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Properties and Structure Investigation of Mechanically Activated Titanium Metal-Matrix Composites

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Abstract
Ti-based metal-matrix composites from mechanically activated powder were obtained by high energy pulsed methods: electrical explosion of the wires and magnetic-pulsed compaction. The grinding of $Ti+TiO_2$ powder led to a significant decrease of Ti content. At that the oxide component of the powder underwent both quantitative and structural (qualitative) changes: in addition to the initial phases of anatase and rutile, mechanically activated compositions contained crystal structures of lower oxides. The magnetic-pulsed compaction of composite powders destroyed the oxide shells in "metal core-oxide shell" structure turning the composite compact to a conductive material with high mechanical properties.
Keywords:*Titanium composite, mechanical properties, conductivity, microstructure.*

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I. INTRODUCTION

Metal-matrix composites (MMCs) consist of non-metallic reinforcements (i.e., ceramic) in metal matrices.Theyshowhightoughness, wear and fatigue resistance and relatively light-weight. One of the metallic composites with remarkable mechanical properties is titanium metal matrix composite (Ti-MMC) that is widely used in instrumentation, aircraft, space and automotive industries, as well as in biological applications [1–5].

There are two distinct types of composite systems, depending on the length-to-diameter ratio (l/r) of the reinforcement: continuous reinforced MMCs with l/r>>1 (i.e., fibers) and discontinuously reinforced MMCs with l/r≥1 (i.e., particles or whiskers) [6-8]. It is considered generally that continuously reinforced MMCs have better mechanical and physical properties comparing to discontinuously reinforced ones. Unfortunately this effect can turn miserable due to the anisotropy of the properties over the volume of the sample. In order to overcome this problem one can use particles for the reinforcement as the best structural homogeneity is reached here and a higher volume fraction of them can be incorporated into metal matrices as compared to the whisker reinforcement [9]. Moreover such structure gives an opportunity to obtain the composite with the elevated properties by forming the fine-grained material.

The fine-grained structure synthesis process by powder technology demands nanosized powders as the initial media and an effective method of their compaction. Suchpulsedmethodsas electrical explosion of the wires (EEW) [10] and magnetic-pulsed compaction (MPC) [11] fully meet this challenge. EEW allows obtaining weakly aggregated composite $Ti+TiO_2$nanopowders with uniform volume distribution of the oxide phase, whereas MPC allows obtaining dense fine-grained bodies due to high-speed compaction.

Thus the purpose of this work is the synthesis of Ti-based MMCs using high-energy pulsed methods and their physical properties investigation.

II. EXPERIMENT

2.1. Initial powder

The initial powder was obtained by EEW method from industrial titanium wire with a diameter of 0.5mm and a length of the blasted section of 70–160mm. Thecapacitanceofthe capacitor bank was 3.2-6.4μF. The battery was charged to a voltage of 25–28kV. Explosions were carried out at a frequency of 0.4Hz with a controlled supply of oxygen at a flow rate of 0.17-1cm3/s. Argon was used as the carrier gas. The powder particles were formed as metal spheres surrounded by a thin oxide shell preventing their agglomeration. The average particle size, d_{BET}, was 150nm.

The presence of dielectric titanium dioxide in "metal core – oxide shell" structure eliminated the electrical conductivity of the powders.

2.2. MechanicalActivationofthe Powder

Mechanical activation (MA) of the powder was carried out by grinding in a planetary ball mill AGO-2s (Russia) with 9.5mm steel balls. Theballs/powderweightratio was 3:1. The initial powder was processed by mechanical action at two rotation speeds of the milling chambers: 910 and 1820 rpm, which are 50 and 100% of the nominal frequency, respectively. In both cases the grinding of the powder was carried out in Ar atmosphere for 15 minutes.

Table 1: Characteristics of Ti-based powders obtained by EEW and subjected to mechanical activation.

