Analysis of mechanical properties and microstructure of Ti-Al-C composites after spark plasma sintering

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Abstract: *Titanium rich alloys of Ti-Al-C and Ti-Al-B system was synthesised by Spark Plasma Sintering (SPS) and Field Activated Pressure Assisted Sintering (FAPAS) methods with altering sintering temperatures in the range of 950°C-1020°C with different current for the duration of 5 minutes. The initial powders of the composite 85%Ti and 15%Al was processed by High Velocity Energy Distribution (HVED) in a solution of kerosene at the specific energy of 25MJ/kg to reduce the size of the particles to nano-scale. This method of using different parameters of production technology have helped to analyse the most efficient, energy saving, and less waste generation technological process with improved mechanical properties. The metal-based samples were examined by optical and electron microscopy. Mechanical properties of composites were determined by measuring microhardness.*

The aim of the investigation was to determine the dependence between parameters of production technology and properties of Ti-AlC composites.

Keywords: TI-AL-C COMPOSITE, MICROSTRUCTURE, OPTICAL ANALYSIS, SEM, HVED.

1. Introduction

The method of production of the Ti-Al-C composite is directly proportional to the mechanical properties of the composite. The most common way of producing the composite by conventional casting was used in the early stages but however the occurrence of microstructural defects such as pores or cracks will lower the mechanical properties of the composite. To make it more stable, powder metallurgy was carried out by the scientists in which during gas atomization Ti-Al fine grain metallic powders will be obtained with homogeneous composition. Afterwards with the process of spark plasma sintering, the powdered particles will be compacted with the help of high intensity current and stress. A lot of industries are attracted to this because of its several advantages like speed, cheap and simplicity [1]. P Wang et al. [2] synthesized ternary layered carbide Ti2AlC by spark plasma sintering using elementary powder mixtures which showed great mechanical properties. D Wang et al. [3] synthesized Ti-Al-Cr-Nb alloy by spark plasma sintering with different sintering temperatures and stresses showed good mechanical properties. C Magnus et al. [4] synthesized a dual max phase composite by spark plasma sintering under vacuum condition by altering the composition of the raw materials showed good mechanical properties. It is clear that to obtain good mechanical properties, importance should be provided to the parameters of the production phase such as sintering temperature, current, inclusion particles, time etc.

The tests which are conducted to determine structure and properties of metal-based composites are Vickers hardness nano-indentation test for hardness property, microstructure analysis with the help of scanning electron microscope (SEM) and energy dispersive X-ray spectrometer (EDS) for determining the plastic deformation and other properties, etc. The phase analysis can be a key to determine the certain phases formed in the composite because of sintering temperature and chemical composition. The test results from X-ray diffraction can show the intensity of peaks of phases by which we can come to conclusion whether the composite material is stable or not [5].

In this paper, analysis of the tests such as Vickers hardness test, microstructural analysis and chemical composition were discussed in detail to compare the results with the experimental results obtained.

2. Methodology

The raw materials such as titanium (99.9% purity) and aluminum (99.9% purity) were used in the preparation of samples for the testing. These samples were prepared with varying sintering temperature, current, and time which is explained in detail in the Table 1. Spark plasma sintering and field activated pressure assisted sintering methods are opted for the preparation of samples. These methods are chosen since it is a high energy saving technology with less processing time and procedure. A technique of high voltage electric discharge W1=1kg and Wsum=25MJ/kg being applied on the powders rather than using an addition of inclusion particles. By this method, the Ti-Al-C and Ti-C powders are synthesised by the reaction between the discharge and the initial powders to result in homogeneous mixing of compounds.

The mixture obtained is compacted by the sintering process and the preservation of reinforcing phases takes place in the matrix.

The whole process of producing the sample was done in the Institute of Pulse Processes and Technologies, National Academy of Science of Ukraine with the help of Dr. Olha Syzonenko and coauthors.

