders used in this study were obtained by milling titanium sponge, a product of the calcium hydride reduction of titanium oxide. For the experiments, titanium powder of the 80–250 µm fraction was used, containing an additional 3.5% Si and 0.4% Fe. Three main pressing schemes (Fig. 1), which are discussed in detail in [13], were tested for sample pressing in this study.

The pressing was carried out using a 1 mm thick copper shell, which had to be removed before the final sintering according to the technology described in [14]. To facilitate the process, a layer of low-melting polymer material was applied to the inner surface of the shell, which was removed before sintering by heating the material to 200 °C.

The samples in the form of single-layer and multi-layer tubes were made from titanium powder by MPC using a radial compression scheme (Fig. 2a). In this case, single-layer (Fig. 2b, sample O) and two-layer samples (Fig. 2c, sample M) were produced. The dimensions of the samples are provided in Table 1. The blanks were pressed at a pressure of 400 MPa. Sintering was performed in a vacuum oven at 1240 °C for four hours, under a vacuum of 10^{-4} mm Hg. The workpieces were previously heated to 200 °C to remove

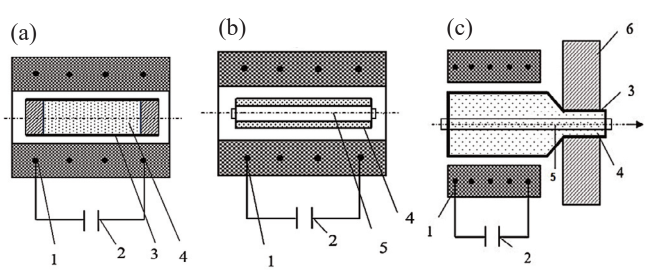


Fig. 1. Pressing schemes using the MPC method: rod-shaped parts (a), tubular shape (b), samples with a length-to-diameter ratio greater than 5 (c). 1 – coil (working tool), 2 – capacitor battery, 3 – shell, 4 – powder, 5 – mandrel, 6 – die.

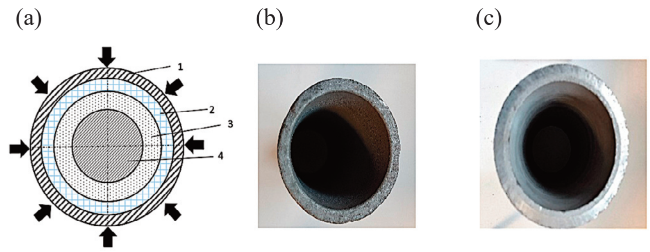


Fig. 2. Schematic diagram of radial MPC of powder: 1 – copper shell, 2 – polymer interlayer, 3 – powder, 4 – mandrel (a). The end sections of single-layer (outer ring O) (b) and two-layer (inner ring M) (c) samples.

Table 1. Geometric dimensions of titanium powder samples after sintering

Sample	O single-layer	M two-layer
D, mm	91	116
L, mm	207	77
t, mm	5	10

D – specimen diameter, *L* – specimen length, *t* – specimen thickness

the electrically conductive shell. Two-layer samples (M) were obtained by pressing an additional layer of powder onto an already sintered tubular workpiece and performing joint sintering using the same technology.

The MPC single-layer samples (O) were fabricated using a magnetic pulse machine with a stored energy value of 20 kJ [14]. Annealed copper shells with a thickness of 1.5 mm and a length of 120 mm were employed as Cu containers with an internal thickness of 1.0 mm. The sintering process was carried out in two stages: initial preheating to 200 °C, followed by the removal of the shell, and final sintering under a vacuum of 10^{-4} mm Hg at a temperature of 1100 °C.

3. Results and discussion

The single-layer sample O microstructure is presented in Fig. 3a, with the chemical composition measurements of the select regions provided in Fig. 3b.

The structure and properties of the two-layer sample M are presented in Figs 4–6. The results of this study demonstrate that the outer surface exhibits increased porosity and a loose microstructure, while the inner surface of the two-layer sample, produced by double pressing, shows a uniform and denser structure.

The microstructure of sample M with indentations is presented in Fig. 7. The hardness measurements indicate that the hardness of the outer layer (Fig. 7a) is significantly lower than that of the inner layer (Fig. 7b).

To measure the air permeability of porous samples, a KM-8 cathetometer and a laboratory air flow meter with the measurement range of 0.5–6 L/min were used.