Powder	ν, rpm	S_{BET}, m^2/g	a-Ti, wt. %	TiO_2 (anatase), wt. %	TiO_2 (rutile), wt. %	$TiO_{0.48}$, wt. %	c-TiO, wt. %
Ti-based	0	8.9	95	2	3	0	0
	910	7.9	67	1.6	4.8	10.7	15.9
	1820	6.1	65	4.8	6.6	12.7	10.9

where: ν - rotation speed of the milling chamber, S_{BET} - specific surface area of the powder, a- and c- - alpha and cubic phases, respectively.

It is seen from Table 1 that mechanical activation leads to the decrease of the specific surface area of the composite powders. Itmeansthathigh-energy grinding results in agglomeration of the powder particles [12]. Moreover, this effect becomes more severe with the increasing grinding speed. According to X-Ray analysis the mechanical activation of Ti-based powder reduces the amount of metallic titanium significantly (down to ~30%) as more metal particles are exposed to oxidizing during milling. It was found that during mechanical activation the oxide component of the powder undergoes both quantitative and structural (qualitative) changes: in addition to the initial phases of anatase and rutile, these compositions contain a large amount of the crystal structures of defective lower oxides.

2.3. Powder Compaction

The composite powders were shaped on magnetic-pulsed press [13] into discs with a diameter of 15mm and a height of ~1.5mm. Compaction was carried out after preliminary degassing in a vacuum (residual pressure ~10Pa) at room temperature and a temperature of 400°C. The treatment of the powders by pulsed pressure with amplitudes of 1.0-1.8GPa allowed obtaining compacts with relative density in the range of 0.77–0.95.

2.4. MeasurementMethods

The powder particles size was determined from the specific surface area, measured by BET method from nitrogen adsorption on a TriStar 3000 V analyzer after degassing of the samples at T = 200°C in vacuum for 2 hours. The X-rayanalysiswas performed on a D8 DISCOVER diffractometer using copper radiation (Cu $K_{\alpha 1.2}$ λ = 1.542Å) with a graphite monochromator on a diffracted beam. The density of the compacted samples was determined by the standard method of hydrostatic weighing. MMC microstructure was studied by atomic force microscopy on a Solver 47p instrument. For the samples resistivity analysis the LCR-76100 precision impedance meter with a DC resistance measurement limit and a resolution of 10μΩ was used.

The mechanical properties of MMC were studied by indentation method on Nanotest 600 device using a Vickers diamond pyramid. Themaximumloadwas 1N, the loading and unloading times were 20 seconds each. The indenter penetration depth was ~6.6μm. A 5 seconds dwell time was used for microhardness and elastic modulus determination. Creepwasmeasuredby the penetration of the indenter under the maximum load for 30 seconds.

III. RESULT AND DISCUSSION

The mechanical properties analysis of the compacted bodies showed that an increase of the pulsed pressure amplitude leads to a proportional densification of the powders (Figure 1). This dependence was found to be in good correlation with [14] meaning that under the chosen experimental conditions no shock pressing wave is formed. This eliminates the destruction of grain boundaries, a density decrease and the sample integrity violation.

It is seen (Figure 1) that cold compaction at low pressure leads to a decreased density of the mechanically activated powders. This is explained by a hard oxide phase formation upon milling (Table 1). It should be noted that this effect becomes insignificant at high (1.8GPa) pressure indicating the maximum possible compactibility at the chosen experimental conditions. The additional densification is expected upon the introduction of thermal energy, which is spent on the effective particles sliding against each other and their plastic deformation. In present work the elevated up to 400°C temperature led to additional increase of the relative density of the compacts up to 4%.