Table 1: *Parameters of production technology for evaluating the properties*

Sample	Initial powders	Sintering methods	Sintering temperature, °C	Current, A	Time, min
13.2	15%Al+85%Ti	SPS	985	995	5
14.4	15%Al+85%Ti	SPS	1020	915	5
G13.2	15%Al+85%Ti	FAPAS	950	840	5
G14.2	15%Al+85%Ti	FAPAS	960	770	5

Preparation of metal-based composite samples for microscopic analysis was performed according to ASTM E3-11: 2017 [6]. The mounted, ground samples were polished using aqua-pol-diamond suspension (3µ, and then, 1µ grain size). After the process of polishing, the samples are immersed in the alcohol for few minutes to further remove the traces of debris.

After polishing the surface of the sample, experimental tests were conducted on it to determine the mechanical properties of the composite. The microhardness test was carried out on Innova test machine (Fig. 1). This machine is a universal machine which has the built-in option of choosing any kind of hardness testing method. The Vickers hardness test method was opted with load being 2.5kgf and dwell time of 10 seconds. The diamond indenter was used in this nano-indentation microhardness test. For each sample 30 indentations were performed, and the hardness values were noted down.

The structure of the samples was examined using:

- Optical microscope Nikon equipped with video camera Nikon DS-2 16 MP and objectives Nikon TU Plan Fluor 10×/0.30 and Nikon TU Plan Fluor 100x/0.90. NIS-Elements D software was used to analyze the photographs.
- Scanning electron microscope (JEOL IT 500 HR) equipped with an EDS analyzer (EDAX Octane Elite

Super). EDS was used for determining the chemical composition of phases.

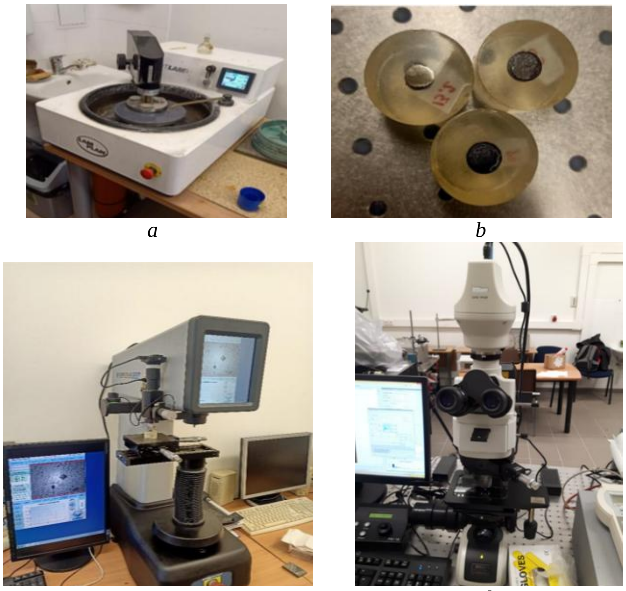


Fig. 1. Experimental setup: grinding of samples with Lamplan machine, b – prepared samples, c - the Innova test machine for determining hardness value, d – examination of the structure using optical microscope Nikon

3. Results and Discussion

3.1. Vickers hardness nano-indentation test analysis

The Vickers hardness nano-indentation test was carried out in the Innova test machine with load and dwell time being 2.5kgf and 10 seconds respectively. The samples were indented in 30 different spots with the diamond indenter to analyze the hardness values to a full extent. The figure 2 (a) and (b) shows the hardness values of the sample 13.2 and 14.4. These samples are sintered by SPS process at two different temperatures such as 985°C and 1020°C. There is almost a constant range of values all over the surface of the specimen 13.2. A peak of 668.7 HV is recorded when the nano-indentation was carried out on small network structures of phases near the edges. But it does not affect the other mechanical properties of the composite. The presence of very small pores in the sample 13.2 was the reason for achieving a less hardness value of 407.4 HV. The values of the sample 13.2 shows that there is a good densification achievement happened.

