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Experimental study of laser treatment of Ti_3AlC_2 MAX phase

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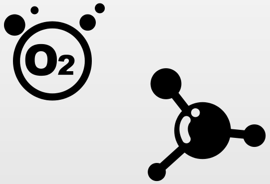
Introduction

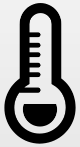
Nowadays, additive technologies, or in other words, 3D-printing technologies, are developing very actively. With the development of additive manufacturing (AM) technologies based on the selective sintering or melting of powder materials with laser or electron beams, the production of materials and products from them with predetermined properties is determined by the properties of the starting materials and molten or sintered materials formed under high-energy exposure.

The properties of such materials strongly depend on the structure and phase composition of the formed material, which are determined by:

The elemental and chemical composition



The gas atmosphere



The thermal conditions during high-energy exposure

MAX phases

Ternary carbides and nitrides, so-called MAX phases, the new class of materials, that has very specific properties, **combining the properties of both metals and ceramic materials**. Like metals, these compounds show high thermal and electrical conductivity, as well as fairly high resistance to thermal shock. These properties of the MAX phases open up broad prospects for their application in various industries.

Nowadays, the majority of the research in the field of MAX phases of the Ti-Al-C system can be divided into two main groups:

Synthesis of MAX-phases

- Involve optimization of the experimental parameters for obtaining the MAX phases;
- Studies in this field are associated with high-temperature processes such as SHS, SPS, and synthesis under pressure, such as HIP.
- These processes either do not imply the formation of products (SHS, SPS) or provide for the formation of high-density products from MAX-phases of simple geometry (HIP).

Production of composite materials based on MAX-phases

- The investigation of the MAX-phase-based composites includes complex structural studies and mechanical tests of complex Ti-Al-C materials.
- The most of the works devoted to this imply either the above-described technologies used for the synthesis of MAX-phases (SHS, SPS, HIP) or sintering with long exposures.

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Research objective

The formation of bulk products from MAX-phases is an urgent and promising task today. Additive technologies are able to solve the problem of forming complex shapes of products from MAX-phases. However, it is necessary to investigate the influence of the modes of laser action on the MAX-phases. This will help to understand how the MAX phase decomposes under the action of a laser and which modes allow the formation of the most dense structure.

A feature of the described works is the study of the formation of phases and structure-forming processes in simple samples. The formation of the complex geometry of the samples is not described since it is a non-trivial task.

This work aimed at obtaining bulk samples from the powder of the MAX-phase Ti_3AlC_2 using the selective laser sintering (SLS) method, followed by studying the structure and phase composition of the obtained samples.

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Materials and methods

- The Ti_3AlC_2 powder (Wuhan Golden Wing, China; purity no less than 98%, d50 = 10 μm) was used as the initial material. Samples for studies were obtained by the SLS on a LUCH-500 selective laser sintering/melting installation (developed and created at the Tomsk Polytechnic University).

SLS printing parameters:

- Laser power: from 10 to 500 W;
- Scanning speed: from 1 to 20,000 mm/s;
- Laser beam diameter: 150-300 μm;
- Working area temperature: up to 400 °C;
- The inert gas overpressure: up to 0.7 atm;
- Substrate material: Rose's alloy.

- The morphologies and the microstructure formed in different parts of the samples was studied using LEO EVO 50 scanning electron microscopy (Zeiss, Germany). This microscope also has the EDX point microanalysis for local determination of elemental composition. The samples were also studied using optical microscopy. The phase composition was analyzed by X-ray diffraction (Cu Kα-radiation) on a Shimadzu XRD 7000S installation equipped with the OneSight high-speed 1280-channel detector.

Results and discussion

- *The initial powder*

The elemental analysis of the powder carried out using a scanning electron microscope indicates the presence of tin in the powder. Literature* has described a method for synthesizing Ti_3AlC_2 powder using tin as an additive that inhibits the growth of the Ti_2AlC phase and maximizes the yield of the Ti_3AlC_2 phase. The powder used in this work was obtained by the self-propagating high-temperature synthesis (SHS), and tin during the synthesis is distributed along the grain boundaries, forming thin layers. Such interlayers are most often not detected by X-ray diffraction analysis.

The EDX point analysis, carried out using the SEM, indicates the presence of tin in an amount not exceeding 2 wt.%. Therefore, the specified phase composition and purity of the powder correspond to the manufacturer's statements.

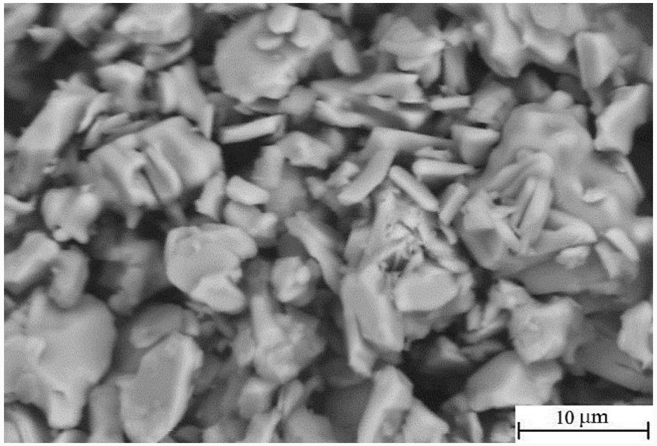


Fig. 1. The morphology of Ti_3AlC_2 powder.