In this experiment, the dependence of airflow through the porous wall of a pipe section was determined as a function of pressure. The results for two porous pipe samples are shown in Fig. 8 and in Table 2.

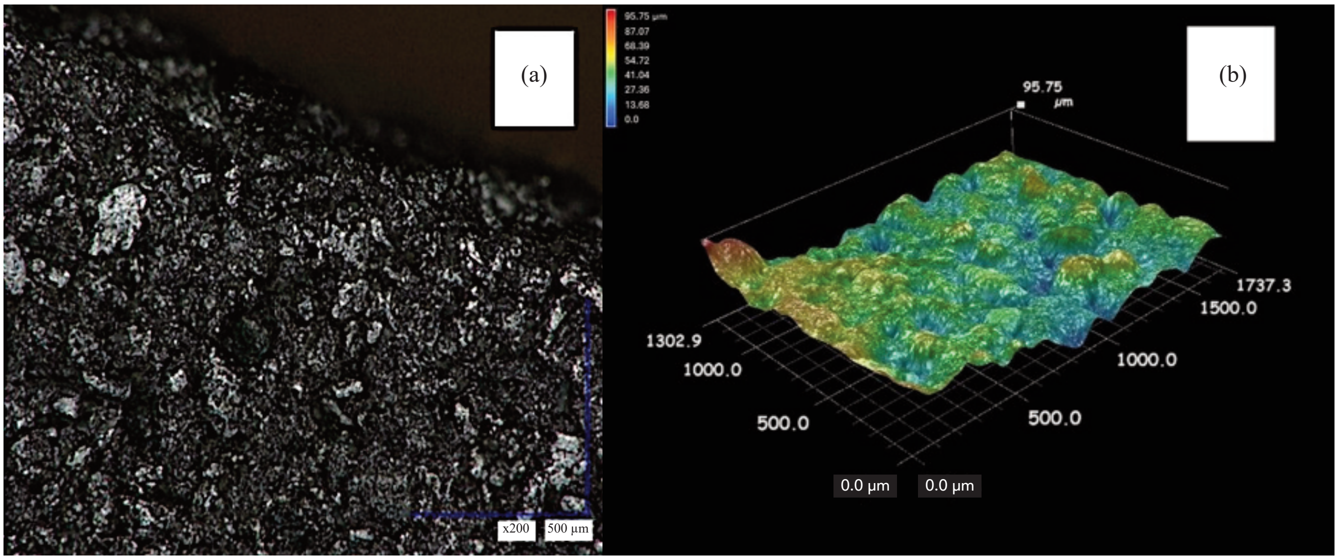


Fig. 3. SEM micrograph of the individual regions of the studied sample O (a) and surface macrostructure (b).

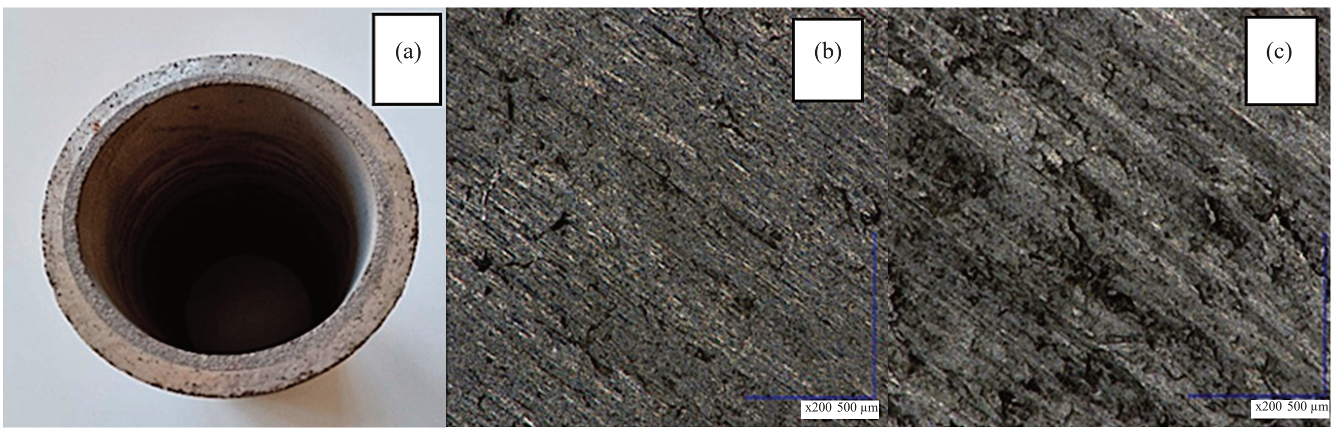


Fig. 4. Overall view of the two-layer titanium powder tube sample M (a) and the microstructure of the outer layer (b) and inner layer (c).