On reviewing the hardness values of the sample 14.4, there is wide range of difference between some points. A maximum value of 764.9 HV is achieved in the test where the indentation was carried out on the large network structure of hard phases. To contrast, some minimum hardness values such as 243 HV and 323.4 HV was achieved is some places around the clusters where a non-uniform distribution of particles surrounded. On comparing the two samples sintered by SPS process, the sample 13.2 has a good hardness value throughout the sample. Even though the sample 14.4 has the highest hardness value than 13.2, there are some places with poor hardness value range.

In the samples G13.2 and G14.2, the raw materials underwent an initial process in HVED solution before sintered by FAPAS method. The sintering temperature of 950°C and 960°C was used to sinter the sample G13.2 and G14.2 respectively. The result showed a minimum value of 439.2 HV (Fig. 2, c and d). The drop in value may be because of the unreacted hard particles accumulated near the areas which caused pores while sintering. The values remained in a good range in the other spots of the sample where nano-indentation was performed. A peak value of 1335.5 HV was identified at the place of clusters formed by the over allocation of particles at a same place. When the sample G14.2 was tested, it showed good hardness values throughout the whole readings. The slight increase in temperature may be the reason for uniform distribution of particles and formation of very few pores of small lengths than the big pores formed in the sample G13.2. A hardness of value of 879.2 HV was recorded which is lesser than the peak of G13.2 but the overall values show that the composite has good hardness values.

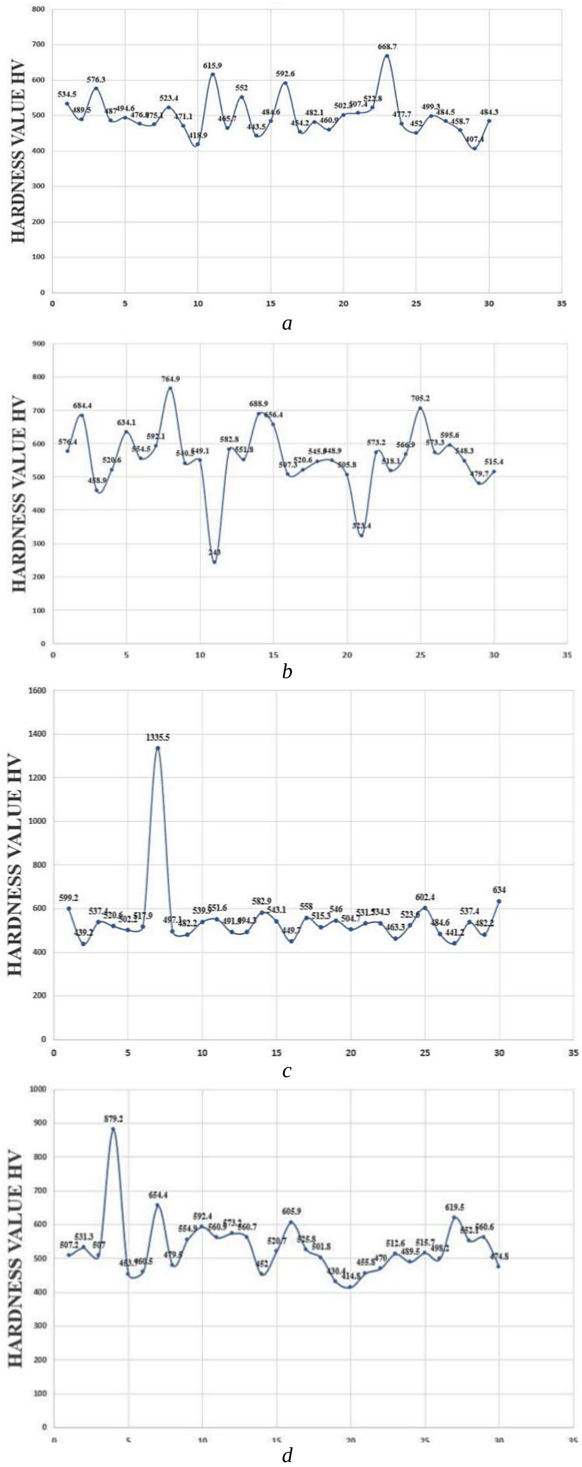
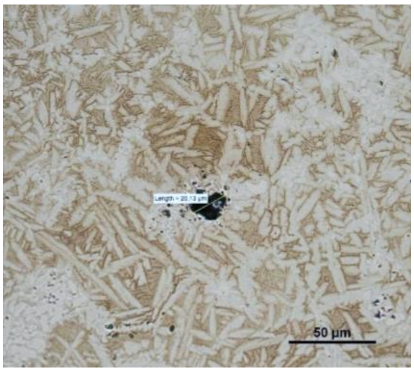