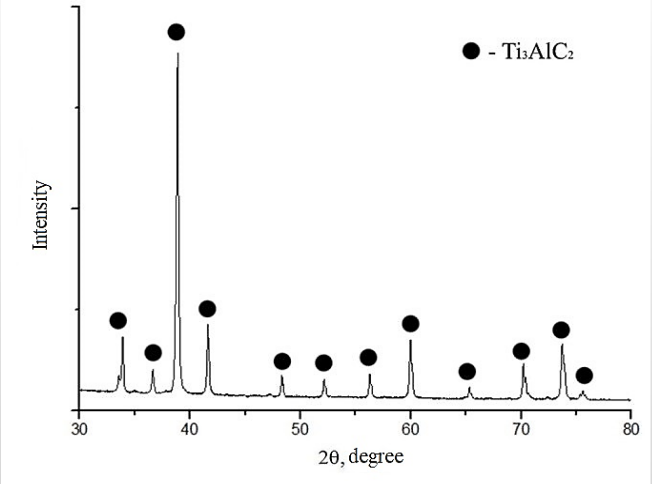


Fig.2. X-ray diffraction pattern of the initial powder.

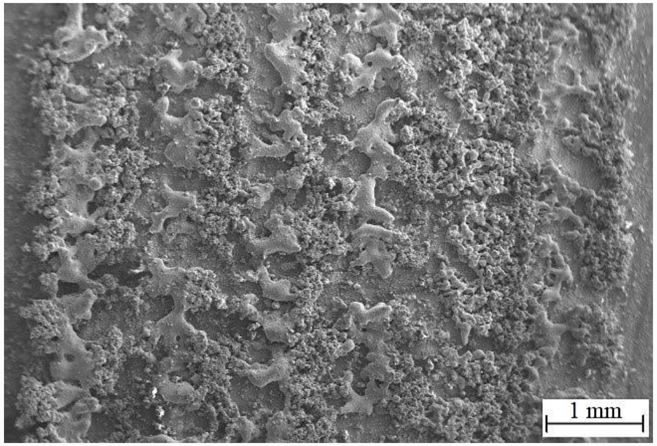
* A. Mingxing, et al. "Synthesis of Ti_3AlC_2 powders using Sn as an additive," Journal of the American Ceramic Society, vol. 89, № 3, pp. 1114-1117, 2006.

Results and discussion

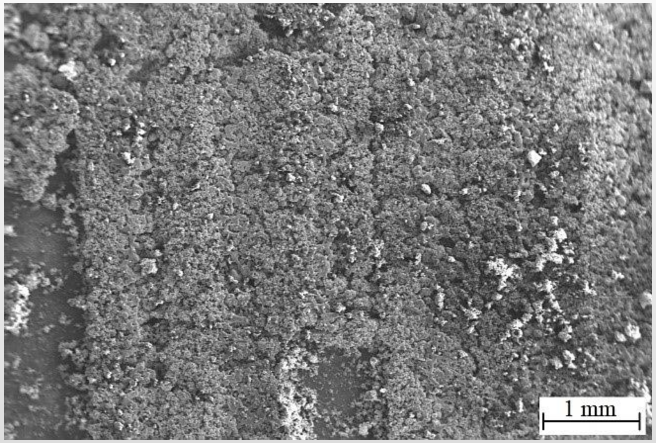
- *SLS samples*

The sintered powder structure away from the laser track center corresponds to the initial one, which suggests the conservation of the MAX phase.

The conservation of the MAX phase after SLS is possible only at relatively low power input. So, when processing at a power of 80 W, the sample almost completely consists of crystallized drops of titanium carbide (fig. 3a). In comparison, at a power of 60 W, there are practically no melt regions (fig. 3b). In this case, the use of less power is preferable both for maximizing the yield of the MAX phase after SLS and obtaining the densest structure.



(a)



(b)

Fig.3. The surface of the samples obtained at a power of 80 W (a) and 60 W (b).

Results and discussion

- *SLS samples*

The XRD investigation has shown that the MAX phase content on the surface of the sample depends on the laser beam power. At high powers (more than 80 W), the complete absence of the MAX phase on the surface is possible. At a power of 60 W, only 10 wt.% of the Ti_3AlC_2 phase is detected on the surface. The phases of titanium carbide and tin were also detected. The results of the XRD analysis are consistent with the results of the EDX analysis, which confirms the destruction of the MAX phase and the formation of titanium carbide in the center of the laser track in SLS samples.

