HIGH-ENERGY SYNTHESIS OF METALOMATRIC COMPOSITES HARDENED BY MAX PHASES OF Ti-Al-C SYSTEM

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Abstract: *The possibility of synthesizing the Ti_3AlC_2 and Ti_2AlC MAX phases by SPS (spark-plasma sintering) consolidation of the charge obtained as a result of mechanoactivation and a charge obtained by HVED (high-voltage electric discharge) of processing titanium and aluminum powders in kerosene has been experimentally studied. The influence of the charge activation method on phase formation and physical properties of consolidated materials is shown.*
KEYWORDS: *METAL-MATRIX COMPOSITE, MAX PHASES, MECHANOACTIVATION, HIGH-VOLTAGE ELECTRIC DISCHARGE, SYNTHESIS, SPARK-PLASMA SINTERING.*

1. Introduction

The creation of new materials with high physical-mechanical and operational properties, which will ensure the necessary reliability of products in extreme conditions, is relevant for the development of modern technology. Unfortunately, the possibilities of obtaining them using traditional metallurgical methods have been largely exhausted. Therefore, an important challenge facing scientists and engineers is to develop physical principles for the creation of new materials in the conditions of high-energy preparation of powders and extreme high-speed effects of temperatures and pressures during consolidation by spark-plasma sintering (SPS), which will allow obtaining materials with higher physico-mechanical properties than those obtained by traditional metallurgical methods.

Problem discussion

The MAX phase means a triple system $M_{n+1}AX_n$ having a hexagonal close-packed structure, in which M is a transition metal, A is an element of the A subgroup of the Periodic Table, and X is carbon or nitrogen. An essential distinction of these materials is the layered structure of their hexagonal crystal lattices, in which the layers of M and A atoms alternate in a certain sequence. This ensures their high physical and mechanical characteristics, which combine the properties of ceramics and metals. Among the many MAX phases which are synthesized nowadays Ti-based MAX-phases are of greatest interest: Ti_2AlC, Ti_2AlN, Ti_3AlC_2 and especially Ti_2AlC which can be used under conditions of high temperatures and increased corrosion due to its layered structure [1 - 3]. Therefore, the creation of metal matrix composites, which have a Ti-Al-C system in their MAX phase structure, is a topical scientific task.

The objective of this work is to investigate the possibility of creating high-energy methods of exposure to metal matrix composites hardened by MAX phases of the Ti-Al-C system.

2. Objective and research methodologies

To obtain metal matrix composites reinforced with nanolaminant MAX phases, a new approach was applied, which consists in the synthesis of strengthening phases (including MAX phases) in two stages: at the first stage occurs dispersion, preparation of the powder surface and synthesis of dispersed reinforcing inclusions. At the second stage, the material with dispersible reinforcing additives occurs during the SPS while maintaining the nanostructure.

At the first stage, two different methods of synthesis and activation were used: mechano-chemical with the use of a planetary mill and high-voltage electric discharge processing of powders in a hydrocarbon liquid (HCF) [4].

Mechanoactivation of the initial large-dispersed powders of titanium, aluminum and graphite in an atomic ratio of 3:1 and 1:2 was carried out at room temperature in a Fritsch Pulverisette 6 planetary mill in an argon atmosphere for 3 hours. The rotational speed of the mill was 400 rpm. The beads to sample mass ratio was 20:1.

The obtained powders were examined by an optical microscope Biolam-I using the Image-J software according to [5, 6]. As a result, histograms were obtained for the distribution of their particles along the diameter and shape factor (Fig. 1, 2) and it was identified that more than 80% of the particles of the mixture are larger than 20 μm, 27% of particles are less than 10 μm. The predominant form of particles is spongy (45%) and fragmentation (up to 30%).