Fig. 2. Hardness value graph for the Ti-Al-C composites treated by HVED and sintered: a – SPS, 985°C, 995 A; b – SPS, 1020°C, 915 A; c – FAPAS, 950°C, 840 A; d – FAPAS, 960°C, 770 A

3.2. Optical analysis of the microstructure of composites

The optical micrographs of the polished un-etched surface of the as-synthesized Ti-Al-C composites are shown in the Fig. 3.

The microstructure of sample 13.2 consists of very few pores/voids not bigger than the length of 20.13µm (Fig.3, a). Also, the hardness values of the sample 13.2 which was taken in 30 different spots are almost between the range of 407.4- 668.7 HV. Hence a uniform densification has taken place. This adds an extra support to the statement regarding the density. In a recent study involving Ti-Al-C composite, the author S. Riaz et.al [7] results on the microstructure were almost similar to this project. By using 85%Ti, 10%Al and 5%C as initial powders, the composite was sintered by arc furnace method at 1050°C. The microstructure was composed of elongated plate-like structured β-Titanium as matrix, the carbides spread over the matrix as small dendritic structure and Ti_3AlC (P phase) as dark phase.



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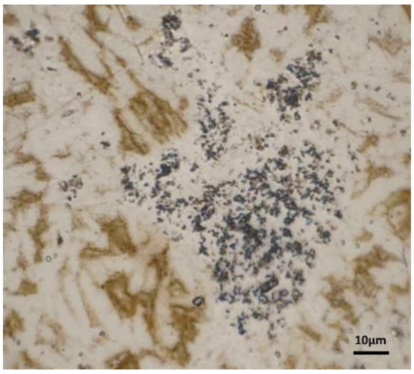

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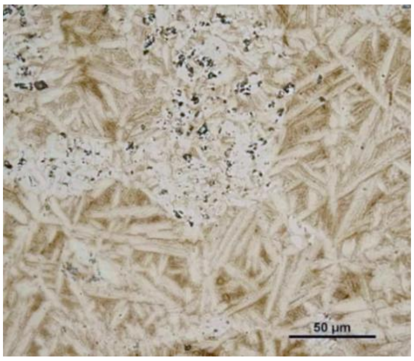

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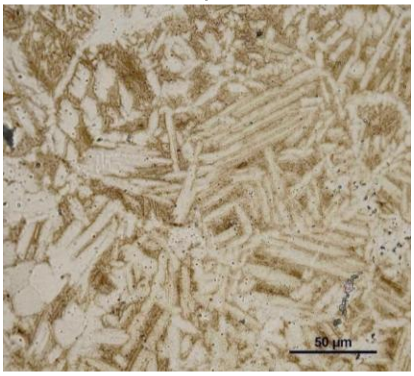