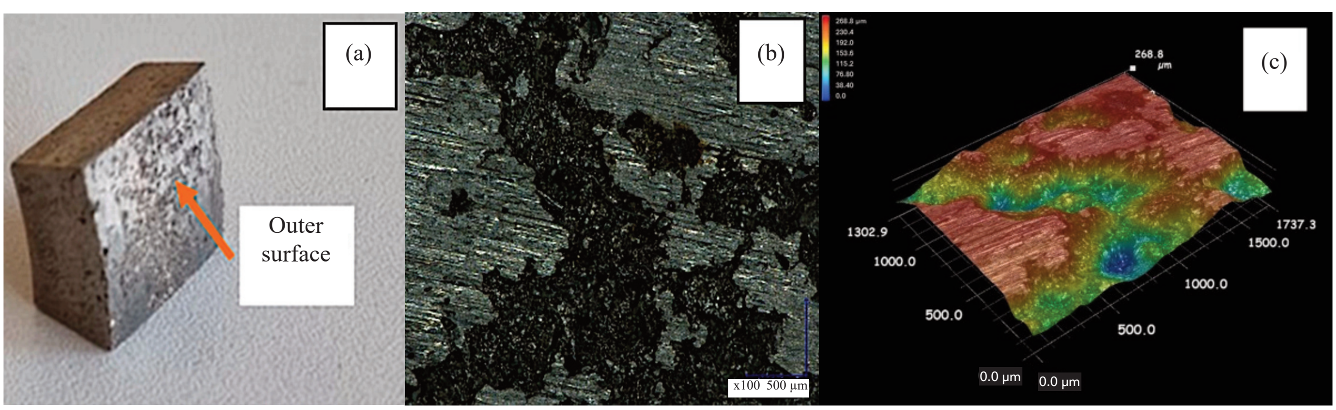


Fig. 5. Overall view of the face of the two-layer titanium powder tube sample M (a) and the microstructure of the outer layer (b) and macrostructure of the surface (c).

The graphs indicate that the air movement in both samples O and M follows Darcy's law, as expected for low flow rates of the filtering medium. The obtained data can be used to evaluate the air permeability of the samples: $Q/\Delta p$, where Q is the air flow rate and Δp is the pressure drop across the sample wall. For multi-layer samples, a more appropriate measure is the linear air permeability, expressed as $Q/\Delta p \cdot L$, which, however, characterizes the product rather than the material itself. For comparison of the samples, the average porosity is also used: $\Pi = 1 - \rho_1/\rho_2$, where ρ_1 is the effective density of the sample and ρ_2 is the density of the alloy from which the sample is made. The results are presented in Table 2.

As shown in Table 2, the linear permeability of sample M is two orders of magnitude higher than that of sample O.