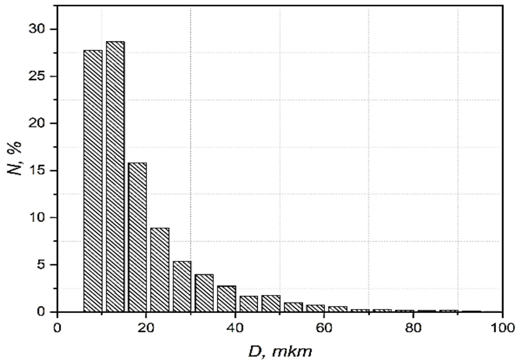


Fig. 1. *Size distribution of powder particles of the Ti-Al-C system obtained by mechanoactivation*

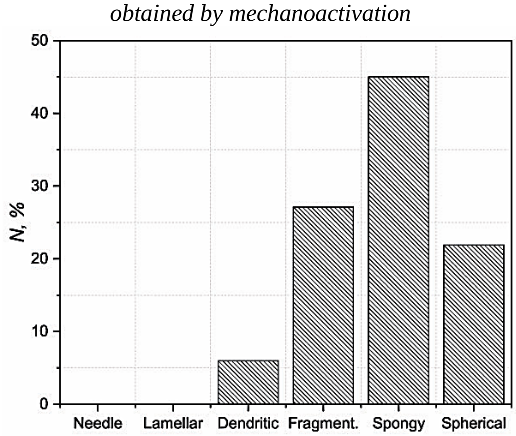
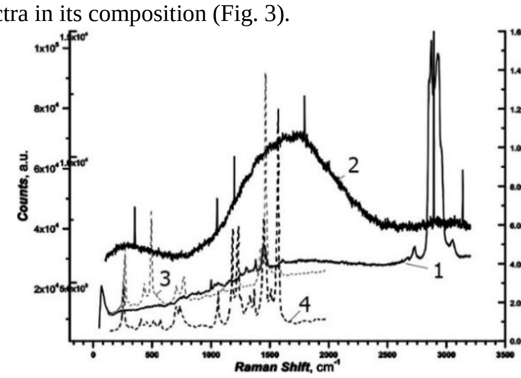


Fig. 2. *Form distribution of powder particles of the Ti-Al-C system obtained by mechanoactivation*

Using hydrocarbon liquid (kerosene) as a working medium for the HVED processing of the initial mixture of Al-Ti powders helps avoid administering additional graphite into the mixture. In a high-voltage discharge conditions are created (the pressure in the discharge channel reaches 1 GPa and the temperature in the discharge channel can reach 50,000 K) to pyrolyze kerosene to form solid-phase nanocarbon. Synthesized carbon nanoparticles are able to enter carbidization reactions with powder particles, forming nanostructural strengthening phases [7]. Thus, the analysis of kerosene after the HVED treatment by the Renishaw InVia Micro Raman unit showed the formation of the C_{60} and C_{70} spectra in its composition (Fig. 3).



1 – initial kerosene; 2 – kerosene after HVED; 3 – C_{60}; 4 – C_{70}

Fig. 3. *Kerosene spectroscopy after HVED processing*

Optical microscopy of the charge obtained as a result of the HVED processing of 85% Ti + 15% Al source powders in kerosene with specific energy W_{Σ}=25 MJ / kg showed that more than 50% of the particles have an average diameter of less than 20 µm, and 17% of particles are less than 10 µm (Fig. 4). The predominant form of particles is spongy (45%) and spherical (35%) (Fig. 5).

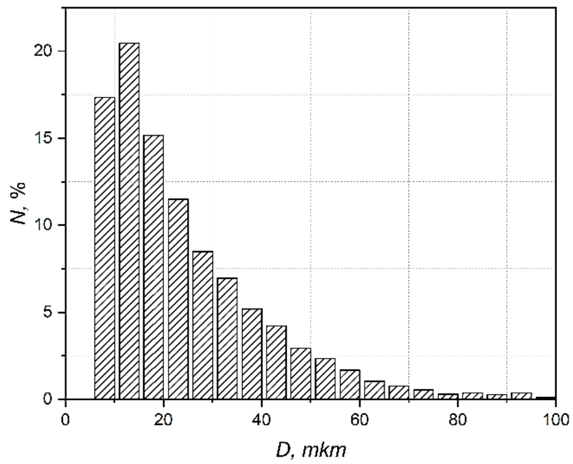


Fig. 4. *Size distribution of powder particles of the Ti-Al-C system, obtained by HVED processing*