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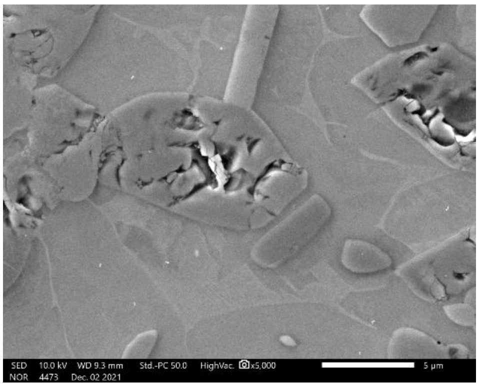
Fig. 3. *Optical micrographs of Ti-Al-C composites after such treatment: a – SPS, 985°C, 995 A; b – SPS, 1020°C, 915 A; c – FAPAS, 950°C, 840 A; d – FAPAS, 960°C, 770 A. Here – elongated β-Ti crystals, slightly brown – possible Ti-Al-C chemical compounds, and dark carbides*

According to author Syzonenko O. et.al [8], the same case of experimenting with 15%Al and 85% of Ti at 1100°C for 5 minutes by SPS with HVED is discussed. The results showed that due to the presence of phases C60 and C70 after the HVED process, the composite had the formation of Ti_2AlC max phase and Ti_3AlC in it. The optical microscopy results of the author's specimens depict that the dark phase formed in the composite as Ti_3AlC which also consisted of few traces of TiC. The light phase is formed of Ti, and it has some traces of the Ti_2AlC phases. In the present work with Ti-Al-C composite, the reaction between the HVED solution and the initial powders, a solid-liquid reaction takes place which forms the C. When comparing the results obtained from the literature, the microstructure consists of elongated needle like shapes all over the sample which may be the β-Ti matrix (Fig. 3, a-d)., the dark phase formed in the samples may be of Ti_3AlC or similar carbides is visible on the surface of the matrix (Fig. 3, b, c).

3.3 SEM analysis of Ti-Al-C composites

The electron microscopic analysis of the composite samples was carried out by Scanning electron microscope (SEM) at scientific institute COMTES Fht., Czech.

The Fig. 4 shows secondary electron scanning images of the sample 13.2 which was sintered by SPS method at 985°C for 5 minutes with current 995 A. The figure (a) shows the presence of small pores in the surface of composite. Due to the effect of sintering or poor compaction of materials these small pores could have generated. But in the figure (b) there is presence of cracks in the surface of hard carbides. In case of poor binding of materials in some places due to the effect of sintering and over allocation of particles in a place. According to the author S. Riaz et.al [7], the proper allocation of the particles is dependent on the current which is applied during the sintering process.



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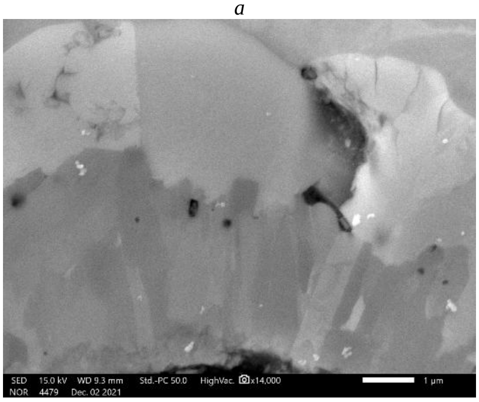
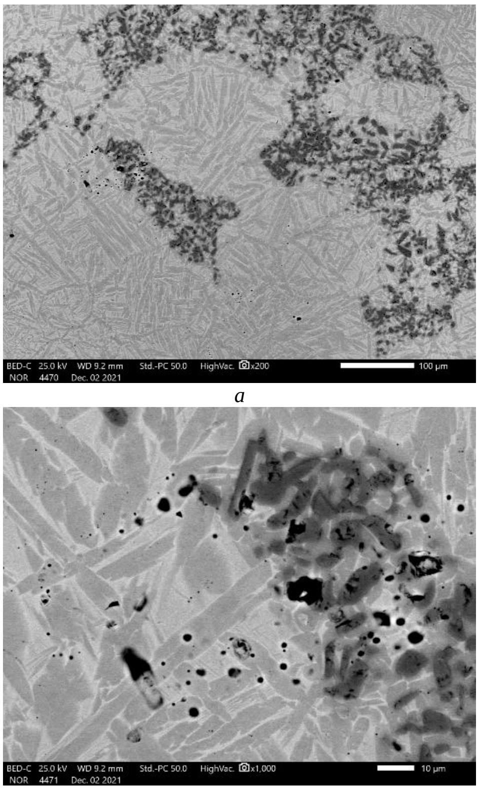