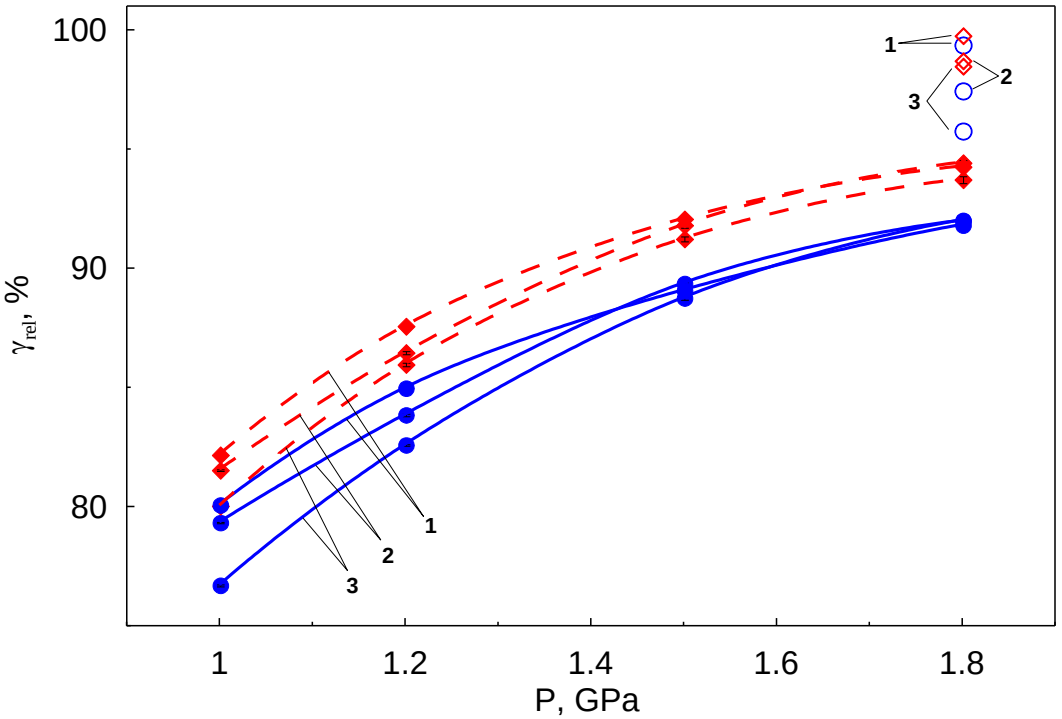


Figure 1: Dependence of Ti-MMCs relative density from the applied pulsed pressure at different milling chamber rotation frequencies: 1 – 0 rpm, 2- 910 rpm and 3- 1820 rpm. Solid lines – cold compaction at room temperature, dotted lines – compaction at 400°C, hollow symbols – sintered at 1200°C MMCs

When analyzing the microhardness of the composites its correlation with their density and phase content was found. Denser samples with low metal content showed better hardness (Figure 2). It is seen that the maximal density for such tendency is 90% as application of higher pulsed pressure doesn't lead to a sufficient hardening. It should be noted that the obtained level of microhardness exceeds the microhardness of pure industrial titanium (~1.5GPa) by 3 times.