Bulk SLS-samples were obtained at powers from 20 to 60 W. The samples were separated from the substrate, and then the unsintered powder was removed using compressed air. After that, the obtained samples were ground into powder, and this powder was studied by XRD.

It was found that all samples have more than 95 wt.% of the MAX phase in their composition, the second phase is titanium carbide (fig. 4).

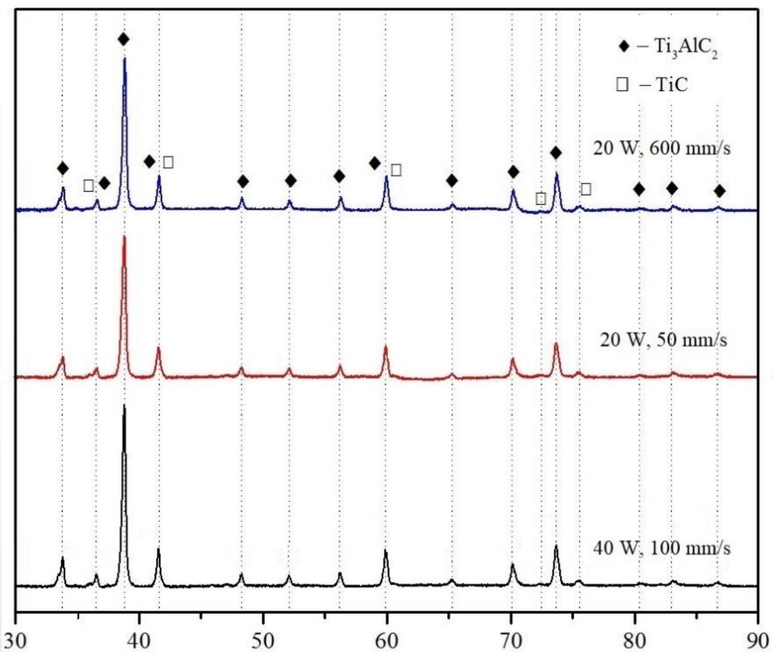


Fig. 4. XRD patterns of samples obtained with different power input.

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Results and discussion

❑ *SLS samples*

During the study of samples obtained at different laser powers and scanning speeds, it was found that at low powers, the heat input doesn't affect the phase composition. The titanium carbide content can't be significantly minimized since it depends on the number of tracks in the sample because the TiC phase is localized in the central part of the laser track.

The same hatch distance and the thickness of the powder layer were used in all the samples studied. Therefore, the titanium carbide content was unchanged. An increase in the hatch distance and the thickness of the powder layer can lead to a decrease in the titanium carbide content in the SLS-samples. At the same time, it leads to a lower strength of the SLS-samples as a result of less sintering of the powder particles.

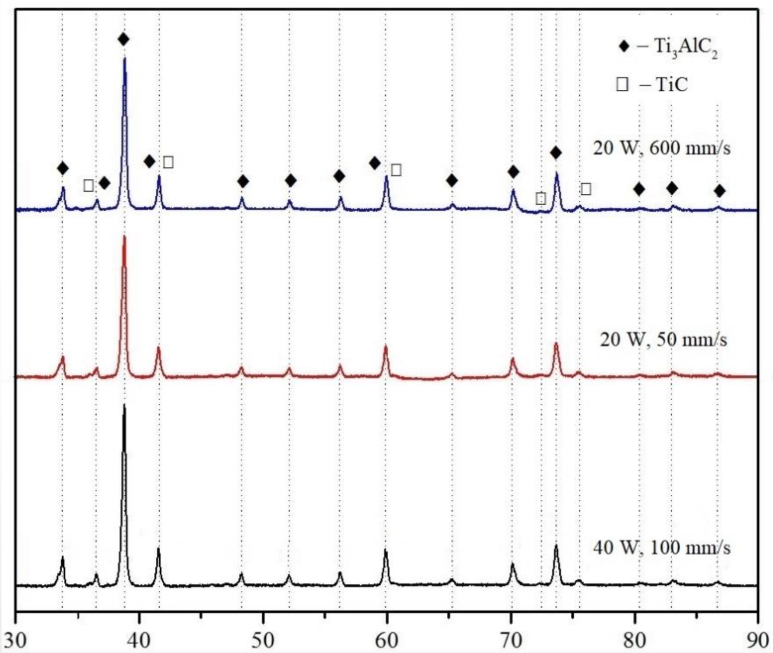

Fig. 4. XRD patterns of samples obtained with different power input.

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Conclusion

1. It was found that the maximum yield of the MAX phase in the samples after SLS is observed when the laser power is less than 60 W, and the scanning speed is more than 50 mm/s.
2. The scanning speed affects the sintering of the powder between the tracks and between the layers, so it must be varied simultaneously with the hatch distance and the powder layer thickness.
3. The maximum MAX phase destruction with the titanium carbide formation is observed in the center of the laser beam. The highest recorded content of the MAX phase of Ti_3AlC_2 in the sintered layer is 95%.

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Thanks for your attention!