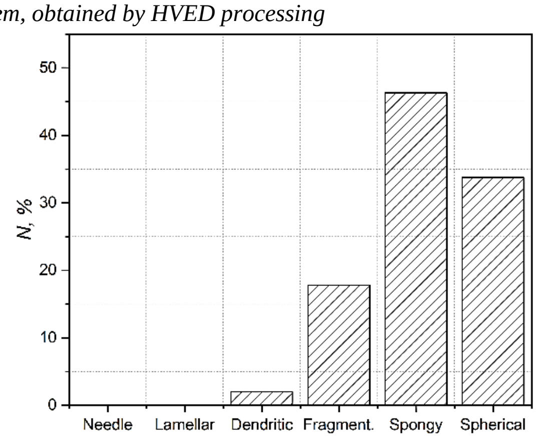
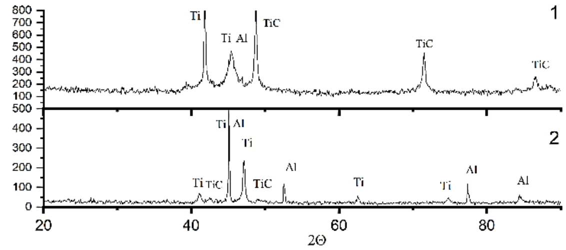


Fig. 5. *Form distribution of Ti-Al-C powder particles obtained by HVED processing*

X-ray diffraction studies were carried out on a laboratory HZG-4A diffractometer in COKα-radiation and allowed to establish that the powders obtained as a result of mechanoactivation and HVED have not only the similar granulometric, but also the phase composition (Fig. 6) and differ only in allotropic modifications of carbon in the mixture.



1 - mechanosynthesis; 2 - HVED

Fig. 6. *Phase composition of powders after mechanoactivation and HVED preparation.*

Experiments on the consolidation of the obtained compositions were carried out by the spark-plasma sintering method at the Gefest-10 unit developed at the IPPT NASU [8]. The unit allows consolidating the powders under mechanical loading in vacuum by passing a superposition of direct and alternating currents with an amplitude of 1.1 kA with a pulsating component frequency of 10 kHz through the stock. SPS was carried out in graphite matrices (graphite MPG 6) with an internal diameter of 10 mm, where 3 g of charge were filled. The pressing pressure in all the experiments was ca. 40 MPa, and the sintering pressure was ca. 60 MPa, the isothermal holding time was 5 minutes.

X-ray diffraction studies of the consolidated mechanically activated Ti3Al1.1C2 powder sintered at 1100 ° C for 5 min showed that the phase composition changes significantly in the SPS process: a very strong graphite line appears and the relative content of TiC decreases, and the proportion of Ti3AlC2 MAX phase increases (Fig. 6). The carbon source can be either a carbide phase or a graphite matrix in which sintering was carried out.

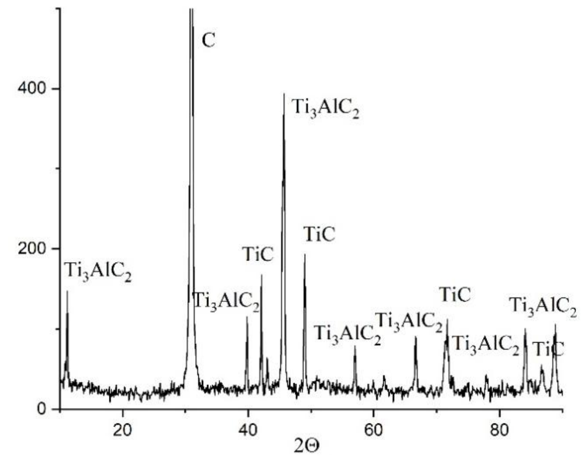


Fig. 7. *Phase composition of the SPS-consolidated mechanically activated powder, CoKα-radiation*

The study of the phase composition by the Rigaku Ultima IV diffractometer of SPS-consolidated powders obtained by the HVED processing of a 85% Ti + 15% Al mixture in the previously considered regime showed that Ti2AlC MAX phase and ternary carbide Ti3AlC (Fig. 7) originate in them, which can be due to the presence of nanocarbon such as C60 and C70. The increased graphite peak is worth noticing, too.