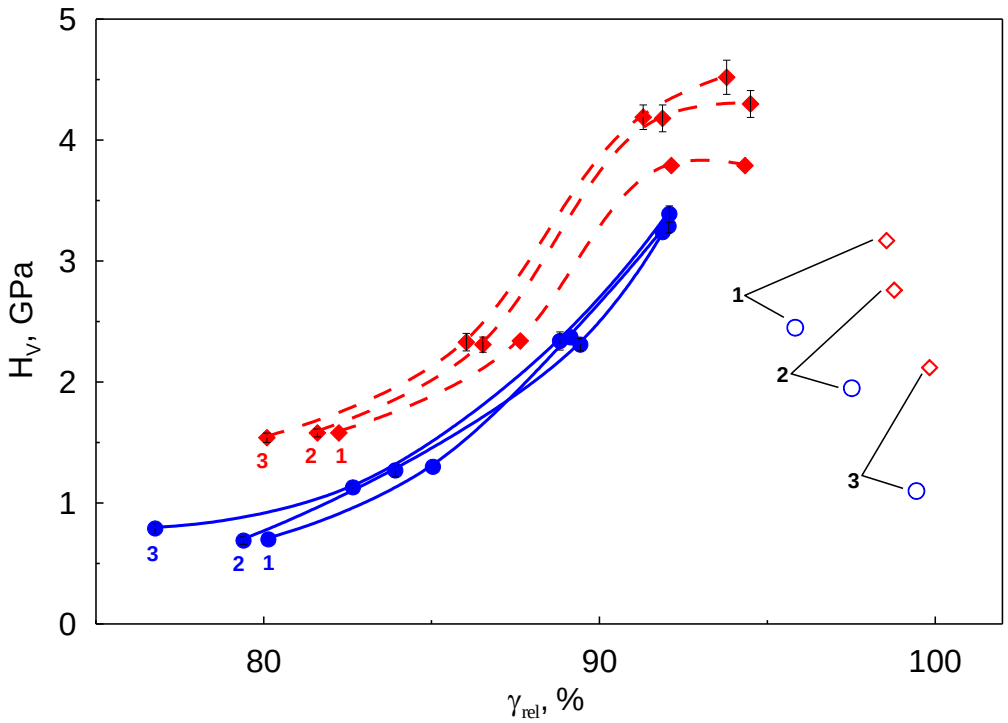


Figure 2: Influence of compact's relative density on their microhardness. 1- 95 wt.%, 2- 67 wt.%, 3- 65 wt.% of Ti. Solid lines – cold pressing at room temperature, dotted lines – compaction at 400°C, hollow symbols – sintered at 1200°C MMCs

A more detailed analysis showed that, regardless of the grinding intensity, sintering in argon at a temperature of 1200°C leads to an increase in the relative density of the samples by 3-5%, which approaches to the full density of MMC (hollow symbols on Figure 1). It is interesting to note that despite such high density the averaged microhardness of the sintered specimens decreases approximately by a factor of 2 (hollow symbols on Figure 2). It is explained by the development of structural instability in titanium alloys, leading to their embrittlement at temperatures above 500°C [2]. This fact is confirmed by the analysis of the indenter penetration into Ti -MMC composite at a load of 15N (Figure 3). In case of green body indentation a smooth classic loading curve is observed. In case of sintered MMC one can see a sufficient increase of the penetration depth at a load around 6N indicating macro destruction of the surface (insertion in Figure 3).

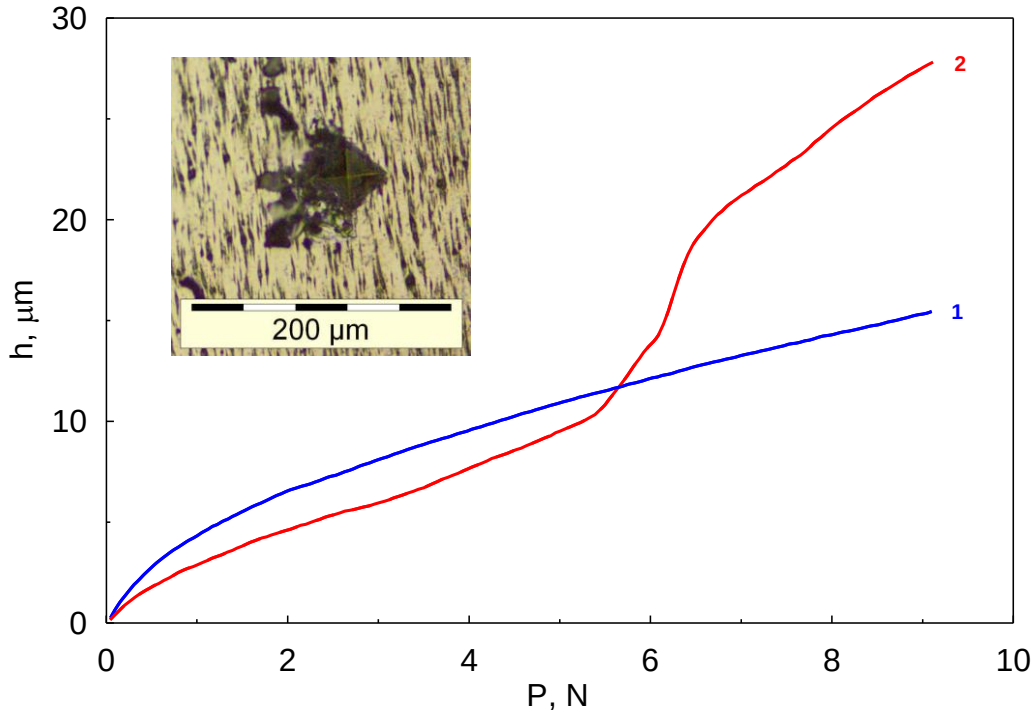


Figure 3: Dependence of the indenter penetration depth form the applied load. 1 – Ti-MMC green body, 2 – sintered at 1200°C Ti-MMC. Insertion – indenter imprint on the surface of the sintered Ti-MMC