b

Fig. 4. *Secondary electron scanning image of Ti-Al-C sample: a – porous carbides are visible inside β-Ti matrix; b – small cracks inside carbides (at the upper right corner of the image)*

The back scattered electron images show the clear formation of matrix, dark phases, and light phases. As discussed before in the optical microscope analysis, the elongated needle shaped structures are the β- Titanium, the dark spots like pores are the presence of carbides and the light phase is Ti_3AlC. It is evident from the author S. Riaz work that the occurrence of martensitic transformation of β-Ti to α-Ti occurs when the sintering temperature is above 1250°C or when the ratio of Al powders increased. The other way of transformation is the quenching of water or cooling for some time after the sintering process. Now it is clear that the sample of Ti-Al-C composite sintered at 985°C temperature has not gone through the martensitic transformation of β-Ti to α-Ti. The uniform distribution of the elongated needle shaped β-Ti acts as the matrix which is evident in the Fig. 5 (a). The Fig. 5 (b) shows the grain growth of carbides into irregular dendritic form. Titanium inside the composite is more precipitative in the carbides when the sintering temperature is less than 1050°C. By the action of sintering parameters and the precipitation of titanium carbides tends to form porous structured dark phases at the grain boundaries of elongated β-Ti crystals.



a

b

Fig. 5. *Back scattered electron image of Ti-Al-C sample: dark carbides, slightly grey β-Ti and light particles – possible Ti_3AlC*

Further studies of Ti-Al-C composite materials sintered by different technologies are planned, as well, as the initial powder treatment by HVED process is going to be investigated in more detail. The following X-ray experiments will show exact phase composition of these composites allowing to collect more data about structure and properties of Ti-Al-C composites.

4. Conclusions

Investigation of the dependencies between the parameters of production technology and the mechanical properties of the Ti-Al-C composite was successfully carried out letting the formulation of such conclusions:

1. The hardness values of nano-indentation test shows that the samples such as 13.2, 14.4, G13.2, and G14.2, possess the peak hardness values of 668.7 HV, 764.9 HV, 1335.5 HV, and 879.2 HV, respectively. When comparing the values, the samples sintered by FAPAS method possess a range of good hardness values followed by the samples of SPS. A hard brittle nature on the surface of some areas of samples tends to provide a very high peak of values but the overall values is poor because of the formation of voids and cracks.

2. The optical microscopic analysis showed the formation of β-Ti in the shape of elongated needle-shaped formation distributed all over the surface as matrix. The hard black surface in the shape of small dendritic structures on the matrix are the carbides which are over accumulated in some grain boundaries of the 13.2 and 14.4 samples of Ti-Al-C composite. The slightly brown phase may be the Ti_3AlC phase. The samples 13.2 and 14.4 has comparatively less pores in the surface of the composite than the sample G13.2, G14.2.

3. The electron microscopic analysis on the sample 13.2 showed a clear imaging of the elongated needle-shaped structures of the β-Titanium, the dark spots are the presence of carbides and the white phase can be Ti_3AlC. The composite 13.2 has not gone through the martensitic transformation of β-Ti to α-Ti. Depending on sintering parameters and the precipitation character of titanium carbides, it tends to form porous structured dark phases at the grain boundaries.

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