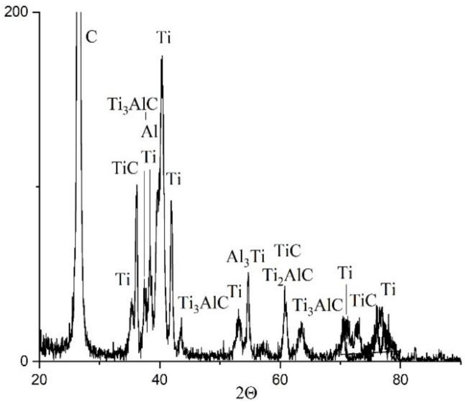
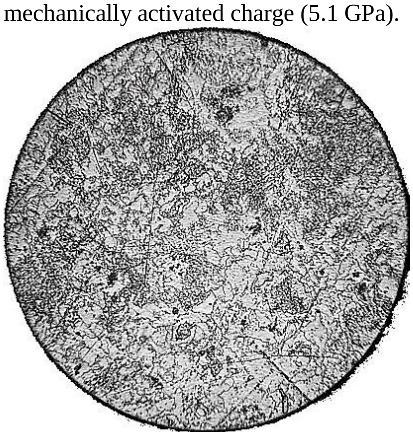


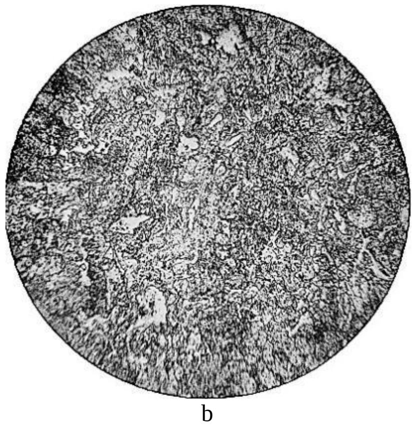
Fig. 8. *Phase composition of the SPS-consolidated HVED-activated powder, CuKα-radiation*

The different phase composition of the consolidated samples led to changes in their structure and physical properties. Thus, it is clear from optical microphotographs (Fig. 8) that the dark carbide phase Ti3AlC and TiC prevails in the samples obtained from the HVED-activated charge (Fig. 8, b). The microhardness of the dark zones measured with the PMT-3 device is 11 GPa. Light zones are reinforced titanium with a microhardness of 7 GPa. Moreovere, in the light zones there are areas with a microhardness of up to 13.5 GPa (Ti2AlC MAX phase). In the samples obtained from the mechanically activated charge (Fig. 8, a) prevail the light zones with a microhardness of 7 GPa with inclusions of 13.5 GPa (Ti3AlC2). At the same time, the number of dark zones with a microhardness of 11 GPa appropriate to TiC only is much smaller.

The different hardness and varied distribution of the components in the consolidated material affected the overall Vickers hardness of the material (as per DSTU ISO 6507-1: 2007). Thus, HV5 of HVED-consolidated samples from the activated charge is 7 GPa, which is almost 2 GPa higher than for samples from mechanically activated charge (5.1 GPa).



a



b

a - mechanically activated charge material; b - HVED-activated charge material

Fig. 9. *Microstructure of consolidated materials, x 250*

4. Conclusions

This pilot study dealt with finding out the possibility of creating metal matrix composites strengthened by MAX phases of the Ti-Al-C system by SPS consolidation of the charge after mechanoactivation and HVED processing of titanium and aluminum powders in kerosene has been investigated.

It is shown that the charge prepared in the course of mechanoactivation and the charge obtained in the process of HVED processing in kerosene have a similar granulometric and phase composition and differ only in allotropic forms of carbon in their composition.

The effect of allotropic forms of carbon on the formation of new phases in the SPS process is established. Thus, in the mechanically activated charge which contains graphite occurs the formation of the Ti3AlC2 MAX phase, and in the HVED-activated charge that contains C60 and C70 the Ti2AlC MAX phase and the Ti3AlC ternary carbide are formed.

It is shown that the metal-matrix composite of the Ti-Al-C system that contains the Ti2AlC MAX phase and the Ti3AlC ternary carbide has a Vickers hardness of 7 GPa, which is almost 2 GPa higher than in the samples that contain the Ti3AlC2 MAX phase (5.1 GPa).

5. Literature

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