When studying the effect of pressing conditions on the elastic properties of the composites, a proportional correlation was also found with the density of the compacts, however, the temperature effect in this case is less pronounced compared, for example, to microhardness.

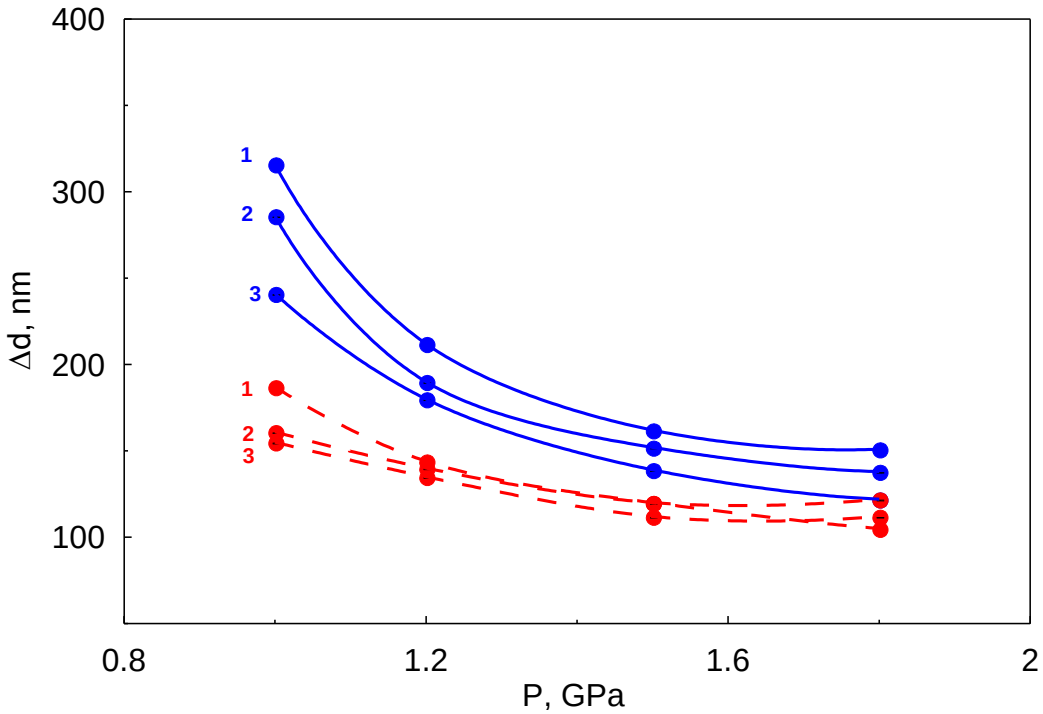


Figure 4: Composite' creep dependence on the applied impulse pressure at different milling chamber rotation frequencies: 1 – 0 rpm, 2- 910 rpm and 3- 1820 rpm. Solid lines – cold compaction at room temperature, dotted lines – compaction at 400°C

Upon the creep measurements of composites with different densities, a tendency to a decrease in plasticity with an increase in the applied pressing pressure was found (Figure 4). These data correlate well with the increasing modulus of elasticity, however, the effect of a sharp change in the composition of the powder as a result of mechanical activation in a planetary mill is not observed. Thus, density appears to be a determining factor of elastic-plastic properties in the composites.