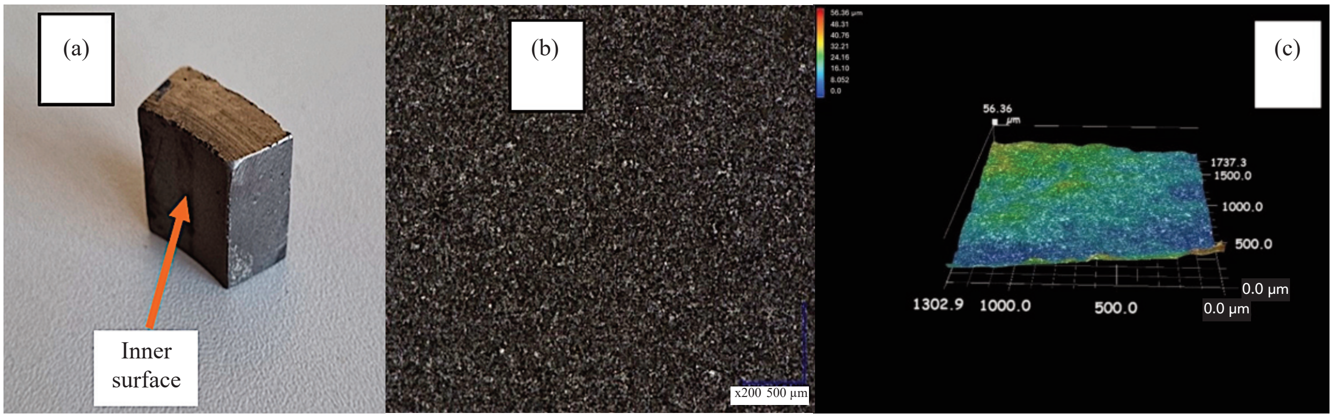
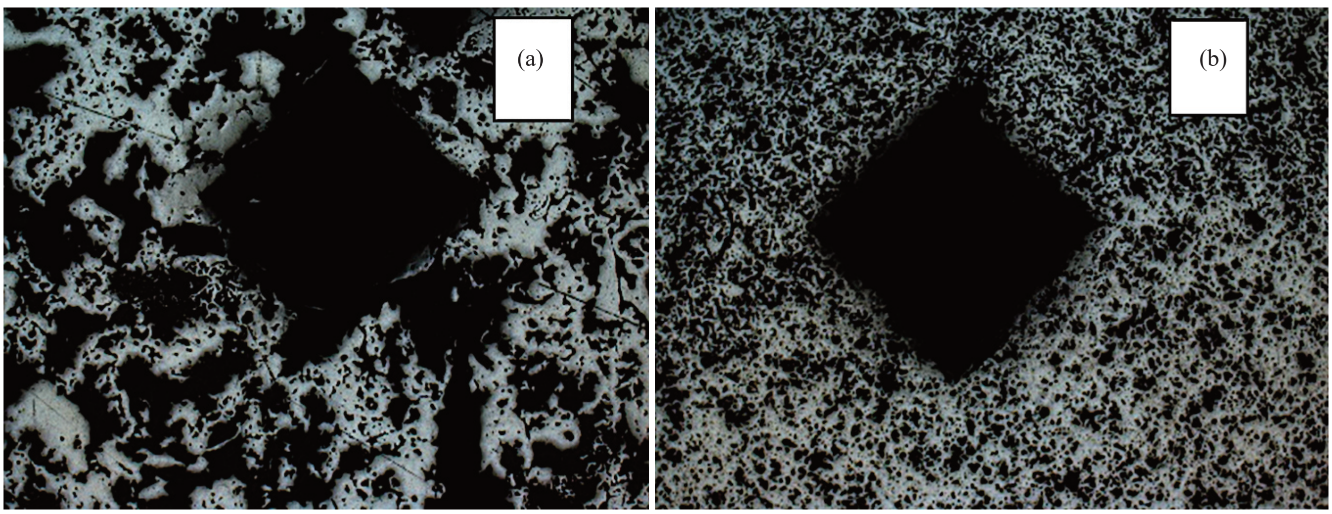


Fig. 6. General view of sample M of a two-layer titanium powder tube (a), microstructure (b) and macrostructure of the inner surface (c).



HV30 – 42.37 ± 1, HVm – 304.93 ± 2 HV30 – 44.57 ± 5, HVm – 356.43 ± 3

Fig. 7. Micrographs of sample M with indentations after microhardness measurements: less porous part (a) and more porous part (b).

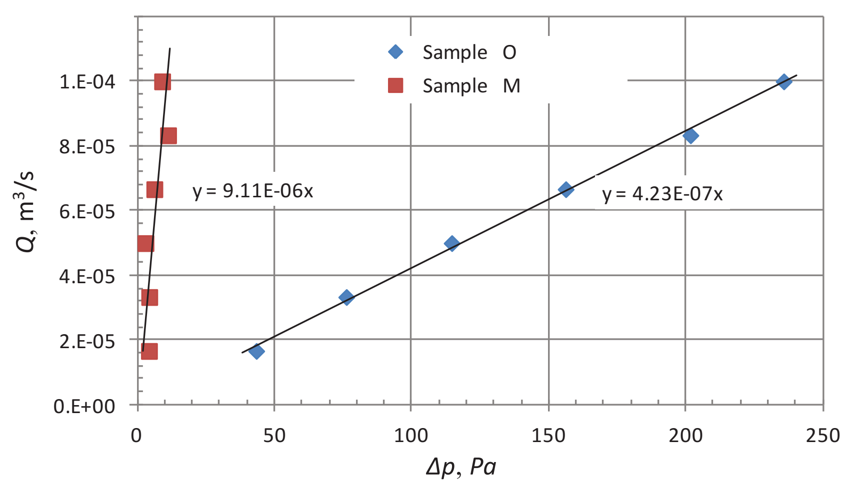


Fig. 8. Dependence of the airflow Q through the sample wall on the pressure drop Δp across it.