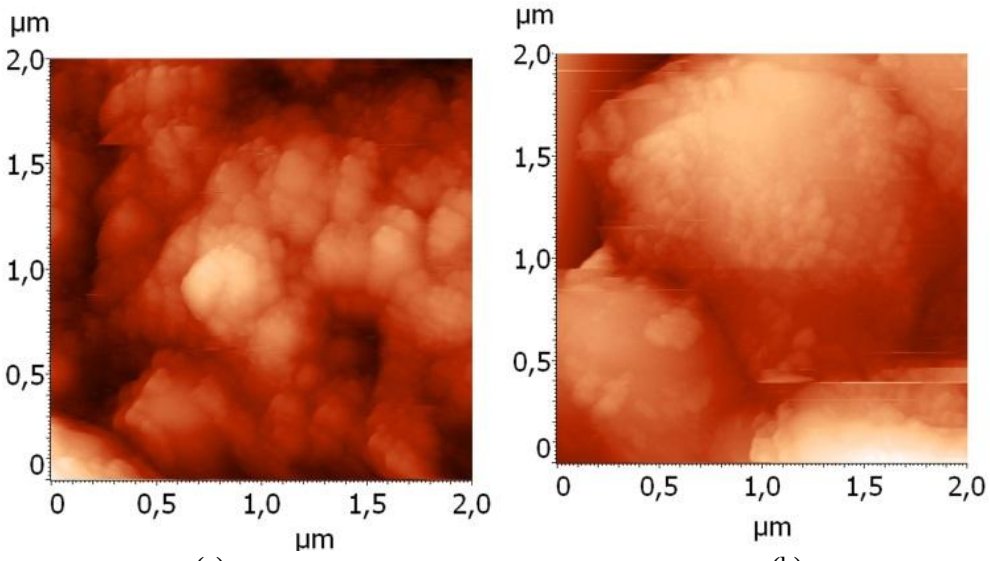
The results of calculations of resistivity (ρ) and specific conductivity (σ), are presented in Table 2. For comparison, the resistivity of pure Ti at room temperature is 0.042μOhm·cm. Since the oxide phase has a significantly higher resistivity (~$1 \cdot 10^8$μOhm·cm), the MMC conductivity is expectedly lower than that of Ti. It's worth mentioning that MMC obtained from initial powder possesses some electrical conductivity. It is explained by the destruction of non-conductive oxide shells upon plastic deformation during MPC and by metallic bonds formation.

Table 2: Electrical characteristics of Ti-MMCs.

ν, rpm	T_c, °C	γ, g/cm^3	ρ, μOhm·cm	σ, $\cdot 10^3$Sm/cm
0	20	4.01	1.73	0.58
	400	4.27	0.21	4.76
910	20	3.73	12.28	0.08
	400	4.15	0.41	2.15
1820	20	3.57	17.43	0.06
	400	4.02	0.87	1.42

The table shows that the most important factor affecting the conductivity of the composites is the compaction temperature. Thus, samples pressed at 400°C are characterized by a conductivity 10–20 times higher than samples pressed at room temperature with all other conditions being the same. It is important to note that mechanical activation leads to a decrease in conductivity by about 2 times, which is explained by the formation of a large amount of oxide phases during the grinding process.

When comparing the microstructure of composites obtained from different powders, the fact of the formation of large aggregates upon intensive grinding is confirmed (Figure 5). Moreover, such a structure correlates with a decreasing specific surface area of the powders (Table 1).



(a) (b)

Figure 5: AFM fracture images of composites obtained form (a) initial powder and (b) activated at 1820 rpm powder. Compaction pressure - 1.5GPa, compaction temperature – 20°C

IV. CONCLUSION

As a result of mechanical activation of $Ti+TiO_2$ powders by grinding, the proportion of metallic titanium decreases significantly (by approximately 30%). In this case, the oxide component of the powder undergoes both quantitative and structural (qualitative) changes: in addition to the initial phases of anatase and rutile, mechanically activated compositions contain crystal structures of lower oxides.

The magnetic-pulsed compaction of composite powders destroys the oxide shells in "metal core-oxide shell" structure turning MMC to a conductive material.

The dynamic plastic deformation at 400°C allowed obtaining Ti-MMC with high mechanical properties: relative density - 0.95, microhardness - 4.7GPa, reduced modulus of elasticity - 114GPa, creep - 112nm.

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