At the same time, the porosity of sample O is significantly higher than that of sample M. This suggests that porosity is not a determining factor for air permeability.

Regarding the technological advantages of each sample type, the intended application must be considered. Sample O with its lower density and substantially lower permeability would be suitable for the creation of more compact filtration devices. Conversely, sample M with its higher density and greater permeability offers superior thermal and physical properties, making it more suitable for use in evaporators. Microhardness does not significantly influence the performance in this context. The strength of titanium samples, how-

Table 2. Summary of O and M sample parameters

Parameters	O	M
Air permeability of sample: $Q/\Delta p$, $m^3/s{\cdot}Pa$	$4.23{\cdot}10^{-7}$	$9.11{\cdot}10^{-6}$
Conventional air permeability of material: L, $m^2/ s{\cdot}Pa$	$8.07{\cdot}10^{-8}$	$1.7{\cdot}10^{-6}$
Linear air permeability of tube: $Q/(L{\cdot}\Delta p)$, $m^2/s{\cdot}Pa$	$2.04{\cdot}10^{-6}$	$1.18{\cdot}10^{-4}$
Sample mass, g	1167.2	386.1
Sample volume, cm^3	526.8	134.3
Sample density, g/cm^3	2.22	2.88
Alloy	Ti-Si-Fe	Ti-Si-Fe
Alloy density, g/cm^3	4.54	4.54
Porosity, %	51.2	36.7

ever, is primarily dependent on their porosity. The use of two-layer samples could enhance their strength while maintaining, or even increasing, their porosity.

4. Conclusion

This study highlights the relationship between the microstructure, porosity, and permeability of titanium samples, demonstrating that air permeability is not solely determined by porosity. The results indicate that while sample O has higher porosity, sample M exhibits higher permeability. Depending on the intended application, materials with different properties can be chosen: sample O is better suited for compact filtration devices, whereas sample M offers superior thermal and mechanical properties for evaporators. Additionally, the strength of titanium samples is primarily influenced by their porosity, and the use of two-layer samples can enhance strength while maintaining desirable porosity. These findings open up possibilities for optimising titanium-based materials for various industrial applications.

Data availability statement

Data are available upon request.

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Magnetimpulsspressitud poorsete titaanist proovide omaduste ja struktuuri hindamine

Ervins Blumbergs, Viktors Mironovs, Jekaterina Nikitina, Michail Maiorov ja Vjaceslavs Zemcenkovs

Viimastel aastakümnetel on tehtud palju uurimistööd ja jõupingutusi titaani ja titaanisulamite kasutamiseks masina- ja keemiatööstuses, meditsiinis ning teistes valdkondades. Titaanipõhised pulbermaterjalid mängivad selles protsessis tähtsat rolli. Titaan sobib hästi erinevateks tehnilisteks rakendusteks tänu oma suurele tugevusele, väikesele mahukaalule ja suurepärasele korrosioonikindlusele. Titaanpulbrist valmistatud toru- ja rõngakujulistel toodetel on oma kindel nišš. Käesoleva uuringuga kavatseti selgitada võimalusi toota titaanpulbrist ühe- ja mitmekihilisi torukujulisi tooteid, rakendades magnetimpulss-paagutuse tehnoloogiat. Uuringus kasutati titaankäsna jahvatamisel saadud titaanpulbrit osakeste suurusega 80–250 µm. Valmistatud toodete mikrostruktuuri analüüs näitas, et võimalik on valmistada 30–50% poorsusega toru- ja rõngakujulisi tooteid. Leiti, et ühekihiliste torukujuliste toodete välispind on poorsem ja väiksema kõvadusega kui sisepind, mida saab seletada kergsulava materjali kasutamisega kesta sisepinnal magnetimpulss-pressimisel. Kahekihiliste toodete valmistamisel on võimalik saavutada suurem tugevus, säilitades samal ajal gaasi läbilaskvuse.